

**Evaluation of Drying Technologies and Physico-Chemical
Characterization of Wheat Distillers Dried Grain with Solubles
(DDGS)**

(With a case study application in the Philippines)

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By

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Abstract

Wheat distillers dried grain with solubles (DDGS) is a co-product of ethanol production, primarily utilized as an animal feed ingredient. Reduced protein quality, a highly energy-intensive drying process, and product variability are some of the challenges that currently confront its production in western Canada. The main focus of this research undertaking was to examine, on a laboratory-scale, the effect of condensed distillers solubles (CDS) : wet distillers grain (WDG) blending ratio and drying conditions on the protein quality and physico-chemical characteristics of wheat DDGS. The potential of microwave-based drying methods in minimizing protein damage and energy consumption was investigated. An auxiliary case study was also conducted in the Philippines to apply the knowledge and skills acquired from the PhD research undertaking to a related problem situation in a developing country. It aimed to provide more information about brewers spent grain (BSG) supply and utilization in Misamis Oriental, Philippines and enhanced the efficiency and safety of BSG utilization as an animal feed ingredient.

Laboratory-scale investigations used samples produced at three CDS:WDG blending ratios (by mass): 15:85 (15% CDS), 30:70 (30% CDS), and 45:55 (45% CDS) and dried under forced air convection (40-120°C), and under microwave (420 – 805 W) and microwave convection (nominal settings of 130°C-30% power to 190°C-30% power) methods using a domestic microwave oven. Freeze-dried samples were used as standards in evaluating chemical composition and color of wheat DDGS.

As CDS level in the blend was increased, protein and ash content of freeze-dried samples increased while fat, acid detergent fiber (ADF) and neutral detergent fiber (NDF) content decreased. These trends were attributed to proximate composition differences between CDS and WDG fractions. The CDS fraction had higher protein and ash and lower fat, ADF, and

NDF contents compared to WDG. Variation in the CDS:WDG blending ratio employed in the source ethanol plant could be one of the factors contributing to the observed proximate composition differences between two ethanol plant-sourced wheat DDGS samples. This was verified through proximate analyses of: (i) plant-sourced wheat DDGS samples from two production batches; (ii) CDS and WDG samples obtained on the same production date; and (iii) laboratory-produced wheat DDGS samples at varying CDS:WDG blending ratios.

Protein, ash, and NDF contents of forced-air convection-, microwave-, and microwave convection-dried samples also showed strong linear relationships with CDS level. Fat and ADF content, however, did not exhibit similar strong relationships with CDS level, indicating the influence of drying conditions. Maximum lysine and minimum acid detergent insoluble crude protein (ADICP) contents were achieved in blends with the highest CDS level (45% CDS) and dried under lower drying temperature (80°C), microwave power (676 W), and microwave convection (150°C-30% power) settings. Microwave- and microwave convection drying achieved desirable protein quality associated with lower temperature drying under much shorter times. Laboratory-scale drying of ethanol plant-sourced wet distillers grain with solubles (WDGS) under forced air convection produced DDGS samples with decreased lysine content as drying air temperature was increased.

In terms of physical properties, dried samples with higher CDS level were significantly finer, denser, less flowable, less dispersible, have lower thermal diffusivity and higher internal friction coefficients, and produced denser and stronger pellets. Color parameters of freeze-dried samples were significantly affected by CDS level. As CDS level increased, lightness (Hunter L) decreased while redness (Hunter a) increased. The color parameters of forced-air convection-, microwave-, and microwave convection-dried DDGS samples did not, however, exhibit similar linear trends with CDS level as these were also affected by drying conditions,

such as drying air temperature and microwave power level. Effective moisture diffusivity values, estimated from the drying data, were also significantly affected by drying conditions (drying air temperature and microwave power levels), CDS level, and interaction between drying air temperature and CDS level. Effective moisture diffusivity decreased as CDS level was increased. Physical properties of two commercial (ethanol plant-sourced) wheat DDGS samples, as affected by moisture content, were also assessed.

Techno-economic evaluation results indicated that complete replacement of the conventional hot air drying with microwave drying technology was not yet economically feasible. Although energy consumption during drying was substantially reduced with the use of microwave energy, the cost of electricity to generate microwave energy was high. Incorporating microwave drying toward the end of the hot air drying process was seen as the more economically viable alternative.

Drying of BSG was not commonly practiced in Misamis Oriental, Philippines. The material was typically stored in open concrete bins and commonly fed to dairy cattle in its wet form. A prototype batch dryer, powered either by solar or biomass energy, was developed to improve shelf life and safe use of BSG. Initial tests showed that drying spent grain using a biomass furnace was feasible. Operating the dryer using solar energy was not an attractive option because of the long drying time. Basic physical attributes, proximate composition, and moisture sorption characteristics of the dried BSG were also determined.

Practical implications of the results of these two studies to their respective local contexts were discussed. Recommendations to further improve understanding of wheat DDGS protein quality and physico-chemical characteristics, BSG dryer performance, backyard farmers' sustainable access and safe use of BSG were also presented.

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Nomenclature

Abbreviations

ADF	Acid detergent fiber, % dry matter
ADICP	Acid detergent insoluble crude protein, % crude protein (dry basis)
AOAC	Association of Official Analytical Chemists
BSG	Brewers spent grain
CAN\$	Canadian dollars
CDS	Condensed distillers solubles
CPS	Canada Prairie Spring
db	Dry basis
DDGS	Distillers dried grain with solubles
GAB	Guggenheim, Anderson, and de Boer model
MLPY	Million liters per year
MR	Moisture ratio
MRE	Mean relative percentage deviation
MSE	Mean square error
NDF	Neutral detergent fiber, % dry matter
RH	Relative humidity
SEE	Standard error of the estimate
US\$	United States dollar
wb	Wet basis
WDG	Wet distillers grain
WDGS	Wet distillers grain with solubles

Symbols

A	Adhesion, kPa
A _s	Lateral surface area, m ²
a	Hunter Lab redness parameter
a _s	Hunter Lab redness parameter of a standard sample
a*	CIE Lab redness parameter
b	Hunter Lab yellowness parameter
b _s	Hunter Lab yellowness parameter of a standard sample
b*	CIE Lab yellowness parameter
C	Degree of volume reduction
C _C	Cohesion, kPa
C _{DEP}	Depreciation cost per hour of operation, CAN\$
C _E	Total equipment cost, CAN\$
C _{GM}	General maintenance cost per hour, CAN\$
C _i	Total installation cost, CAN\$

C_M	Magnetron replacement cost per hour, CAN\$
C_{NG}	Cost of natural gas consumption per hour, CAN\$
C_P	Cost of electricity per hour, CAN\$
C_{RD}	Cost of rotary direct-fired dryer, CAN\$
C_T	Training cost per hour, CAN\$
C_W	Cost of water consumption per hour, CAN\$
c_p	Specific heat capacity, $J \cdot kg^{-1} \cdot ^\circ C^{-1}$
d	Kawakita Ludde model parameter associated with porosity
D_{eff}	Effective moisture diffusivity,
E_G	Microwave energy requirement for drying
E_L	Total heat load for a drying scenario
E_S	Amount of energy drawn from the main supply lines
E_P	Electrical energy consumption
e_A	Efficiency of microwave applicator
e_G	Efficiency of microwave generator
e_D	Efficiency of rotary drum dryer
e_C	Combustion efficiency
f	Kawakita Ludde model parameter, the reciprocal of which is associated with porosity
F, G, H	Moisture sorption model parameters
g, h	thin layer drying model parameters
I_1	Investment cost for the known capacity, CAN\$
I_2	Investment cost for the desired capacity, CAN\$
i	Annual interest rate
k	Drying rate constant in thin layer drying models, m^{-1}
l	Slab thickness, m
L	Hunter Lab lightness parameter
L_s	Hunter Lab lightness parameter of a standard sample
L^*	CIE Lab lightness parameter
L_E	Economic life, years
L_v	Latent heat of vaporization, $kJ \cdot kg^{-1}$
L_{ys}	Lysine content, % dry matter
M_e	Equilibrium moisture content, dry basis
M_i	Initial moisture content, dry basis
M_t	Moisture content at any time t, dry basis
M_W	Cooling water requirement per microwave generator
m, r	Jones model parameters
m_w	Amount of water to be evaporated, kg
m_p	Mass of the material to be dried, kg
N	Page model parameter
N_G	Number of microwave generators
N_M	Number of manpower
n	Number of replicates, observations, or data points

P	Compressive pressure, MPa
P_{mw}	Microwave power level, W
P4	40% of rated microwave power level
P6	60% of rated microwave power level
P8	80% of rated microwave power level
P10	100% of rated microwave power level
p_i	Percentage of the total installation cost
Q	Amount of heat transferred per unit time, $\text{kJ}\cdot\text{s}^{-1}$
Q_1	Known capacity of a microwave drying system, MW
Q_2	Desired capacity of a microwave drying system, MW
R^2	Coefficient of determination
R	Linear correlation coefficient
S1	Plant-sourced DDGS sample 1
S2	Plant-sourced DDGS sample 2
S_V	Salvage value
T	Absolute temperature, °K
T_c	Drying air temperature, °C
T_i	Initial product temperature, °C
T_f	Final product temperature, °C
t	Drying time, min
t_A	Annual operating time, h
t_h	Time of heating, s
U_M	Unit training cost, CAN\$ per person
U_{NG}	Cost of natural gas per hour, CAN\$
U_p	Unit cost of electricity, CAN\$ per kWh
U_W	Cost of water per hour, CAN\$
V	Volume of compact at pressure P, m^3
V_o	Volume of compact at zero pressure, m^3
x	Cost capacity factor
ΔT	Temperature difference, °C
ΔE	Total color difference
ε	Porosity, decimal
ρ_b	Bulk density, $\text{kg}\cdot\text{m}^{-3}$
ρ_p	Particle density, $\text{kg}\cdot\text{m}^{-3}$
τ	Shear stress, kPa
σ	Normal stress, kPa
θ	Angle of internal friction

Chapter 1

Introduction and Objectives

1.1 Background

Saskatchewan hosts five of the eight grain-based ethanol plants in western Canada and accounts about 60% of the region's nameplate production capacity (Canadian Renewable Fuels Association 2013). The significant expansion of the province's grain ethanol industry in recent years (Fig. 1.1) translated to the increased availability of wheat distillers dried grain with solubles (DDGS), co-product of ethanol production mainly utilized as an animal feed ingredient.

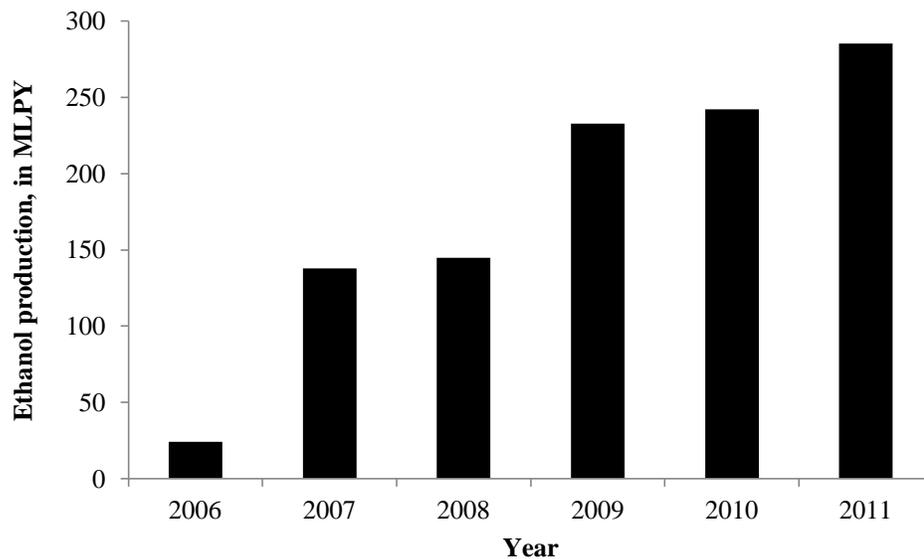


Fig. 1.1 Grain ethanol production in Saskatchewan, in million liters per year (MLPY), 2006 – 2011. (Source of base data: Government of Saskatchewan 2013).

With a total nameplate ethanol production capacity close to 345 million liters per year (MLPY), it is estimated that about 300,000 tonnes of wheat DDGS are available in the province annually (University of Saskatchewan 2010). Sale of this co-product as animal feed or for other uses is an

additional revenue generation stream for grain ethanol plants. In corn DDGS, for example, a spreadsheet model estimated that for every gallon of ethanol produced, DDGS contributed between 7-26% of the total revenue (Hofstrand 2013). A similar revenue contribution may be assumed for wheat DDGS.

Wheat DDGS production and utilization, however, is confronted with a number of challenges. These challenges include protein damage, an energy-intensive drying process, and product variability (University of Saskatchewan 2010). Several animal feed studies, for example, reported low lysine content, availability and digestibility in wheat DDGS, despite its high crude protein content (Nyachoti et al. 2005; Widyaratne and Zijlstra 2007; Lan et al. 2008; Cozannet et al. 2010). Loss of lysine, an essential amino acid in animal diet, lowers the nutritive value of a feed ingredient (Fayle and Gerrard 2002) and limits the utilization of wheat DDGS, particularly as feed ingredient for monogastric animals (Widyaratne and Zijlstra 2007). Nuez-Ortin (2010) also found significant nutrient variability in wheat DDGS samples obtained from two Saskatchewan plants and stressed the importance of product consistency not only for more accurate animal feed formulations but also for improving the market prospects of wheat DDGS. While drying lengthens the shelf life of DDGS, it is also an energy-intensive process, estimated to account about one-third of the energy demand of wheat ethanol plants (Murphy and Power 2008). It has also been identified as one of the processes that adversely affect lysine content and digestibility in wheat DDGS (Nyachoti et al. 2005; Zijlstra and Beltranena 2009). Marked losses of lysine during drying has been attributed to Maillard reaction, which involves the binding of amino groups of amino acids with the carbonyl compounds of reducing sugars (Widyaratne and Zijlstra 2007; Cozannet et al. 2010; Nyachoti et al. 2005) rendering some of the lysine to become biologically unavailable to the animals. These potential negative impacts highlight the need to

investigate alternative drying technologies that could minimize protein damage and energy consumption.

1.1.1 Wheat DDGS production process

Since wheat DDGS is a co-product of ethanol production, its resulting chemical composition and physical characteristics are influenced by the type of feedstock used for ethanol production and by the processes and conditions employed during ethanol and DDGS production. A thorough understanding of the feedstock and the associated processes and conditions would be important in understanding its physico-chemical characteristics.

1.1.1.1 *The wheat grain*

Table 1.1 shows the starch and protein content of some of the market classes of Canadian wheat. Ethanol is produced from high starch, low protein wheat cultivars, such as those classified under the Canada Prairie Spring (CPS) and the Canada Western Soft White Spring market classes. The CPS, for example, has been “recognized as a viable feedstock for ethanol production” (Government of Saskatchewan 2007). Posner (2000) presented the compositional differences among the parts of the wheat kernel (Table 1.2). The endosperm, which comprised the bulk of the kernel, is high in starch. The aleurone is high in pentosans, protein, ash and fat while the pericarp is mainly cellulose and pentosans. The germ, which composed of the embryo and the scutellum, is high in protein, fat, and ash.

Table 1.1 Starch and protein content ^a of some Canadian wheat market classes

Market class	% Starch content	% Protein content
Canadian Prairie Spring	72.4	13.0
Canadian Western Soft White Spring	71.6	12.9
Soft White Spring	69.4	14.0
Canadian Western Extra Strong	69.8	13.5
Hard White Spring	69.1	15.0
Hard Red Spring	68.2	15.5
Canadian Western Red Spring	68.2	15.8

^aArithmetic mean values of the data presented by Hucl and Chibbar (1996)

Table 1.2 Proximate composition^a of wheat kernel and kernel parts

	Whole kernel (%)	Embryo (%)	Scutellum (%)	Pericarp (%)	Aleurone (%)	Endosperm (%)
Common wheat	100	1.2	1.54	7.9	6.7 – 7.0	81 - 84
Proximate analysis						
Protein	12.6	35.0	26.0	5 – 7	18	7.4 – 13.9
Ash	1.9	5.45	5.0 – 8.0	3 – 4	14 – 17	0.28 – 0.40
Fat	1.6	16.3	32.0	3 - 5	10	0.8 – 1.5
Starch	59.2					68
Pentosans	6.7		6.6	34.9	39.0	1.4
Cellulose	2.3		2.0	38.4	3.5	0.3

^a14% moisture basis; Source: Adapted from Posner (2000)

1.1.1.2 *Ethanol production*

Figure 1.2 presents a simplified diagram that outlines the processes involved in dry grind ethanol production and the subsequent whole stillage processing into DDGS (Kwiatkowski et al. 2006; Terra Grain Fuels, Inc. 2009; Poundmaker Agventures Ltd., 2009; NorAmara Bioenergy Corp. 2009; US Department of Energy no date (n.d.)). In its simplest form, ethanol production from whole wheat grain involves 4 major processes: (i) size reduction of whole grains; (ii) enzymatic hydrolysis of starch to sugar; (iii) fermentation of sugars to ethanol by yeasts; and (iv) ethanol recovery and concentration. These processes are briefly described below.

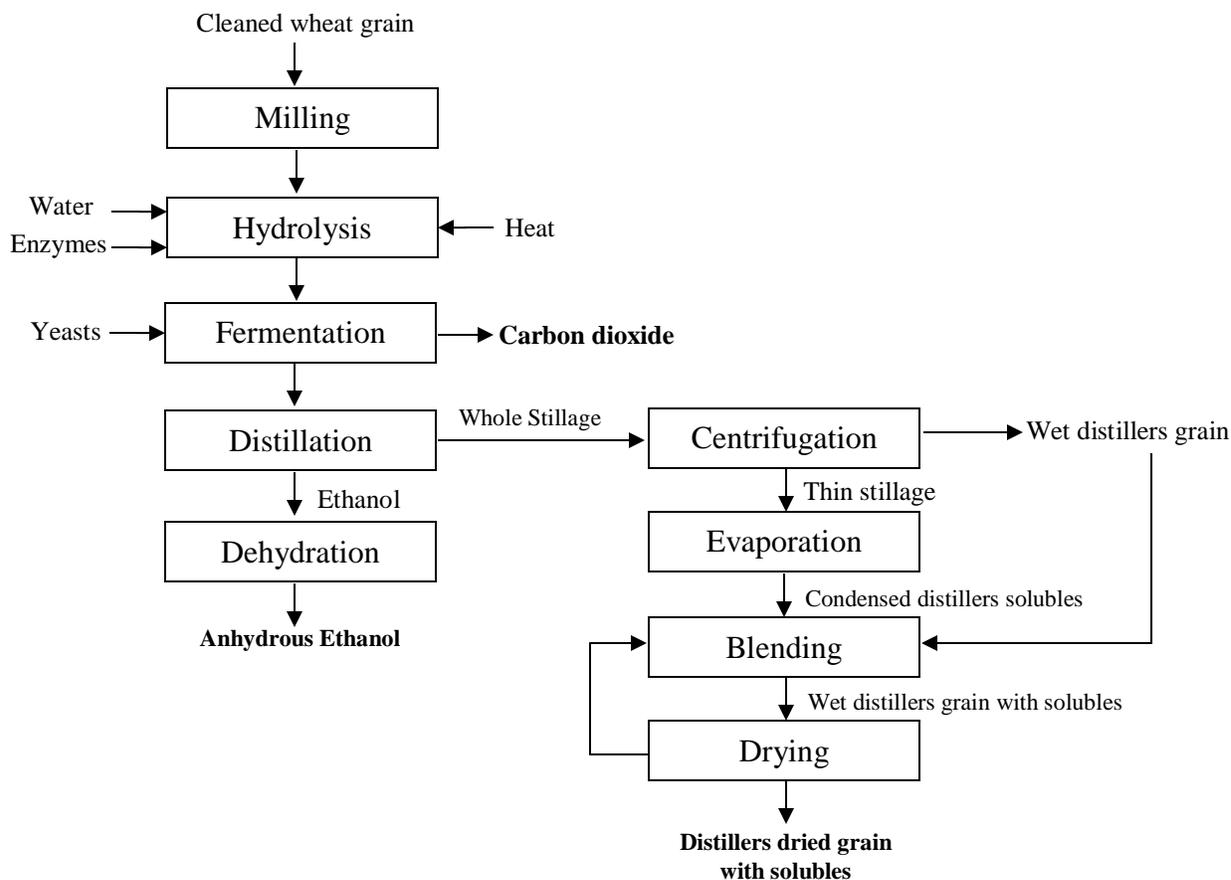


Fig. 1.2 The unit operations comprising the conventional dry-grind process of fuel ethanol and distillers dried grain with solubles production (Based from: Kwiatkowski et al. 2006; Terra Grain Fuels, Inc. 2009; Poundmaker Agventures Ltd., 2009; NorAmera Bioenergy Corp. 2009; US Department of Energy no date (n.d.)).

Size reduction. Ethanol production begins with the grinding of cleaned whole kernels using hammer mills. Breaking up the grain into smaller sizes provides more surface area for heat and mass transfer and better contact with enzymes in subsequent processes (Kelsall and Lyons 2003a; NorAmera Bioenergy Corporation 2009). While particles should be as fine as possible for efficient ethanol production, it is, however, a compromise considering increased energy demand for fine grinding (Rausch et al. 2005) and so as not to cause downstream problems in the

recovery of co-products (Kelsall and Lyons 2003a). Reduced DDGS particle size, for example, is thought to adversely affect handling characteristics of DDGS and ruminant digestion (Rausch et al. 2005).

Starch hydrolysis. After milling, the three sequential steps of gelatinization, liquefaction, and saccharification convert starch to fermentable sugars. Gelatinization involves mixing the milled grain with process water in a slurry tank and heating the mash at 90 - 120°C. Heating sterilizes the mash and weakens the inter- and intra-molecular hydrogen bonds of the glucose units, allowing water absorption (Power 2003). As the starch granules absorb water, they swell and lose their crystalline structure, resulting to a mixture with a “highly viscous, gelatinous consistency” (Franceschin et al. 2008). The liquefaction step involves the introduction of the α -amylase enzyme into the mixture, breaking the α -1,4 glucosidic linkages in amylose and amylopectin at high temperatures (90-110°C), with the shorter-chained oligosaccharides, known as dextrans, as its products (Kelsall and Lyons 2003b). Saccharification is the release of the individual glucose molecules from the liquefied mixture of dextrans, using glucoamylase at around 60°C (Kelsall and Lyons 2003b; Kwiatkowski et al. 2006). Glucoamylases break down the dextrans by hydrolyzing the glucosidic linkages. The liquefied mixture is then cooled to about 32°C in preparation for fermentation (Kelsall and Lyons 2003b).

Fermentation. This process involves the conversion of glucose to ethanol and carbon dioxide using the yeast *Saccharomyces cerevisiae*, through a sequence of biosynthetic reactions known as the Embden-Meyerhof-Parnas (EMP) or the glycolytic pathway (Power 2003). Since fermentation is central to ethanol production, ensuring the provision of the essential requirements for optimum yeast growth is of paramount importance. Several studies have been conducted to

improve the efficiency of the fermentation process, including, but not limited to, kernel fractionation (Singh et al. 2005; Wang et al. 2005; Murthy et al. 2006), use of proteases (Perez-Carillo and Serna-Saldivar 2006; Perez-Carillo and Serna-Saldivar 2007; Perez-Carillo et al. 2008), and better performing starch hydrolyzing enzymes (Wang et al. 2009).

Distillation and dehydration. The last stage involved ethanol recovery and concentration, wherein the products of fermentation are separated using distillation and the ethanol recovered is subjected to purification. Murphy and Power (2008) described this process, which need at least three distillation columns. One column is used to separate the ethanol solution from the unfermentable residues (whole stillage) while the other two are needed to upgrade the ethanol solution to 96% ethanol and to separate it from fusel oils, which are higher alcohol products and are less volatile. Ethanol obtained from distillation is further purified to 99.5 – 99.9% using molecular sieves in a physical absorption process (Murphy and Power 2008).

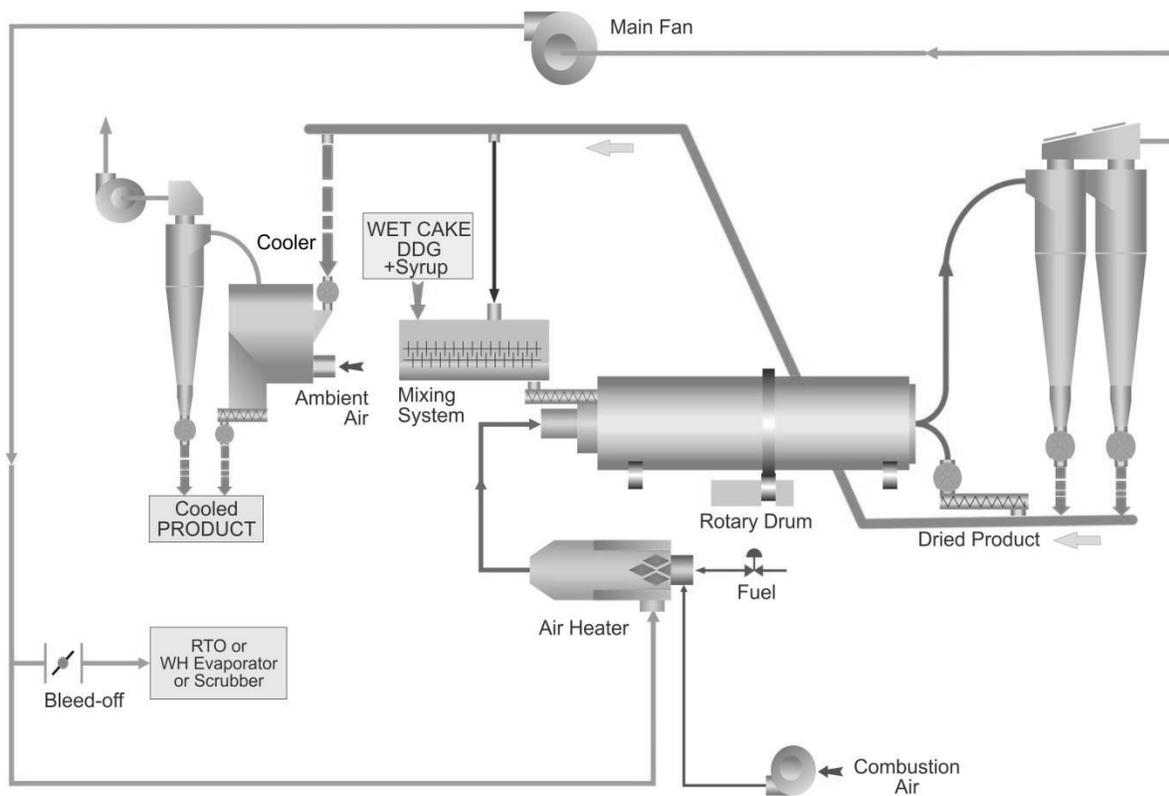
1.1.1.3 *Whole stillage processing*

The conversion of whole stillage obtained from the distillation process into DDGS involves (i) centrifugation, which separates the whole stillage into two streams: wet distillers grain (WDG) and (ii) the thin stillage; (ii) evaporation, wherein the thin stillage is transformed into condensed distillers solubles (CDS); (iii) blending of CDS and WDG, which produces wet distillers grain with solubles (WDGS); and (iv) drying process, which transformed WDGS into DDGS (Kwiatkowski et al. 2006; NorAmera Bioenergy Corporation 2009; US Department of Energy no date (n.d.)).

1.1.2 Drying wet distillers grain with solubles

1.1.2.1 Conventional hot air drying

Conventional hot air drying of wheat WDGS in Saskatchewan is accomplished through the use of rotary direct-fired dryers and ring dryers (FOBI Network 2011). In a rotary drum drying system (Fig. 1.3), WDGS is fed into a slightly inclined rotating cylinder, which is equipped with flights to carry the wet material up and shower it down through the hot air stream, enabling moisture removal (Krokida et al. 2007; Carlson n.d.).



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Fig. 1.3 A rotary drum drying system (With permission from GEA Barr-Rosin).

There is very limited published information about plant-scale rotary drying of wheat WDGS in the province. In the United States, Ileleji and Rosentrater (2008) described two drying configurations (Fig. 1.4) commonly found in old and new generation corn fuel ethanol plants. In the old generation plants (Fig. 1.4a), drying occurred as a single step process using one or more high capacity rotary drum steam tube dryers, after the blending of WDG, CDS, and a DDGS recycle stream in a screw conveyor trough.

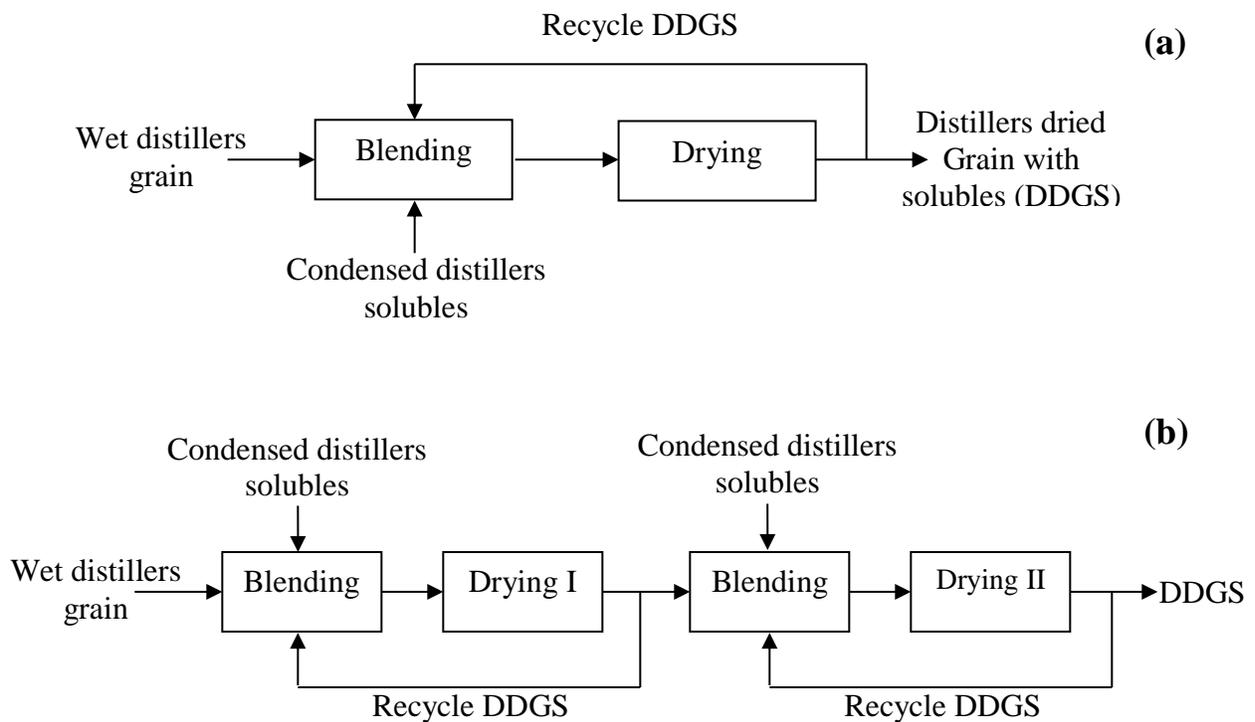


Fig. 1.4 Common drying process configuration found in (a) “old” and (b) “new” corn fuel ethanol plants in the US (Source: Ileleji and Rosentrater 2008).

In new generation plants (Fig. 1.4b), the blending and drying processes were done in two stages. The first stage is similar to what was shown in Fig. 1.4a, where WDG, CDS, and a DDGS recycle stream were blended and then dried. The second stage involved blending part of the resulting DDGS in stage 1, which has about about 35% moisture (wb), with more CDS and

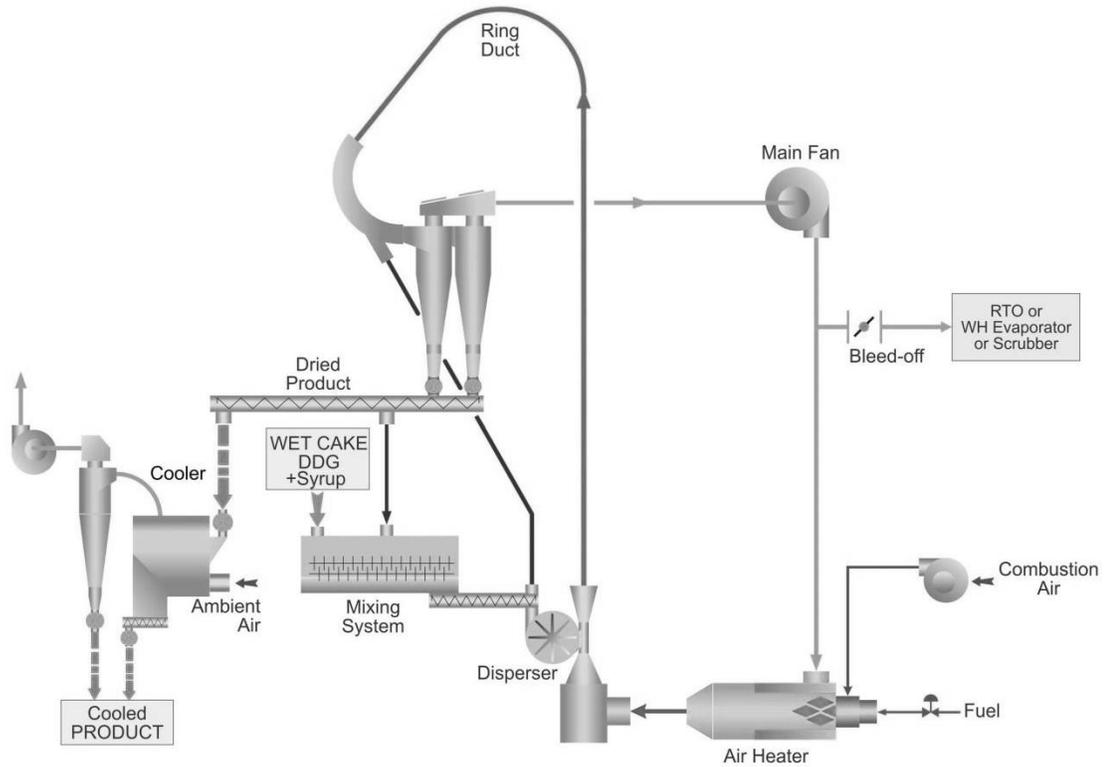
another DDGS recycle stream and subsequently drying the mixture. The inclusion of DDGS recycle streams during blending is thought to speed up the drying process, thus reducing the plant's energy requirements (Ileleji and Rosentrater 2008; Kingsly et al. 2010).

High temperatures are employed during drying of WDGS. In the plant-level study of Kingsly et al. (2010), inlet and outlet temperatures of the two rotary drum dryers were about 415 - 499°C and 105 – 111°C, respectively, while product temperature ranged from 93 to 102°C. Ileleji and Rosentrater (2008) also indicated that product temperature in the rotary dryers could reach up to 149°C with very high inlet temperatures of up to 538°C.

A ring dryer (Fig. 1.5), on the other hand, is a pneumatic drying system, wherein wheat WDGS is dispersed and conveyed through the dryer using a high speed hot air stream (Svoyan 2007; GEA Process Engineering Inc., Barr-Rosin n.d.). The drying system is equipped with a manifold, which facilitates separation of the wetter, heavier solids from the drier lighter particles. The wetter solids are recycled back for further drying while the dried material exits with the exhaust air and passes through a series of cyclone separators to separate from the exhaust air (Svoyan 2007; GEA Process Engineering Inc., Barr-Rosin n.d.). In a visit to a Saskatchewan ethanol plant that employed a ring dryer for its DDGS production, the plant personnel indicated that inlet and outlet temperatures typically ranged from 225°C to 275°C and from 110°C to 130°C, respectively.

Drying costs. Drying is an expensive process. To produce a gallon of ethanol and dry the distillers grain, for example, it was estimated that an ethanol plant would require about 40 MJ (38000 BTU), one-third of which is used to dry distillers grain (BBI International 2003). There is, however, limited information on drying cost estimates for wheat WDGS. In corn DDGS,

Perrin et al. (2009) estimated drying cost at US\$ 25.80 per tonne of dry matter. This estimate, however, was based on the cost of natural gas consumption in drying the material from 55% to 10% moisture (wb) plus the cost of initial moisture reduction using centrifuges and filters. In other studies, such as those by Eidman (2003), Shapouri and Gallagher (2005), and Dale and Tyner (2006), the costs of ethanol and DDGS production were not separately analyzed.



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Fig. 1.5 DDGS production using a ring dryer (With permission from GEA Barr-Rosin).

1.1.2.2 Use of microwave energy

Conventional hot air drying is a highly energy-intensive process and the elevated temperatures employed during the process were identified to be among the major causes of protein damage in DDGS (Nyachoti et al. 2005; Widyaratne and Zijlstra 2007). Thus, there is a need to search for

an alternative method that would minimize both protein damage and energy consumption during drying of wheat WDGS.

The use of microwave energy in drying has been investigated in a wide variety of products, particularly those whose exposure to elevated drying temperatures for long periods of time can seriously damage their nutritional characteristics and other quality attributes. Some of these studies covered fruits (Chung and Furutani 1989; Funebo and Ohlsson 1998; Maskan 2000; Maskan 2001; Mousa and Farid 2002; Sunjka et al. 2004; Dadali et al. 2007a; Meda et al. 2008; Wang et al. 2009), grains and oilseeds (Walde et al. 2002; Lupinska et al. 2009), leafy plant parts (Cui et al. 2004; Ozkan et al. 2007; Sosyal 2004; Alibas 2009), and bulbs, tubers, and root crops (Lin et al., 1998; Sharma and Prasad 2001; Song et al. 2009).

Microwaves are electromagnetic waves with wavelengths ranging from 1 mm to 1 m and frequencies between 300 MHz to 300 GHz (Dibben 2001; Schiffmann 2007). Schiffman (2007) provided the following overview of how microwave energy facilitates moisture removal during drying. Water, a dipole molecule, is one of the primary components of a material that absorbs microwave energy. As microwave travels through a material, the polarity of its electrical and magnetic fields reverses many millions of times per second (915 or 2450 MHz). As the electrical field builds up to its maximum strength, it pulls the water molecules into alignment, and when it starts to decay to zero, the water molecules would resume their normal random orientation. This repeated movement of water molecules due to the flipflopping of the electrical field causes the material to heat up. This heat generation creates a pressure gradient between the core and surface of the material, which facilitates water movement and its subsequent removal from the material.

Drying kinetics and dried product quality characteristics were among the common areas of investigation, whether microwave energy is employed alone or used in combination with other drying methods such as forced air convection and vacuum drying. The following subsections present a summary of some studies focusing on microwave, microwave convection, and microwave vacuum drying of biological materials.

Microwave drying. Reduction in drying time, color maintenance, and ascorbic acid retention were among the cited benefits derived in the microwave drying of okra (Dadali et al. 2007a), spinach (Ozkan et al. 2007; Dadali et al. 2007b), collard leaves (Alibas 2009), parsley (Sosyal 2004), and wood strands (Du et al. 2005). All five studies reported reduction in drying time as microwave power level increased. The color of microwave-dried parsley (Sosyal 2004), collard (Alibas 2009), and spinach (Ozkan et al. 2007) were close to or comparable to the green color of their respective fresh leaves. Higher ascorbic acid retention in microwave-dried collard (Alibas 2009) and spinach (Ozkan et al. 2007) leaves compared to their hot air-dried counterparts was also reported. Other advantages include lower emissions of volatile organic compounds and energy efficiency in wood strand drying (Du et al. 2005) as well as lower number of damaged rapeseeds (Lupinska et al. 2009) compared to traditional hot air drying.

Some of these studies also highlight the need for appropriate selection of microwave power levels to achieve the desired product quality characteristics. Ozkan et al. (2007), for example, reported that the best color values in spinach was achieved at 500 – 750 W, with the greatest loss in lightness observed at 1000 W. Lowest ascorbic acid value was obtained at 90 W, which also had the longest drying time. In collard, Alibas (2009) indicated that the best and worst ascorbic acid values were observed at 750 W and 250 W, respectively, and color values closest to those of the fresh product were recorded at the 750 W microwave power level.

Microwave convection drying. There were also numerous investigations focused on the combined use of microwave energy and hot air in drying. According to Schiffman (2007), microwave energy can be combined with conventional drying systems in three ways: (i) preheating, where the microwave energy is applied at the dryer entrance, before conventional hot air drying starts; (ii) boost drying, where microwave energy is applied after conventional hot air drying rates started to fall off; and (iii) finish drying, where microwave energy is applied near the end of the conventional hot air drying. This phase of the drying process is considered the least efficient because two-thirds of the time may be spent in removing the remaining one-third of the moisture (Schiffman 2007).

Use of both microwave energy and hot air, either applied simultaneously or one after the other, showed advantages over hot air and/or microwave drying of garlic cloves, banana, kiwifruit, and other fruits like apple, strawberry, and tomato. In studying the hot air, microwave, and hot air-microwave drying of banana slices, Maskan (2000) found that the hot air-microwave-dried slices were much lighter and had higher rehydration capacity than the hot air-dried-samples. In kiwifruit slices, those that were hot air-microwave-dried had higher rehydration capacity and lower shrinkage compared to those dried using hot air or microwave energy only (Maskan 2001). In these two studies, microwave energy was applied after conventional hot air drying has reduced the product moisture to about 55% (wet basis). Simultaneous use of microwave energy and hot air during the entire drying process also produced superior quality dehydrated garlic cloves (Sharma and Prasad 2001). The microwave-hot air-dried cloves were lighter in color and retained higher amount of volatile compounds responsible for flavor strength compared to conventional hot air drying. Color degradation of apple, strawberry, tomato and mushroom dried under the microwave-assisted hot air method was also lower compared to hot-air dried samples

(Askari et al. 2009). In all these studies, higher drying rates were observed at higher microwave power levels.

Microwave vacuum drying. Microwave vacuum drying combines the advantages of microwave drying (rapid heat transfer rates) and vacuum drying (lower process temperature, enhanced moisture transfer) and offers the potential of improving product quality and energy efficiency of the process (Yongsawatdigul and Gunasekaran, 1996; Drouzas and Schubert, 1996; Mousa and Farid, 2002; Meda et al. 2008; Song et al. 2009).

Some of these investigations reported that materials dried under microwave vacuum or microwave vacuum in combination with other methods exhibited higher retention rates of pigments (Lin et al. 1998; Cui et al. 2004; Yanyang et al. 2004), nutrients (Lin et al. 1998; Yanyang et al. 2004), and other bioactive substances (Cui et al. 2006). Improvements in drying rates (Sunjka et al. 2004; Yanyang et al. 2004), color (Drouzas et al. 1999), rehydration (Drouzas and Schubert 1996; Lin et al. 1998), and in other quality characteristics (Krokida and Maroulis 1999; Cui et al. 2003) relative to high temperature forced-air drying were also reported. Materials used in these investigations included apple, banana, carrot, Chinese chive leaves, cranberries, *Ganoderma lucidum* extract, garlic, pectin gels, potato, and wild cabbage.

Effect of process variables on drying kinetics and product quality characteristics were also assessed. Careful selection of these process variables is important for optimal process performance and product quality improvement. Studies on drying banana (Drouzas and Schubert 1996), Saskatoon berries (Meda et al. 2008), and potato slices (Song et al. 2009) demonstrated that drying rate was significantly influenced by microwave power level than by vacuum pressure. Chauhan and Srivastava (2009) found that although both microwave power and vacuum

significantly affected selected quality attributes of dried green peas, the effect of microwave power level on linear shrinkage, apparent density, rehydration, and green color ratios, overall acceptability, drying time, and drying efficiency was more pronounced. Meda et al. (2008) reported that total color difference observed in Saskatoon berries was dependent on microwave power and vacuum pressure levels. In cranberries, darker samples were observed at higher microwave power levels (Sunjka et al. 2004).

Drying economics. While the above-mentioned studies showed that the use of microwave energy could be an attractive alternative technology for drying biological materials, the economic implications of incorporating microwave drying technology in commercial processing operations were not explored in the above-mentioned studies. Sims (2008) reported the conduct of a three-day pilot test on microwave drying of corn WDGS. Test results were not presented in the article but some of the predicted benefits indicated include potential operating cost reduction, reduced amino acid damage, and reduced water consumption.

Disman (1966), Ishii (1974), Jolly (1976), Schiffman (2007), and Hasna (2011) presented some of the techno-economic considerations involved in evaluating the economic feasibility of incorporating microwave drying into commercial scale operations. Case studies were also presented to illustrate the application of the analytical/financial tools they provided. Some of the common cost factors identified in these studies include capital costs, efficiency of power supply, microwave generator and applicator, magnetron replacement, electricity, cooling water, and floor space requirement.

In corn ethanol production, there are already models that enable economic and/or technical assessment of corn ethanol production. These include: (i) the spreadsheet model developed and maintained by Hofstrand (2013) using assumptions typical of an Iowa plant, (ii) the descriptive

engineering spreadsheet model created by Dale and Tyner (2006a; 2006b) to better understand how plant profitability is affected by various conditions and to evaluate the costs and benefits of utilizing new or different technologies, and (iii) the process and cost model developed by Kwiatkowski et al. (2006) to evaluate the impact of existing or new technologies, alternate feedstock, and other changes on ethanol production costs. These models, however, did not have a module that specifically looked at the costs of DDGS production, particularly the drying process.

1.1.3 Chemical composition

Table 1.3 presents a comparison of the proximate composition of corn and wheat grains and their corresponding DDGS, WDG, CDS and thin stillage fractions. Wheat grain has higher protein but lower fat content compared to corn. These inherent compositional differences between the two grains could explain the compositional differences between the two DDGS types. With the removal of starch during the ethanol extraction processes, the table shows that the chemical constituents in corn and wheat DDGS were about 150-301% and 142-199%, higher than those observed in their respective whole grains. As such, distillers grain has been primarily used as an animal feed ingredient and has been documented in a number of studies over the years (Palmquist and Conrad 1982; Boila et al. 1994; Ojowi et al. 1997; Robertson 2003; Widyaratne and Zijlstra 2007; Świątkiewicz and Koreleski 2008).

The chemical composition of DDGS, however, is variable. Nuez-Ortin (2010) identified compositional differences of the feedstock used and processing conditions such as amount of solubles blended with wet distillers grain and drying as two possible sources of the nutrient profile variation in wheat DDGS samples obtained from 2 Saskatchewan ethanol plants. In corn

DDGS, Belyea et al. (2010) identified the following possible sources of variation in the chemical composition: (i) ratio of amylopectin and amylose in the feedstock; (ii) particle size distribution of the ground feedstock; (iii) fermentation process parameters; (iv) separation of whole stillage into wet distillers grain and thin stillage; (v) dewatering of thin stillage; (vi) blending of CDS and WDG; and (vii) drying of WDGs. Liu (2008) also examined the chemical composition of whole and size-fractionated corn DDGS samples and found compositional differences in the sized fractions of the material. Finer fractions tended to have higher protein content than the coarser fractions. As particle size increased, fat, total non-starch carbohydrates and total carbohydrates contents of corn DDGS also increased. In their study of particle segregation occurrence within DDGS piles and subsequent spatial nutrient variability, Clementson et al. (2009) also reported correlation between protein and moisture content with particle size of corn DDGS.

Table 1.3 Proximate composition of corn and wheat grain, distillers dried grain with solubles (DDGS), wet distillers grain (WDG), condensed distillers solubles (CDS) and thin stillage, % dry matter.

Chemical component ¹	Whole grain ²		DDGS ²		WDG		Corn CDS ³	Wheat thin stillage ⁵
	Corn	Wheat	Corn	Wheat	Corn ³	Wheat ⁴		
Moisture, % wb	11.23	10.48	8.56	6.24	62.2	70.6	67.7	91.6
Crude protein	10.13	14.28	32.01	39.32	35.0	26.4	22.4	48.5
Crude ash	1.73	2.12	4.32	5.12	2.35	2.7	11.6	8.0
Crude fat	4.59	1.91	16.53	4.98	11.3	6.6	34.4	9.6
NDF	14.47	17.22	49.46	48.07	39.8	74.9	4.9	34.5
ADF	3.66	3.68	14.68	10.99	11.9	24.1	2.92	3.4

¹NDF means neutral detergent fiber, ADF is acid detergent fiber; ²Nuez-Ortin and Yu 2009; ³Cao et al. 2009; ⁴Ojowi et al. 1997; ⁵Ojowi et al. 1996.

1.1.3.1 CDS and WDG composition

The proportion at which the WDG and the CDS streams are blended is one of the identified sources of variation in DDGS composition (Belyea et al. 2010). Table 1.3 shows the differences

in the composition of corn- and wheat-based CDS and WDG fractions. The corn-based CDS, for example, had lower protein and fiber but higher fat content compared to the WDG fraction. In contrast, wheat thin stillage, from where CDS was derived, had higher protein and fat but lower fiber content than its WDG counterpart. Differences between CDS and WDG could be attributed to the kernel components that make up the two fractions. The WDG fraction, which is more fibrous, is primarily derived from bran while the CDS, which is comprised of finer solids, could be mostly derived from the endosperm and germ components of the kernel. Considering these compositional differences between WDG and CDS within each feedstock, blending these two fractions at varying proportions would result to DDGS with differing compositions.

In corn DDGS, protein content decreased as CDS or solubles level increased (Ganesan et al. 2008a; Kingsly et al. 2010; Probst et al. 2013). Ganesan et al. (2008a) and Probst et al. (2013) conducted lab-scale studies while Kingsly et al. (2010) did a plant-scale investigation. Ganesan et al. (2008a) used “solubles” in their study, defined as the “non-water portion of CDS that passed through a filter media” while Kingsly et al. (2010) and Probst et al. (2013) utilized CDS in their investigations. Both Kingsly et al. (2010) and Probst et al. (2013) reported similar trends in lysine, methionine, threonine, and tryptophan, suggesting higher amino acid concentration in the WDG compared to the CDS fraction. Neutral and acid detergent fiber contents of corn DDGS also decreased while fat, ash, sugars, and glycerol content increased when CDS level was increased (Kingsly et al. 2010; Probst et al. 2013). Ganesan et al. (2008a) did not observe a significant trend in fat content when the level of solubles was increased.

1.1.3.2 Protein quality

Table 1.4 presents the amino acid content of wheat grain and wheat DDGS, as reported by Widyaratne and Zijlstra (2007). The lysine content of DDGS was about 38% higher than that of the wheat grain while the rest of the amino acids were 91% to 137% higher. This lower percentage increase could indicate that some of the lysine may have been lost during ethanol extraction and DDGS production processes.

Table 1.4 Amino acid composition of wheat grain and wheat DDGS, % dry matter (Widyaratne and Zilstra 2007).

Amino acid	Wheat grain	Wheat DDGS
Arginine	0.91	1.77
Cysteine	0.48	0.96
Histidine	0.46	0.99
Isoleucine	0.68	1.59
Leucine	1.31	3.01
Lysine	0.52	0.72
Methionine	0.32	0.69
Phenylalanine	0.96	2.16
Threonine	0.54	1.28
Tryptophan	0.23	0.44
Valine	0.84	1.91
Total	19.48	40.21

Despite its higher crude protein and amino acid content, wheat DDGS not only showed lower lysine content (Widyaratne and Zijlstra 2007) but also lower digestibility values (Nyachoti et al. 2005; Lan et al. 2008; Cozannet et al. 2010). Since lysine is usually the limiting essential amino acid in animal feed ingredients, its loss lowers the nutritive value of a feed material (Fayle and Gerrard 2002), ultimately affecting animal growth performance. Widyaratne and Zijlstra (2007) also indicated that lysine damage would be a constraint in the utilization of wheat DDGS as feed for monogastric animals.

Some of the ethanol production processes that may have contributed to lysine loss include liquefaction, saccharification, and fermentation (Cozannet et al. 2010; Widyaratne and Zijlstra 2008) while in DDGS production, dehydration of thin stillage into condensed distillers solubles (CDS) (Cozannet et al. 2010), addition of CDS to WDG (Nyachoti et al. 2005), and drying (Nyachoti et al. 2005; Zijlstra and Beltranena 2009) were among those identified.

Marked losses of lysine during drying and other heat treatment processes have been attributed to the Maillard reaction, which involves the binding of amino groups with the carbonyl compounds of reducing sugars (Widyaratne and Zijlstra 2008; Cozannet et al. 2010; Nyachoti et al. 2005). Among the essential amino acids, lysine is particularly vulnerable to Maillard reaction because its free ϵ -amino group can readily react with the reducing sugars (Mao et al. 1993). This bound lysine is not normally susceptible to enzymatic decomposition, thus rendering the amino acid nutritionally unavailable (Mao et al. 1993). Mavromichalis (2001) further indicated that, during the early stage of the Maillard reaction, lysine can still be detected but may no longer be biologically available. In late Maillard reaction stages, a reduction in the amount of chemically analyzed lysine was observed (Mavromichalis 2001).

The rate of the Maillard reaction depends upon temperature, time, water activity, and chemical composition, among others (Ames 1990; Owusu-Apenten 2004). Increasing temperature and exposure time tend to increase the rate of the Maillard reaction (Mavromichalis 2001; Ames 1990; Owusu-Apenten 2004). The reaction rate also depends on chemical composition (Evangelisti et al. 1999). In their study of dietetic milks, Evangelisti et al. (1999) indicated that the quality, quantity, and nature of the reducing carbohydrates present in the product could also determine the development of Maillard reaction. They found that adding ingredients that increased total reducing carbohydrates resulted in increased amount of blocked lysine

(Evangelisti et al. 1999). In corn DDGS, Kingsly et al. (2010) reported that incorporating higher levels of CDS, for example, resulted in a darker product because more reducing sugars are available to react with proteins during the drying process. The Maillard reaction also occurs less readily in substances with high water activity (Ames 1990; Lingnert 1990). When a product is subjected to high temperatures for a considerable length of time, as in drying processes, its surface dries out, producing a crust with a low water activity, favoring the Maillard reaction (Ames 1990).

Aside from lysine, acid detergent insoluble nitrogen (ADIN) or acid detergent insoluble crude protein (ADICP) and color had also been reported as among the more common indicators of heat damaged proteins. ADICP is the crude protein recovered from residue of acid detergent fiber analysis (Coblentz et al. 2010), with higher concentrations suggestive of greater heat damage (Cromwell et al 1993; Coblentz et al 2010). In a number of corn DDGS studies, color has been suggested as a quick indicator of lysine content and availability. Batal and Dale (2006) found that more yellow and lighter samples had higher total and digestible lysine levels than darker-colored corn DDGS. Similar findings were also reported by Fastinger et al. (2006) in their study of corn DDGS samples collected from 5 Midwestern US ethanol plants. They reported that the lowest lysine content and digestibility were found in the darkest DDGS samples. Cromwell et al (1993) also observed that lighter colored DDGS are more likely to have good nutritional properties.

1.1.4 Physical characteristics

Baseline information on the key physical characteristics of wheat DDGS is important in addressing the challenges posed in existing processing, handling, transportation, storage, and

utilization systems. Understanding these properties is also fundamental in seeking opportunities for process improvement and value addition. Majority of the current information available on wheat DDGS, however, is still predominantly related to its nutritional characteristics as an animal feed ingredient. Because of this lack in information, the discussion in this subsection is mostly derived from corn DDGS studies. References to studies on wheat DDGS are incorporated whenever these are available.

1.1.4.1 *Physical attributes*

Bulk density. Bulk density is an important factor to consider when determining the storage volume of transport vehicles, vessels and other containers (Knott et al. 2004). Transporting and storing low bulk density ingredients, like DDGS, are costly since these have higher spatial requirements. A few studies focusing on the physico-chemical characteristics of corn DDGS showed varied bulk density values, ranging from 391 kg·m⁻³ to 627.3 kg·m⁻³ (Knott et al. 2004; Rosentrater 2006; Bhadra et al. 2009a). In their plant-scale experiments on corn DDGS, Kingsly et al. (2010) observed that bulk density increased as CDS content and particle size increased. They attributed this to the formation of denser agglomerates as CDS content increased. Ganesan et al. (2008a) reported decreases in aerated bulk density values as moisture content increased.

Particle size and size distribution. The importance of particle size and particle size distribution in animal feed formulation, feed digestibility and nutrient availability and materials handling was highlighted in a number of studies. Seerley et al. (1988) reported the influence of particle size on animal growth performance parameters such as average daily gain and feed:gain ratio. For nursery and growing pigs, they recommended wheat particle size of 0.85 mm or larger while about 1.80 mm or larger for finishing pigs. Healy et al. (1994) indicated that a particle size of

500 μm was optimal for corn and sorghum fed to nursery pigs. Wondra et al. (1995) also indicated that a particle size of about 600 μm would be optimal for corn fed to finishing pigs, taking into consideration such factors as milling energy requirement, animal growth performance (average daily gain, average daily feed intake, gain/feed), nutrient digestibility, carcass characteristics (dressing percentage and last rib backfat thickness), and incidence of stomach ulcers and keratinization. Knott et al. (2004) outlined the effect of particle size on nutrient digestibility, feed mixing efficiency, pellet quality and palatability of diets. Clementson et al. (2009) showed that a DDGS bulk with a large particle size distribution led to particle segregation during handling, and consequently, nutrient segregation. Compositional variation due to size differences of particles making up the DDGS bulk was also illustrated by Liu (2008). Sieved DDGS fractions of different particle sizes showed that finer fractions have higher protein concentrations, lower oil and total carbohydrate contents, and lighter in color.

Particle size variability was reported in several corn DDGs studies. Bhadra et al. (2009a) reported corn DDGS particle sizes of 0.50 mm to 1.19 mm from samples obtained from five South Dakota plants. Knott et al. (2004) study of 16 ethanol plants showed particle sizes ranging from from 0.61 mm to 2.12 mm. Rausch et al. (2005) indicated particle sizes of 0.83 – 1.00 mm for their samples obtained from nine ethanol plants. Kingsly et al. (2010) attributed differences in particle sizes to variations in the amount of CDS added during drying. Particle size increased as CDS level in the blend was increased, with CDS acting as a binding agent. Ganesan et al. (2008a) noted significant differences in particle size due to solubles level and moisture content but did not observe specific trends. Rausch et al. (2005) found no significant correlation between the particle size and size distribution of ground corn and resulting corn DDGS samples in their study.

Ileleji and Rosentrater (2008) indicated blending and drying process configuration could be one of the likely causes of DDGS variability. They compared the particle size, particle size distribution, and color of the DDGS produced from older generation ethanol plants (Fig. 1.4a) with those produced from the new generation ethanol plants (Fig. 1.4b). DDGS produced from dryers in the older ethanol plants were darker in color, have larger geometric mean diameter and wider spread in its particle size distribution compared with those produced from the new generation plants.

Color. Color has been suggested in a number of corn DDGS studies as a quick indicator of lysine content and availability (Ergul et al. 2003; Batal and Dale 2006; Fastinger et al. 2006) and good nutritional properties (Cromwell et al. 1993). It is also a major factor that determines the perceived value of DDGS to purchasers (Liu 2008).

In poultry, Ergul et al. (2003) reported that there is a positive correlation between lysine digestibility and the values of the color parameters, L* (lightness) and b* (yellowness). Batal and Dale (2006) also found that more yellow and lighter samples had higher total and digestible lysine levels than darker-colored corn DDGS. Samples in their study were obtained from six midwestern US ethanol plants from 2002 to 2004. Similar findings were also reported by Fastinger et al. (2006) in their study of corn DDGS samples collected from five Midwestern US ethanol plants, where the lowest lysine content and digestibility were found in the darkest DDGS samples. Cromwell et al. (1993) also reported that lightness (color parameter L) was also highly correlated with growth rate and feed/gain, with the darkest colored corn DDGS consumed in the least amount and resulted in the poorest growth rate in swine. Liu (2008), however, did not find a strong relationship between color and nutritional quality.

In their bench-scale study, Ganesan et al. (2008a) reported that the values of the color parameters L* (brightness) and a* (redness) of corn DDGS increased with increases in soluble level. No significant difference due to moisture level was observed in both parameters. In terms of the color parameter b* (yellowness), they reported significant differences due to soluble or moisture content levels but did not observe any noticeable trend. They attributed differences in the L* and a* values to the addition of CDS. Kingsly et al. (2010) also reported increase in the L values and decrease in the a values as CDS level was decreased in their plant-scale experiments. While darkening of DDGS can be attributed partly to CDS addition, they indicated that it is most likely due to Maillard reaction. Increased CDS level in the blend is accompanied by increased amount of residual sugars, thus increasing the rate of Maillard reaction and causing DDGS to become darker.

1.1.4.2 *Flow properties*

Knowing the flow properties of wheat DDGS is important in the efficient design of bulk handling equipment/devices so that incidence of flow problems would be minimized. Peleg (1977) provided an overview of the mechanisms involved in powder flow including methods of evaluating some of the flowability properties. Carr (1965) developed a point score classification system for evaluating the flowability and floodability of dry solids, shown as Tables 1.5 and 1.6.

Carr (1965) evaluated flowability, or the ability of the material to flow steadily and consistently, using four properties: (1) angle of repose; (2) compressibility; (3) angle of spatula; and (4) uniformity coefficient. He defined the angle of repose as the angle that a pile of material makes with the horizontal, while angle of spatula as the average of the angles formed by the material with horizontal when: (i) a flat blade (spatula) is inserted into, and lifted out of, a pile of the

material, and (ii) when that blade containing the material is gently tapped. Compressibility and uniformity coefficient are calculated from measured parameters. The former is the ratio of the difference between packed and aerated bulk density values to packed bulk density while the latter is ratio of the width of sieve opening that passed 60% of the sample to width of sieve opening that passed 10% of the sample (Carr 1965). Higher values of these four properties translate to lower flowability.

Table 1.5 Point score for evaluation of flowability of dry solids (Carr 1965).

Flowability and performance	Angle of repose		Compressibility		Angle of spatula		Uniformity coef.	
	Degree	Points	%	Points	Degree	Points	Units	Points
Excellent, 90-100	25	25	5	25	25	25	1	25
Aid not needed;	26-29	24	6-9	23	26-30	24	2-4	23
Will not arch	30	22.5	10	22.5	31	22.5	5	22.5
Good, 80-89	31	22	11	22	32	22	6	22
Aid not needed;	32-34	21	12-14	21	33-37	21	7	21
Will not arch	35	20	15	20	38	20	8	20
Fair, 70-79	36	19.5	16	19.5	39	19.5	9	19
Aid not needed (but	37-39	18	17-19	18	40-44	18	10-11	18
vibrate if necessary)	40	17.5	20	17.5	45	17.5	12	17.5
Passable, 60-69	41	17	21	17	46	17	13	17
Borderline. Material	42-44	16	22-24	16	47-59	16	15-16	16
may hung up	45	15	25	15	60	15	17	15
Poor, 40 – 59	46	14.5	26	14.5	61	14.5	18	14.5
Must agitate	47-54	12	27-30	12	62-74	12	19-21	12
Vibrate	55	10	31	10	75	10	22	10
Very poor, 20-39	56	9.5	32	9.5	76	9.5	23	9.5
Agitate more	57-64	7	33-36	7	77-89	7	24-26	7
positively	65	5	37	5	90	5	27	5
Very, very poor,	66	4.5	38	4.5	91	4.5	28	4.5
0-19	67-89	2	39-45	2	92-99	2	29-35	2
Special agitator,	90	0	>45	0	>99	0	>35	0
hopper or engineering								

Table 1.6 Point score for evaluation of floodability of dry solids (Carr 1965).

Floodability and performance	Flowability		Angle of fall		Angle of difference		Dispersibility	
	Pts. from Table 1.5	Points	Degree	Points	Degree	Points	Units	Points
80-100 Very floodable Positive rotary seal will be necessary	60+	25	10	25	30+	25	50+	25
	59-56	24	11-19	24	29-28	24	49-44	24
	55	22.5	20	22.5	27	22.5	43	22.5
	54	22	21	22	26	22	42	22
	53-50	21	22-24	21	25	21	41-36	21
60-79 Floodable Rotary seal will be necessary	49	20	25	20	24	20	35	20
	48	19.5	26	19.5	23	19.5	34	19.5
	47-45	18	27-29	18	22-20	18	33-29	18
	44	17.5	30	17.5	19	17.5	28	17.5
	43	17	31	17	18	17	27	17
40-59 Inclined to flood, Rotary seal is desirable	42-40	16	32-39	16	17-16	16	26-21	16
	39	15	40	15	15	15	20	15
	38	14.5	41	14.5	14	14.5	19	14.5
	37.34	12	42-49	12	13-11	12	18-11	12
25 -39 Could flood Rotary seal probably needed depending on drop, velocity	33	10	50	10	10	10	10	10
	32	9.5	51	9.5	9	9.5	9	9.5
	31-29	8	52-56	8	8	8	8	8
0-24 Won't flood Rotary seal will not be needed	28	6.25	57	6.25	7	6.25	7	6.25
	27	6	58	6	6	6	6	6
	26-23	3	59-64	3	5-1	3	5-1	3
	<23	0	>64	0	0	0	0	0

To obtain the flowability classification of a sample, each property measurement was assigned an index value, ranging from 0 to 25, based on the point score classification system (Table 1.5). The individual index values of angle of repose, compressibility, angle of spatula, and uniformity coefficient are then summed to obtain the flowability index, which ranged from 0 (very, very poor flowability) to 100 (excellent flowability).

Carr (1965) further evaluated floodability, or a material's "tendency to liquid-like flow", using four properties: (1) flowability index; (2) angle of fall; (3) angle of difference; and (4) dispersibility (Carr 1965). Angle of fall was defined as the new angle of repose formed after a pile of material on a flat surface was jarred while the angle of difference is the numerical difference between the angle of repose and the angle of fall (Carr 1965). Dispersibility refers to the percentage of material that had been dispersed or lost during a fall and is determined by dropping a 10 gram sample through a plastic cylinder from a fixed height onto a watch glass and measuring the amount collected on the watch glass (Carr 1965). Higher values of the flowability index, angle of difference, and dispersibility indicate higher floodability. The floodability index was determined using Table 1.6, following the same procedure employed in flowability index determination.

In corn DDGS, Bhadra et al. (2009a) reported a flowability index of 79.30–82.40 for samples obtained from five ethanol plants, indicating that it has good flowability but may sometimes need flow promoting devices, like a vibrator or bin agitator, to ensure its flow. In terms of floodability, the reported index values ranged from 48.60 to 70.20, falling within the "inclined to flood" and "floodable" categories in Table 1.5. Ganesan et al. (2008a) also evaluated the flowability of corn DDGS at varying soluble and moisture content levels. They reported that the flowability index was affected by both soluble and moisture content levels. Flowability index generally decreased with increases in moisture content. Both studies used a Hosokawa powder characteristics tester which employed the methods developed by Carr (1965).

1.1.4.3 *Compression characteristics*

Extensive discussions on the fundamental principles of pelleting for animal feed from an engineering perspective had been provided by Thomas and van der Poel (1996) and Thomas et al. (1997; 1998). Granulation of animal feed provides better flow properties necessary in materials handling, facilitates better spatial utilization of storage and transportation capacities because of higher bulk density and eliminates particle/nutrient segregation since pellet composition remains fixed during materials handling (Thomas and van der Poel 1996).

In wheat DDGS, there were two studies that focused on evaluating the effect of pelleting conditions on the bulk density and durability of pellets. None focused on compression characteristics. Opoku et al. (2009) assessed the effect of die diameter (6.4 mm, 7.9 mm) and steam conditioning on the durability of wheat DDGS pellets. Pellets produced from 6.4 mm die with steam addition gave the highest bulk density (46% increase from original, unpelleted DDGS value) and durability (93%) values. Tumuluru et al. (2010) also studied the effect of process variables on the characteristics of wheat DDGS pellets produced from a single pelleting machine and a pilot-scale mill. Under the single pelleting machine, maximum pellet density and durability were obtained when the die temperature was about 50-80°C and feed moisture content of about 5% (wb). Using the pilot-scale mill, maximum pellet density and durability was also obtained using the 6.4 mm die with steam addition.

In corn DDGS, Rosentrater and Kongar (2009) developed MS Excel-based computer model to examine how pelleting impacts on shipping costs. DDGS production rates, pelleting rates, and DDGS sale prices were varied at 100-1000 tonnes per day, 0 – 100%, and US\$ 50 – US\$ 200 per tonne, respectively. In general, they found that as the proportion of pelleting increased,

transportation cost declined because rail car load limits are maximized with increased bulk density of pellets. Transporting the entire DDGS bulk in pelleted form, for example, reduced shipping cost by 89% when the selling price of $\$50 \cdot t^{-1}$ was used.

1.1.4.4 *Moisture relationships*

Moisture content is an important property affecting DDGS handling and storage. Aside from strongly influencing DDGS shelf life during storage, it also significantly affects the physical properties of DDGS. Ganesan et al. (2008a), for example, reported moisture content affected the bulk density, compressibility, and flowability index of corn DDGS.

Wheat DDGS is subjected to varying environmental conditions during handling and storage and like any hygroscopic material, it can gain from or lose moisture to its surrounding environment. Determining the sorption characteristics is essential in maintaining its quality during handling and storage.

Ganesan et al. (2008b) studied the sorption isotherm characteristics of corn DDGS with varying solubles level (10%, 15%, 20% and 25% dry basis) using the static gravimetric method at 10°C, 20°C, 30°C and 40°C over four equilibrium relative humidity levels of 60%, 70%, 80% and 90%. They found that solubles level affected the moisture sorption characteristics of corn DDGS and introduced this as a factor in the modified Halsey equation. They termed this new model as the Ganesan-Muthu-Rosentrater (GMR) model, shown here as Eq. 1.1:

$$M = \left[\frac{-\exp(F T_c + S^G)}{\ln(RH)} \right]^{\frac{1}{H}} \quad 1.1$$

where M = equilibrium moisture content, dry basis (decimal), RH = equilibrium relative humidity (decimal), T_c = temperature, °C, S = soluble level, % db, and F, G, H = empirical coefficients.

Kingsly and Ileleji (2009) also studied the effect of the process variables on the moisture sorption characteristics of corn DDGS. They found that the equilibrium moisture content of DDGS samples decreased when the CDS level was lowered. They also proposed a modified four-component model (Eq. 1.2) for the prediction of corn DDGS sorption characteristics using chemical composition of protein, sugar, minerals, starch, fiber, and glycerol.

$$X_c = X_v + W_f X_f + W_s X_s + W_p X_p + W_g X_g \quad 1.2$$

where X is the equilibrium moisture content (db, decimal); W is the weight fraction of the component (db, decimal); c is the total (combined effect of all components); v is the vacuole (sugars and minerals); f is the fiber (cellulose); s is the starch; p is the protein; and g is glycerol.

1.1.5 Summary

This background highlights the need to establish baseline information on the physico-chemical, and drying characteristics of wheat DDGS, considering the limited published literature focusing on the subject. Information generated could assist in addressing specific challenges of existing supply chains, such as inefficient drying process, protein damage, inconsistent product quality, and high logistics cost.

The CDS:WDG blending proportion seemed to be one of the central influences of the observed variation in the physical and chemical characteristics of the resulting DDGS. While this has

been tackled in a number of corn DDGS investigations, this has remained currently unexplored in wheat DDGS.

1.2 Research Objectives

The main objectives of this PhD project were: (i) to evaluate alternative methods of drying wheat distillers grain with solubles that would minimize protein damage, energy consumption, and associated costs; (ii) to describe the physico-chemical characteristics of wheat distillers dried grain with solubles; and (iii) to assess the effect of drying methods/conditions and the blending process on the physical and chemical characteristics of wheat DDGS. Specifically, it aimed to:

1. Describe the physico-chemical characteristics of commercially available wheat DDGS;
2. Determine the drying characteristics of wheat wet distillers grain with solubles under forced air convection, microwave-, microwave convection, and microwave vacuum methods and conditions;
3. Assess the effect of drying methods/condition and condensed distillers solubles and wet distillers grain blending proportion on protein quality, proximate composition, and physical properties of wheat DDGS;
4. Evaluate the economic feasibility of incorporating a microwave- or microwave-assisted drying technology in Saskatchewan ethanol plants for wheat DDGS production.

The secondary aim of this PhD project was to apply the knowledge and skills acquired from research experience and academic training in addressing a related issue/need under the conditions and realities of a developing country, i.e. the Philippines.

1.3 Organization of the Thesis

This thesis is organized and formatted according to the guidelines for manuscript-style theses of the College of Graduate Studies and Research at the University of Saskatchewan. It has ten chapters, seven of which are research manuscripts. The manuscripts presented in Chapters 2, 3, 4, and 5 have been published in peer-reviewed journals. The manuscript in Chapter 6 has been accepted for journal publication with minor revisions while those in Chapters 7 and 8 are yet to be reviewed. Within each of these seven manuscripts, transition sections on “Contribution of the Ph.D. Candidate” and “Contribution of the Paper to the Overall Study” are incorporated.

The remaining three chapters include this introductory chapter, a chapter which summarized the main findings of this study (Chapter 9), and one on the general conclusions and recommendations for future research (Chapter 10). A list of references is provided after Chapter 10.

The Appendix contains a paper on microwave vacuum drying of wheat distillers grain with solubles (Appendix A), sample calculations for the techno-economic evaluation presented in Chapter 7 (Appendix B), supplementary information for the case study application in Chapter 8 (Appendix C), and email permission from two companies on the use of selected diagrams (Appendix D).

1.4 Manuscript Content of the Thesis

Six of the seven manuscripts were focused on three major areas: (i) evaluation of drying technologies for wheat distillers grain with solubles, (ii) physico-chemical characterization of the co-product, and (iii) evaluation of the effect of blending proportion and drying method on the

physical and chemical properties of wheat DDGS. Chapter 2 determined the drying characteristics of ethanol plant-sourced wheat distillers grain with solubles under three methods: forced air convection, microwave, and microwave convection and how these laboratory-scale drying methods affected lysine content and color of the dried product. Chapter 3 evaluated the effect of condensed distillers solubles (CDS): wet distillers grain (WDG) blending proportion and drying method on protein quality of wheat DDGS. Chapters 4 and 5 assessed the effect of CDS:WDG blending proportion and drying method on proximate composition, physical attributes, flow properties, compression characteristics, frictional properties, and thermal properties. Chapter 4 focused on forced air convection drying while chapter 5 concentrated on microwave and microwave convection drying methods. Chapter 6 described the physico-chemical characteristics of ethanol-plant sourced wheat DDGS samples. The effect of moisture content on the physical properties was also assessed. A techno-economic evaluation was performed in Chapter 7 to assess the feasibility of incorporating microwave technology in drying the wet distillers grain with solubles in Saskatchewan ethanol plants. The evaluation was limited to the drying process and compared the cost of drying under microwave- and combined hot air and microwave methods with the cost associated with conventional hot air drying and with historical market prices. The last manuscript (Chapter 8) is a case study implemented in the Philippines, intended to apply the student's knowledge and skills acquired in the PhD program and aimed toward improving the productivity of backyard animal production.

Chapter 2

Drying characteristics and lysine content of wheat distillers grain with solubles under three drying methods

A similar version of this chapter has been published in the *Drying Technology* journal:

- Mosqueda, M. R. and L.G. Tabil. 2011. Drying characteristics and lysine content of wheat distillers grain with solubles under three drying methods. *Drying Technology* 29(7): 797-807.

Contribution of the PhD Candidate

The PhD candidate conducted literature review, planned and executed the drying experiments, analyzed the data sets, and prepared the manuscript for this initial investigation on the drying characteristics of wheat distillers grain with solubles. Her research supervisor, Lope G. Tabil, provided guidance during planning of experiments and editorial advice during manuscript preparation. An external laboratory in Saskatoon was engaged for lysine analysis since University of Saskatchewan laboratories did not have the capability to perform the test.

Contribution of the Paper to the Overall Study

This paper addressed, in part, two of the main objectives of the study: (i) evaluation of alternative drying methods for wheat distillers grain with solubles that would minimize protein damage, energy consumption, and associated costs; and (ii) determination of the physico-chemical characteristics of the dried product. It described the thin layer drying characteristics of

wheat wet distillers grain with solubles (WDGS) under three methods (forced air, microwave, and microwave convection) and evaluated the effect of drying temperature, microwave power level, and microwave convection setting on the lysine content and color of wheat DDGS samples. There is still very limited information about these characteristics for wheat DDGS.

The results of this study would be useful, as reference material, to ethanol plant managers and quality assurance personnel as well as to animal feed processors interested in examining their processes to improve the nutritive the nutritive quality of wheat DDGS. Although the drying conditions employed in the study were milder compared to those found in commercial operations, the study was still able to demonstrate the effect of these milder drying conditions on lysine content and color of the co-product. These results might also be of interest to microwave drying system manufacturers investigating on the potential use of their equipment in drying wheat WDGS.

2.1 Abstract

The drying characteristics and lysine content of wheat distillers grain with solubles were studied under three methods: forced air drying, microwave drying, and microwave convective drying. For forced air drying, temperature was set at five levels (40, 60, 80, 100, and 120°C), maintaining air velocity and relative humidity at 0.7–0.8 m/s and less than 8%, respectively. Four power levels (40, 60, 80, and 100%) and four combination settings (130°C–30% power, 150°C–30% power, 160°C–30% power, and 190°C–30% power) of a domestic microwave oven were used for microwave- and microwave convection drying. Experimental data were fitted to four common thin-layer drying models, and the Page model was found to best describe the drying behavior of the distillers grain under the three methods. Lysine content and the color parameters

(L, a, and b) were also determined and assessed for linear correlation with temperature and microwave power. Lysine content and L values decreased with increases in drying air temperature. Lighter colored distillers dried grains with solubles (DDGS) samples tended to have higher lysine content. Microwave-dried samples did not show significant differences in lysine content and in L values across the four power levels.

2.2 Introduction

The use of wheat as feedstock for ethanol production is expanding in Saskatchewan, Canada, with four of its five existing ethanol plants started operations within the last 4 years (University of Saskatchewan 2009). With this current expansion in Saskatchewan and in nearby provinces as well, wheat distillers grain, a co-product of ethanol production, is increasingly becoming more available in western Canada. It has been estimated that the five Saskatchewan plants collectively produce close to 345 million liters of ethanol and about 330,000 t of distillers dried grains with solubles (DDGS) per year (University of Saskatchewan 2009).

The use of distillers grain with solubles, whether in wet or dry form, as an economical partial replacement for corn, barley, soybean meal and inorganic phosphorus in animal feeds has been documented in a number of studies over the years (Robertson 2003; Widyaratne and Zijlstra 2007; Świętkiewicz and Koreleski 2008; McKinnon and Walker 2008; Martinez-Amezcuca and Parsons 2007). Ojowi et al. (1997) showed that the wheat-based wet distillers grains can be used effectively as an energy and protein source for growing and finishing cattle. McKinnon and Walker (2008) and Gibb et al. (2008) reported no adverse effects of the controlled incorporation of wheat DDGS on animal diets as partial replacement for barley grain. Gibb et al. (2008) further reported that the inclusion of wheat DDGS in feedlot diets of British cross heifers had no

significant impact on carcass traits like weight, dressing percentage, backfat thickness and incidence of abscessed livers. In their evaluation of feedlot cattle fed with corn and wheat DDGS (at 20% and 40%, respectively) and with the barley-based control diet, Aldai et al. (2009) also observed no striking difference between the three diets and indicated that feeding cattle with wheat DDGS had no negative effects on the meat quality characteristics.

Wet distillers grains, however, are easily spoiled and are difficult to transport without drying. While drying does improve the shelf life and transportability of these grains, it presents some concerns to both fuel ethanol and animal feed producers. For instance, traditional drying operations, such as the use of heated air in rotary drum dryers, required significant amount of energy to operate (Murphy and Power 2008; Tang and Cenkowski 2001; Tang et al. 2005). It has been estimated that the drying process accounted for about one-third of the total energy consumption of ethanol plants (Murphy and Power 2008). Further, excessive heating during drying also reduces the availability and digestibility of lysine (Widyaratne and Zijlstra 2007; Świątkiewicz and Koreleski 2008; Lan et al. 2008; Schutz et al. 2006; Batal and Dale 2006; Fastinger et al. 2006), one of the essential amino acids in animal diet. Some studies on corn DDGS have suggested the use of color as a quick and reliable indicator of lysine availability and digestibility, with darker colored corn DDGS samples indicating lower lysine availability and digestibility (Batal and Dale 2006; Fastinger et al. 2006). Alternative energy saving drying technologies which also minimize the negative impact on the DDGS nutrient composition, therefore, needs to be investigated.

The use of superheated steam and microwave energy, as alternatives to the conventional hot air drying, have already been investigated in a number of studies. Superheated steam drying offers a number of potential advantages, including reduced energy consumption if the exhausted steam is

recovered and used elsewhere, higher drying rates, no fire and explosion hazards, and product pasteurization, sterilization and deodorization (Mujumdar 2007). Drying characteristics in superheated steam, as well as the accompanying changes in some of the quality parameters, of shrimp (Prachayawarakorn et al. 2002), potato (Iyota et al. 2001), Asian noodles (Pronyk et al. 2008), sugar beet pulp (Tang et al. 2000) and oil palm fibers (Hasibuan and Wan Daud 2009) in superheated steam have already been studied. A few studies have also investigated its use in drying spent grains. For example, Stroem et al. (2009) studied the rotary superheated steam drying of brewer's spent grains (BSG) at varying feed rate (14 and 23 kg·h⁻¹), steam temperature (200 and 240°C), and steam velocity (1 and 2.5 m·s⁻¹). They reported that the energy consumption per kg of water removed (with steam recovery) was 0.65 – 1.47 MJ when BSG moisture was reduced from 72% to a range between 8.8% and 59%. Hot air dryers typically consumed between 4 – 6 MJ·kg⁻¹ of water removed (Mujumdar 2007; Stroem et al. 2009). Zielinska et al. (2009) studied the drying kinetics of distillers spent grain spread on a solid Teflon sphere using superheated steam temperatures of 110 to 160°C. They reported effective diffusivity values of 3.24 x 10⁻¹⁰ to 2.4 x 10⁻⁹ m²·s⁻¹ and drying rates of 0.02 to 0.83 min⁻¹ during the initial phase of drying. Further, Tang et al. (2005) and Pronyk et al. (2004) also studied the effect of superheated steam drying conditions on the nutrient components of brewer's and distillers spent grains. They reported that except for starch, the other nutrient components (β -glucan, pentosan and protein) were not significantly affected with varying steam temperature (110° – 180°C) and drying time (3, 6, and 11.5 min for brewer's spent grain; 4.5, 9 and 17 min distillers spent grain). The researchers, however, indicated that while some protein denaturation may have occurred, this was not reflected in the average protein content measurements (Tang et al. 2005; Pronyk et al. 2004). Tang et al. (2005) also developed a modified Page equation to

describe the thin layer drying characteristics of the brewer's and distillers spent grains under superheated steam drying at varying steam temperature (110° – 180°C) and velocity (0.28 – 1.15 m·s⁻¹).

The use of microwave energy is also increasingly becoming an attractive alternative to the conventional heated air drying of biological materials. Unlike hot air drying where internal heat penetration is slow, energy transfer rates in microwave drying are much higher as microwaves are primarily absorbed by water and ions in the product, leading to volumetric heating as well as faster heating of moist areas than the drier ones (Datta 2001). Microwave energy, either singly or in combination with vacuum and/or hot air drying, has been employed in investigating the drying characteristics of many biological products, such macademia nuts (Chung and Furutan 1989), parsley (Sosyal 2004), wood strands (Du et al. 2005), spinach (Ozkan et al. 2007), chard leaves (Alibas 2006), apples (Funebo and Ohlsson 1998; Feng and Tang 1998), mushroom (Funebo and Ohlsson 1998), banana (Maskan 2000), kiwifruits (Maskan 2001), okra (Dadali et al. 2007), wheat (Walde et al. 2002), saskatoon berries (Reddy 2008), and flax fiber (Tripathy 2009). Microwave-assisted drying method offers shorter drying times, without significant loss of product quality with proper selection of process variables (Chung and Furutan 1989; Sosyal 2004; Du et al. 2005). In drying carrot slices and Chinese chive leaves, for example, microwave-assisted drying methods (microwave-vacuum, microwave-vacuum + air, microwave-vacuum + vacuum) produced samples with higher total chlorophyll and carotenoid retention compared to those dried using the conventional hot air method (Cui et al. 2004). Vacuum-microwave and combined air and vacuum-microwave drying also produced dehydrated Saskatoon berries with higher total phenolics and anthocyanin content compared to the air drying method (Kwok et al. 2004). In dehydrating garlic cloves, combined microwave-hot air drying produced more superior

quality samples, in terms of color and flavor strength, compared to hot air drying (Sharma and Prasad 2001). Color degradation in apple, strawberry, tomato and mushroom dried under the microwave-assisted hot air method was also lower compared to hot-air dried samples (Askari et al. 2009). In drying parsley, Soysal (2004) further observed that microwave drying was able to maintain a good green color in the dried flakes, comparable to those in fresh leaves.

There is still, however, a very limited number of published studies on drying distillers grain using microwave energy. The objectives of this study, then, were: (1) to determine the thin layer drying characteristics of wheat distillers grain with solubles under three methods: forced air, microwave, microwave-convection drying; (2) to compare the energy consumption of these three drying methods; (3) to determine the effect of the different drying conditions on lysine content and on the color of wheat DDGS samples; and (4) to verify whether there is a correlation between lysine content and color of wheat DDGS.

2.3 Materials and Methods

Wet distillers grains with solubles (WDGS) were obtained from a southern Saskatchewan wheat ethanol processing plant in October 2009. The WDGS were placed in sealed plastic buckets and were stored in a -13°C freezer until these were used. Before the start of a drying experiment, a WDGS bucket was removed from the freezer and was left in the sample preparation area at ambient room temperature (22°C) for at least 24 h to allow thawing. Samples obtained from the bucket were placed in plastic bags and left in the sample preparation area for equilibration with room temperature (22°C).

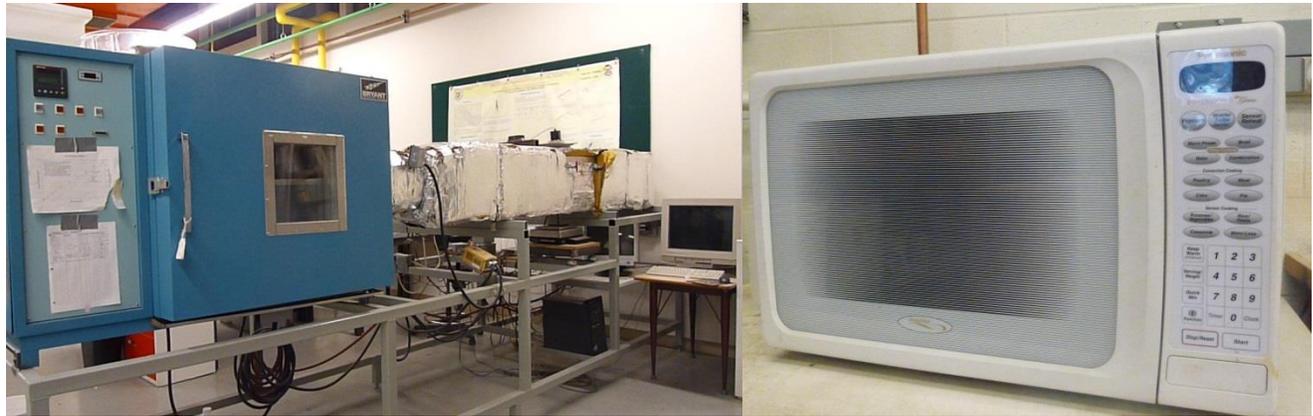
2.3.1 Forced air convection drying

An air recirculating drying unit (Fig. 2.1a) was used to determine the drying characteristics of wheat WDGS. It consisted of an air conditioning unit equipped with humidity and temperature sensors, a vane-axial circulating fan, a drying chamber with three scale-mounted trays, and a duct system. The unit was connected to a computer equipped with LabView 8.2 (National Instruments, Austin, TX) for data capture. Detailed description of the system set-up is provided by Kashaninejad and Tabil (Kashaninejad and Tabil 2004) and Kashaninejad et al. (2007). Five drying air temperature settings (40°C, 60°C, 80°C, 100°C and 120°C) were used, with air velocity and relative humidity maintained between 0.7 – 0.8 m·s⁻¹ and below 8%, respectively. Desired test conditions were verified at least one hour before the start of each drying experiment. One hundred grams of the sample were thinly spread on a tray and were placed inside the drying chamber until the moisture reached about 8 - 12% (wet basis). Two repeat runs for each drying condition were conducted.

2.3.2 Microwave drying

Drying runs were conducted, in duplicates, using a Panasonic® microwave/convection oven (model NNC980W, Panasonic Canada, Ltd., Missisauga, ON) (Fig. 2.1b) at four power levels: 40%, 60%, 80%, and 100% microwave power (Matsushita Electric Industrial Co., Ltd. 2000). About one hundred grams of sample were thinly spread on a piece of parchment paper and placed on top of the 0.38 m glass turntable inside the oven cavity. The turntable containing the sample was removed from the oven every 2 min during the drying period and weighed. Drying continued until the moisture content of the sample reached about 8 - 12% (wet basis). Periodic removal of the sample to measure mass loss during microwave drying has been employed in a

number of studies (Chung and Furutan 1989; Sosial 2004; Alibas 2006; Maskan 2000; Maskan 2001).



(a)

(b)

Fig 2.1 The drying units used for the forced-air convection (a) and microwave and microwave convection (b) methods.

2.3.3 Microwave convective drying

This third method utilized a combination of microwave energy and convective heat in drying the sample using the same Panasonic microwave/convection oven. To measure mass of the sample without taking it out from the oven, a polypropylene tray was used as sample holder, instead of the glass turntable (Reddy 2008; Tripathy 2009). Inside the oven cavity, the tray was suspended from a weighing scale using a Teflon thread. The scale was connected to a computer to facilitate data acquisition every 2 s.

About twenty five grams of sample were thinly spread over a parchment paper on the tray and were dried at four combination settings until the target moisture content of 8 – 12% (wet basis) is reached. Three drying runs were conducted at each of the four combination settings: 130°C-30% power, 150°C-30% power, 160°C-30% power, and 190°C-30% power (Matsushita Electric

Industrial Co., Ltd. 2000). Temperature profile for these settings, measured above the surface of the wheat WDGS to be dried, is shown in Fig. 2.2.

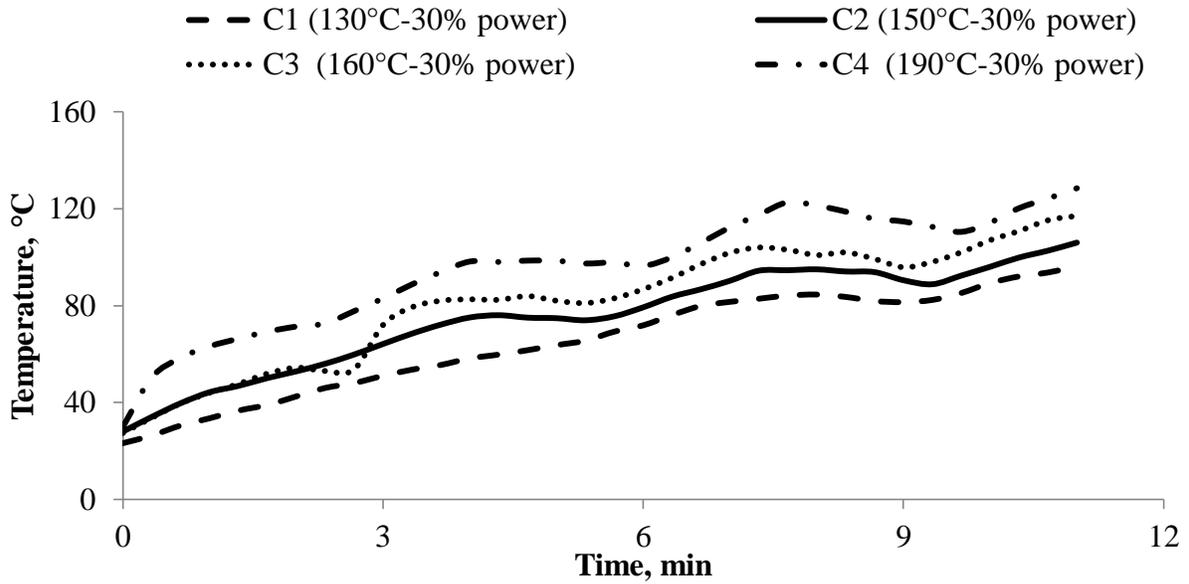


Fig 2.2 Temperature profiles at different microwave convection settings (C1, C2, C3, C4), measured above the surface of the wheat distillers grain with solubles to be dried.

2.3.4 Power output estimation

Power requirements for the forced air thin layer drying was estimated from the nameplate power requirements of the air conditioning unit, vane axial fan motor and the electronic weighing scale used.

In estimating the power output at each of the microwave settings, the method employed by Reddy (2008), Tripathy (2009), and Chung and Furutan (1989) was used as basis. Two glass beakers, containing about 1 kg of distilled water each, were heated inside the oven for 5 minutes. Temperature rise of water and of the beakers was used to calculate the power output using the following equation:

$$Q = \frac{m c_p \Delta T}{t_h} \quad (2.1)$$

where m = mass of substance (kg), c_p = specific heat capacity ($\text{J kg}^{-1} \text{ }^\circ\text{C}^{-1}$), ΔT = temperature difference ($^\circ\text{C}$), before and after heating, and t_h = time of heating (s).

The time of half response was used in comparing the estimated power consumption of the different drying methods (Kashaninejad et al. 2007). Time of half response is the time required by the sample to reach a moisture ratio equal to 0.5 or the time required to remove half of the initial moisture of the sample (Kashaninejad et al. 2007).

2.3.5 Moisture content determination

Moisture content of samples before and after drying was determined using a laboratory oven. Two to three grams of wet samples were dried at 130°C for 3 h (American Association of Cereal Chemists 1995) while the moisture content of dried samples was determined at 103°C for 24 h (American Society of Agricultural Engineers 2008).

Equilibrium moisture content (EMC) for each of these drying conditions was determined experimentally using the same methods previously described above. Samples for EMC determination were exposed to the same drying conditions until mass loss ceased. Three runs were conducted under each drying condition. The EMC was used in calculating the moisture ratio (MR),

$$MR = \frac{M_t - M_e}{M_i - M_e} \quad (2.2)$$

where M_t = moisture content, dry basis, at any time t , M_i = initial moisture content, dry basis, and M_e = EMC, dry basis.

2.3.6 Drying models

Four thin layer drying models (exponential, Henderson and Pabis, Page and Thompson), shown in Table 2.1, were investigated for their suitability in describing the behavior of wheat DGS under the three drying methods. Bruce (1985) used the exponential model as one of the models to describe exposed thin layer drying of barley. Although another model was able to better describe the barley drying curves, the exponential model was still useful for its simplicity and computational speed (Bruce 1985). This one-parameter model adequately described the drying behavior of kiwifruits under hot-air and microwave drying methods (Maskan 2001), as well as that of banana during the microwave/air and microwave finish drying (Maskan 2000). The drying of grape seeds, waste products from wine and juice processing, was also well-described by the exponential model (Roberts et al. 2008). The Henderson and Pabis model assumed that moisture movement in small grains during thin layer drying occurs by diffusion and that all resistance to mass transfer is at the outer surface of the kernel (Watson and Bhargava 1974). The model corresponds to the first term of a general series solution of Fick's second law (Kahveci and Cihan 2007). Page model was used by Misra and Brooker (1980) to model the thin layer drying of shelled corn. It was also found to adequately describe the forced air thin layer drying kinetics of purslane (Kashaninejad and Tabil 2004) and flax fiber (Tripathy 2009) as well as microwave drying characteristics of parsley (Sosyal 2004), okra (Dadali et al. 2007) and spinach (Ozkan et al. 2007). Lastly, the Thompson model, a second-order exponential curve, has been suggested to represent the drying behavior of shelled corn within the 60° - 150°C temperature range (Thompson et al. 1968).

Table 2.1 Thin layer drying models* fitted to experimental data.

Name	Model	Reference
Exponential	$MR = \exp(-kt)$	Bruce (1985)
Henderson and Pabis	$MR = g \exp(-kt)$	Watson and Bhargava (1974)
Page	$MR = \exp(-kt^n)$	Bruce (1985); Misra and Brooker (1980)
Thompson	$t = g \ln(MR) + h (\ln(MR))^2$	Thompson et al. (1968)

*MR – moisture ratio; k – drying rate constant (min^{-1}); t – drying time, min; g, h, n are model parameters

Experimental data generated were fitted to these models using SPSS 14.0 for Windows (SPSS Inc., Chicago, IL). Coefficient of determination (R^2) and the mean square error (MSE) were used to determine goodness of fit at the 0.05 significance level. R^2 indicates the proportion of the variability in the experimental data set that is explained by the drying model while MSE measures the average of the squared differences between the observed and the drying model-predicted values. Thus, the model with the highest R^2 and lowest MSE would best describe the drying characteristics of wheat DDGS.

2.3.7 Lysine content and color measurement

The color parameters (Hunter L , a , and b) of the wheat distillers grain with solubles before and after drying were determined using the HunterLab spectrophotometer (Hunter Associates Laboratory Inc., Reston, VA) in two duplicate runs.

Wheat DDGS generated from the above drying runs as well as two samples of wheat WDGS were sent to the laboratory of MCN BioProducts, Inc. (Saskatoon, SK, Canada) for amino acid analysis using the Waters AccQ•Tag method (D. Culbert, Senior Research Assistant, personal communication, 13 January 2010). The method involves hydrolysis and dilution, derivatization of amino acids, and subsequent separation and analysis using an HPLC system. AccQ•Fluor™

derivatization buffer and AccQ•Fluor reagent (Waters Corporation, Milford, MA) were used during the derivatization step (Waters Corporation 1996). Two duplicate tests were made from each sample and the average lysine content was reported.

The relationship between drying temperature and wheat DDGS color, between drying temperature and lysine content, and between lysine content and wheat DDGS color were assessed through scatter plots and regression and correlation analyses using MS Excel 2007 (Microsoft Corporation, Redmond, WA) and SPSS 14.0 for Windows (SPSS Inc., Chicago, IL).

2.4 Results and Discussion

2.4.1 Drying characteristics

Figures 2.3, 2.4 and 2.5 show the changes in moisture ratio of wheat distillers grain with solubles with drying time under forced air, microwave, and microwave convection methods. Initial wheat WDGS moisture of $2.33 \text{ kg}\cdot\text{kg}^{-1} \text{ db}$ (standard deviation = $0.11 \text{ kg}\cdot\text{kg}^{-1} \text{ db}$) and the EMC values shown in Table 2.2 were used in developing these drying curves. Increases in drying air temperature, microwave power, or microwave-convection settings decreased drying times. Wheat WDGS samples dried under the 40°C forced air method, for example, took an average of about 60 min (standard deviation = 0.24 min) to reach $\text{MR} = 0.5$. When drying air temperature was increased from 40°C to 120°C , average drying time decreased by 76% to achieve the same moisture ratio. As expected, the microwave and microwave convection methods provided shorter drying times compared to forced air convection method. To reach $\text{MR} = 0.5$ under the microwave convection method, average drying times at the 130°C -30% power and 190°C -30% power combination settings were 4.28 min (standard deviation = 0.34 min) and 3.32 min

(standard deviation = 0.13 min), respectively. Further, samples dried under the forced air method have longer drying times compared to those dried under the two microwave-assisted methods. Wheat WDGS samples, for example, took about 34 min (standard deviation = 0.9 min) to dry to about 8% (wet basis), while those dried at 40% microwave power, took an average of 14 min only. Moreover, a constant rate period was apparent in forced air drying at all temperature settings while none was observed during the microwave- and microwave-convection drying.

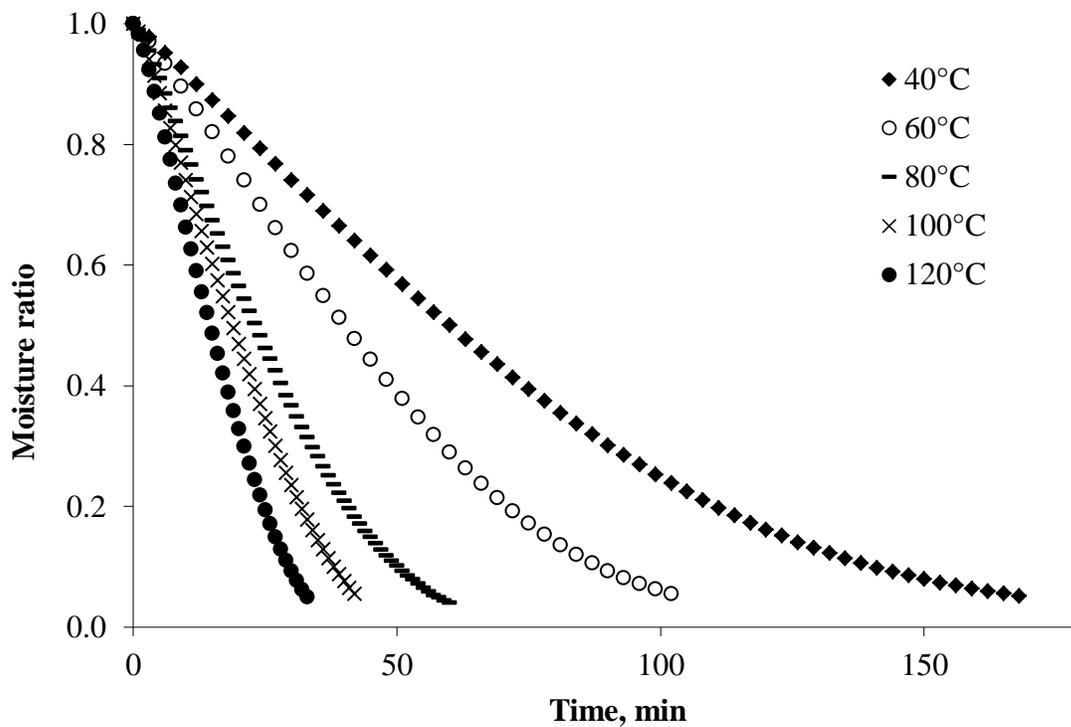


Fig 2.3 Effect of temperature setting on moisture ratio during forced air convection drying of wheat distillers grain with solubles. Values averaged from two drying runs.

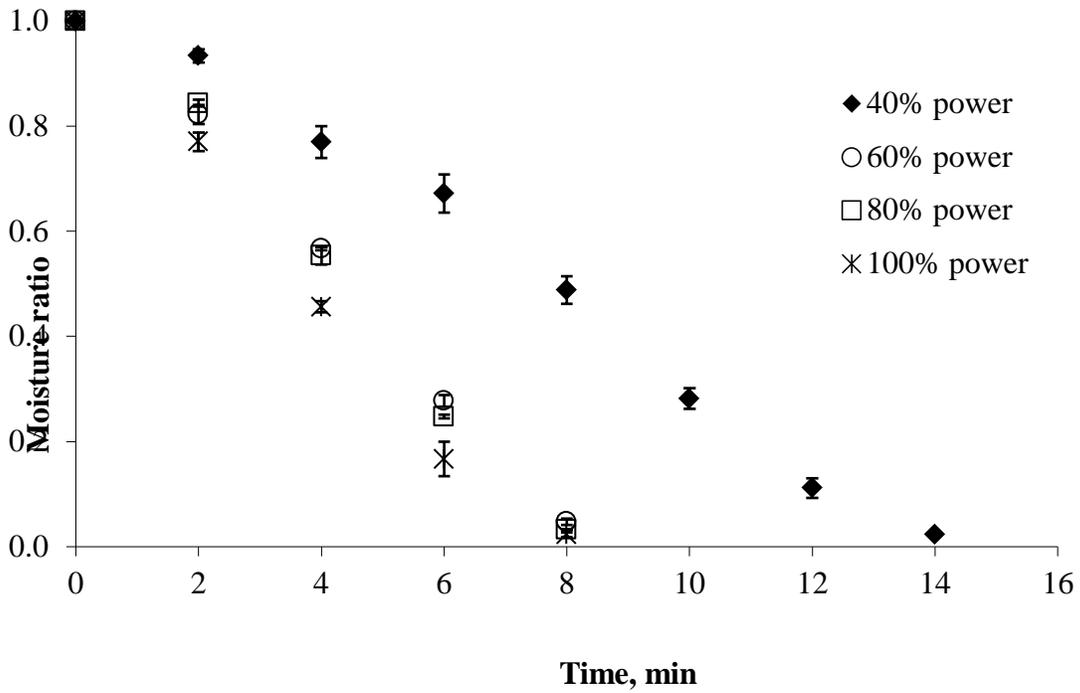


Fig 2.4 Effect of microwave power setting on moisture ratio during microwave drying of wheat distillers grain with solubles. Values were averaged from two drying runs.

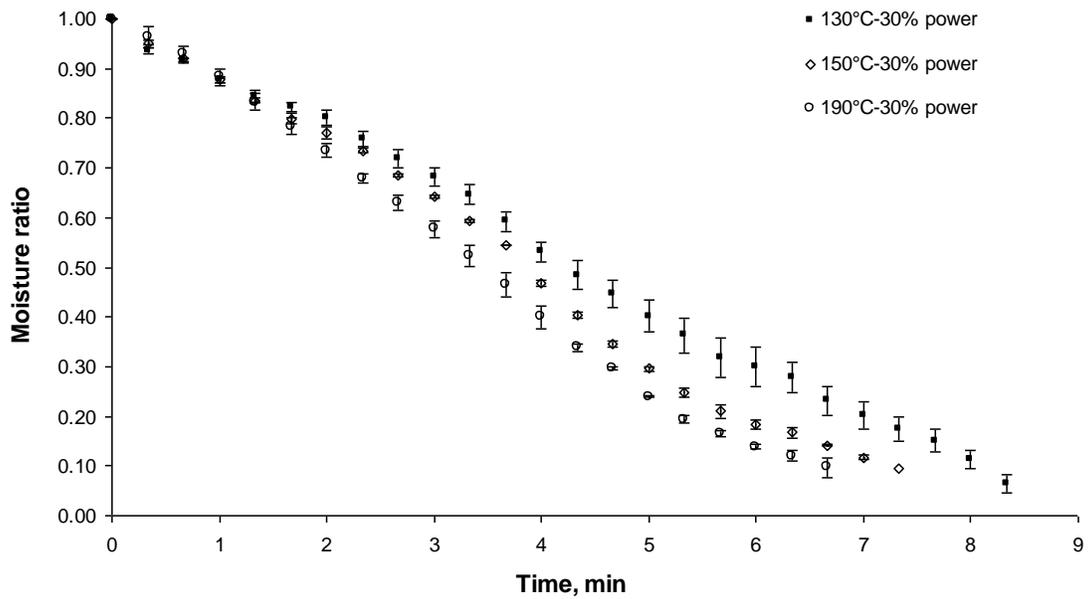


Fig 2.5 Effect of combination setting on moisture ratio during microwave convection drying of wheat distiller's grain with solubles (average of three runs).

Table 2.2 Equilibrium moisture content of wheat distillers dried grain with solubles (DDGS) under three drying methods

Drying Method	Setting	Equilibrium Moisture Content, % dry basis*
Forced Air	40°C	4.62 (0.29)
	60°C	3.17 (0.45)
	80°C	2.15 (0.19)
	100°C	1.50 (0.14)
	120°C	0.78 (0.16)
Microwave	40% power	2.95 (0.35)
	60% power	1.70 (0.58)
	80% power	0.90 (0.13)
	100% power	0.43 (0.09)
Microwave Convection	130°C-30% power	3.14 (0.48)
	150°C-30% power	2.63 (0.34)
	160°C-30% power	1.36 (0.18)
	190°C-30% power	1.06 (0.10)

* Values in parentheses represent standard deviation

These drying curves were also fitted to four models (exponential, Page, Henderson and Pabis, and Thompson) to describe the drying characteristics of wheat WDGS. Table 2.3 summarizes the results of the nonlinear regression analyses done. Although the first three models adequately described the drying characteristics of wheat WDGS under the forced air method (R^2 ranged from 0.941 to 0.999 and MSE values from 0.000 to 0.005), it was the Page model that gave the closest fit to the experimental data ($R^2=0.997-0.999$; $MSE=0.000$). Under the microwave and microwave convection drying methods, the Page model ($R^2 = 0.989$ to 0.997 ; $MSE = 0.001$ to 0.002) also best described the observed drying behavior of wheat distillers grain with solubles. It can be recalled that a modified Page model was also used by Tang et al. (2005) to represent the drying behavior of distillers spent grain using superheated steam (120, 145 and 160°C steam temperature at $0.70 \text{ m}\cdot\text{s}^{-1}$ steam velocity). Comparison with other studies on microwave-assisted drying methods cannot be made at the present time since there were no published studies found on wheat or corn WDGS.

Table 2.3 further shows that the Page model parameter k increased when the drying air temperature, the microwave power level, or the microwave-convection combination setting was increased. Increases in k , defined as the drying rate constant in min^{-1} , translate to steeper drying curves and thus, faster drying, as demonstrated in Figs. 2.3, 2.4, and 2.5.

Table 2.4 summarizes the relationships between the Page model parameters (k and n) and the drying process variables under forced air and microwave drying methods. Linear models explained more than 90% of the variability between the Page model parameters (k and n) and drying air temperature under the forced air drying method. Under microwave drying, a linear model was able to explain about 88% of the variability between k and microwave power level. The standard deviation (square root of MSE) of the observations about the regression line was also the least at 0.01. A nonlinear model ($R^2 = 0.811$, $\text{MSE} = 0.012$), on the other hand, was able to explain the variability between the Page model parameter n and microwave power level much better than a linear model ($R^2 = 0.61$, $\text{MSE} = 0.012$).

2.4.2 Estimated energy consumption

Table 2.5 shows the estimated energy consumption of drying wheat WDGS to an $\text{MR} = 0.5$ under forced air convection, microwave, and microwave convection methods. In forced air drying, when the drying air temperature was increased, drying time and, consequently, energy consumption decreased. Estimated energy consumption across the five forced air drying settings ranged from 1.3 to 5.8 kWh. In both microwave and microwave convection drying, there was no marked difference in the energy consumption across the various power levels and combination settings. Similar observations were made by Alibas (2006) in drying chard leaves. Compared to

Table 2.3 Parameters of various thin drying models in forced-air, microwave and microwave convection drying of wheat distillers grain with solubles

Model and Model Parameters		Forced air convection drying					Microwave drying				Microwave-convective drying			
		40°C	60°C	80°C	100°C	120°C	40% power	60% power	80% power	100% power	130°C, 30% power	150°C, 30% power	160°C, 30% power	190°C, 30% power
Exponential	k	0.013	0.200	0.035	0.043	0.055	0.110	0.203	0.196	0.234	0.182	0.212	0.220	0.232
	R ²	0.972	0.960	0.956	0.943	0.941	0.869	0.908	0.888	0.927	0.924	0.912	0.916	0.922
	MSE	0.002	0.004	0.004	0.005	0.005	0.018	0.015	0.018	0.012	0.006	0.008	0.007	0.007
Page	k	0.003	0.004	0.008	0.008	0.012	0.009	0.037	0.032	0.064	0.071	0.076	0.085	0.095
	N	1.328	1.418	1.443	1.521	1.541	2.174	2.018	2.137	1.853	1.595	1.706	1.682	1.665
	R ²	0.999	0.999	0.998	0.997	0.997	0.989	0.995	0.997	0.997	0.988	0.993	0.994	0.997
	MSE	0.000	0.000	0.000	0.000	0.000	0.002	0.001	0.001	0.001	0.001	0.001	0.001	0.000
Henderson & Pabis	g	0.015	0.022	0.039	0.049	0.064	0.124	0.218	0.213	0.247	0.209	0.249	0.257	0.273
	h	1.101	1.121	1.127	1.135	1.142	1.120	1.085	1.082	1.063	1.116	1.145	1.140	1.144
	R ²	0.985	0.978	0.976	0.967	0.967	0.893	0.919	0.901	0.934	0.947	0.943	0.947	0.953
	MSE	0.001	0.002	0.002	0.003	0.003	0.017	0.017	0.021	0.015	0.004	0.005	0.005	0.004
Thompson	a	-89.250	-59.813	-34.840	-28.908	-22.240	-10.390	-5.039	-6.352	-5.047	-7.105	-5.956	-5.863	-5.533
	b	-11.591	-8.811	-5.394	-5.157	-3.870	-1.818	-0.645	-1.179	-0.782	-1.617	-1.263	-1.312	-1.224
	R ²	0.995	0.992	0.992	0.988	0.987	0.910	0.923	0.958	0.972	0.981	0.969	0.975	0.978
	MSE	11.544	7.564	2.450	1.892	1.323	2.518	1.341	0.557	0.368	0.111	0.149	0.109	0.088

Table 2.4 Relationship between Page model drying parameters (k and n) and drying process variables (drying air temperature T_c (°C), and microwave power level P_{mw} (watts) under forced air and microwave drying methods.

Drying Method	Equation	R^2	MSE
Forced Air	$k = 0.0001T_c - 0.0018$	0.931	0.000
	$n = 0.0026T_c + 1.2386$	0.956	0.000
Microwave	$k = 0.0001P_{mw} - 0.0486$	0.877	0.000
	$n = -4E-06(P_{mw})^2 + 0.003P_{mw} + 1.248$	0.811	0.012

Table 2.5 Estimated energy usage to reach MR = 0.5 under different drying conditions*

Drying Method	Setting	Initial weight of samples for drying, g**	Estimated power output, W	Average time of half response, min	Estimated energy consumption, kWh
Forced Air	40°C	100.15 (1.53)		60.03 (0.24)	5.76 (0.02)
	60°C	103.66 (1.72)		40.16 (0.74)	3.85 (0.07)
	80°C	105.12 (1.11)	5757.67***	22.66 (0.42)	2.17 (0.04)
	100°C	97.09 (2.38)		18.84 (0.79)	1.81 (0.08)
	120°C	99.98 (0.48)		13.90 (1.00)	1.33 (0.10)
Microwave	40% power	104.26 (0.11)	419.92	7.39 (0.06)	0.05 (0.000)
	60% power	103.67 (1.90)	676.56	4.28 (0.02)	0.05 (0.000)
	80% power	109.34 (0.99)	701.14	4.22 (0.10)	0.05 (0.001)
	100% power	104.17 (0.76)	805.01	3.62 (0.18)	0.05 (0.002)
Microwave convection	130°C, 30% power	24.36 (2.00)	302.74	4.28 (0.34)	0.02 (0.001)
	150°C, 30% power	25.09 (0.42)	315.98	4.40 (1.01)	0.02 (0.005)
	160°C, 30% power	24.55 (0.61)	326.58	3.62 (0.25)	0.02 (0.002)
	190°C, 30% power	23.94 (0.55)	331.95	3.32 (0.13)	0.02 (0.001)

* Values in parentheses represent standard deviation.

** Initial weight was obtained from the average of two replicates for forced air and microwave drying and three replicates for microwave convection drying.

***sum of the nameplate power requirements of the air conditioning chamber, vane axial fan motor and weighing scale.

forced air drying, the other two drying methods (microwave and microwave convection) have lower energy consumption in attaining an $MR = 0.5$. Using the EMC values in Table 2.2, the Page model parameters in Table 2.3, the existing material capacities of the forced air thin layer dryer (300 g) and of the domestic microwave oven (100 g), and a target moisture content of 12% (wet basis), energy consumption was estimated at 61.5, 5.5, and 2.6 MJ per kg of water removed for 120°C forced air, microwave, and microwave convection drying. These values show the significant energy saving potential of microwave-assisted methods in drying wheat DDGS. However, these values were higher compared to those obtained by Stroem et al. (2009) in drying brewer's spent grain using superheated steam. They estimated energy consumption at 0.65 – 1.47 MJ per kg of water removed, with steam recovery. Rotary drum drying parameters used include 200-240°C steam temperature, 8.8-59% final product moisture, 14 and 23 kg·h⁻¹ feed rate (Stroem et al. 2009).

2.4.3 Average lysine content

The third and fourth columns of Table 2.6, respectively, shows the average lysine content of DDGS samples generated from the three drying methods and the average lysine content ratio of dried and wet samples. In general, decreases in average lysine content were observed in dried samples compared to wet samples. Table 2.7 shows an example of the amino acid profile of a wheat DDGS sample dried at 120°C under the forced air method.

Table 2.6 Average lysine content and color parameter values of wheat distillers grain with solubles (DDGS) samples (N=2) obtained under three drying methods*

Drying Method	Nominal setting	Average lysine content**		Average color parameters		
		%	$\frac{\text{dried sample}}{\text{wet sample}}$	<i>L</i>	<i>a</i>	<i>b</i>
Forced Air	40°C	1.16 (0.02)	1.05	40.30 (1.37)	7.42 (0.16)	14.03 (0.58)
	60°C	1.13 (0.08)	1.02	42.15 (0.71)	6.86 (0.32)	14.70 (0.03)
	80°C	1.07 (0.02)	0.96	39.68 (0.98)	6.66 (0.24)	12.68 (0.25)
	100°C	1.10 (0.02)	0.99	38.65 (0.73)	7.47 (0.31)	13.56 (0.54)
	120°C	1.00 (0.03)	0.90	37.46 (0.96)	7.11 (0.57)	13.04 (0.00)
Microwave	40% power	1.08 (0.09)	0.98	38.96 (1.24)	5.95 (0.32)	10.80 (0.95)
	60% power	0.93 (0.05)	0.84	38.38 (0.64)	6.43 (0.50)	10.83 (0.99)
	80% power	1.04 (0.01)	0.94	38.71 (1.31)	6.38 (0.10)	11.28 (0.44)
	100% power	1.10 (0.02)	1.00	40.37 (2.67)	5.87 (0.23)	11.12 (0.67)
Microwave	130°C, 30% power	1.11 (0.01)	1.00	43.24 (1.08)	6.69 (1.20)	12.94 (2.60)
Convection	150°C, 30% power	1.05 (0.06)	0.95	42.66 (0.37)	6.03 (0.47)	11.61 (0.39)
	160°C, 30% power	1.00 (0.03)	0.91	39.19 (0.46)	7.22 (0.14)	11.81 (0.47)
	190°C, 30% power	1.06 (0.03)	0.96	41.45 (0.59)	6.39 (0.84)	12.11 (1.44)

10.3 * Values inside the parentheses represent standard deviation, Hunter L, a, b indicate lightness, redness, and yellowness levels, respectively.

** Average lysine content values were based on dry matter.

Table 2.7 Amino acid analysis results (dry matter basis) from one wheat DDGS sample dried at 120°C under forced air drying method.

Amino Acid	%
Aspartic	1.13
Serine	2.03
Glutamic	11.40
Glycine	1.77
Histidine	0.00
NH ₃	1.93
Arginine	1.07
Threonine	0.50
Alanine	1.21
Proline	3.56
Cystine	0.34
Tyrosine	0.97
Valine	1.51
Methionine	0.40
L-Lysine	0.99
Isoleucine	1.18
Leucine	2.19
Phenylalanine	1.96
Total	34.14

In forced air drying, a negative linear correlation ($R = -0.87$) between drying air temperature and average lysine content was significant at the 0.05 level. The relationship between lysine content and temperature using Arrhenius law (van Boekel 2008), shown below, was also found significant at the 0.05 level, where Lys is lysine content (decimal) and T is absolute temperature (K).

$$\ln(\text{Lys}) = -5.0935 + 201.02 \left(\frac{1}{T} \right) \quad R^2 = 0.75 \quad (2.3)$$

Decreases in average lysine content at elevated temperatures are attributed to Maillard reaction, which involves the binding of amino groups of amino acids with the carbonyl compounds of

reducing sugars (Fayle and Gerrard 2002; Owusu-Apenten 2004). Lysine is the most susceptible among the amino acids to Maillard reaction because its free amino group at the epsilon carbon unit can readily react with reducing sugars (Lingnert 1990). It is also usually the limiting essential amino acid in animal feed ingredients, thus, lysine loss lowers the nutritive value of a feed material (Fayle and Gerrard 2002). The rate of of Maillard reaction depends upon temperature, time, pH, water activity and chemical composition (Owusu-Apenten 2004; Ames 1990). For example, increasing temperature and exposure time tend to increase the rate of Maillard reaction (Owusu-Apenten 2004; Ames 1990; Mavromichalis 2001). Variations in these reaction parameters will affect the final outcome of a Maillard reaction, such as color, aroma compounds and nonvolatile products.

Mavromichalis (2001) further indicated that at the early stage of Maillard reaction, lysine can still be detected in chemical measurements although it may no longer be biologically available. In late Maillard reaction stages, a reduction in the amount of chemically analyzed lysine would be observed (Mavromichalis 2001).

In both microwave and microwave convection drying, the average lysine content of the dried samples was not significantly different ($p > 0.05$) across the various settings. As illustrated in Figures 2.3, 2.4 and 2.5, drying time decreased as the microwave power level or the microwave convection setting was increased. This shorter time of exposure, as well as the lack of hot dry air surrounding the sample surface in the microwave oven, may have retarded Maillard reactions (Yaylayan and Roberts 2001). A number of researchers have also reported that microwave heating does not favor Maillard reaction (Ames 1990; Yaylayan and Roberts 2001; Reineccius and Whorton 1990). Although samples were simultaneously exposed to microwave energy and heated air during microwave convection drying, exposure times also became progressively

shorter as the temperature of the heated air was increased. Thus, no significant differences in lysine content were observed across the four combination settings.

2.4.4 Color parameters

The last three columns of Table 2.6 show the color parameters (L , a , b) of the dried samples under the three drying methods. The color parameter L represents lightness, with values ranging from 0 (black) to 100 (white) (Hunter Associates Laboratory, Inc. 2008). Negative a and b values represent greenness and blueness, respectively, while positive values of a and b represent redness and yellowness, respectively (Hunter Associates Laboratory, Inc. 2008). For the wheat DDGS samples generated from the three drying methods, the a and b values were all positive, indicating yellowness and redness, respectively, while the L values (37-43) were closer to the minimum (black).

The relationships between the color parameters and the process variables of the three drying methods were also assessed. In general, color changes of wheat DDGS samples under the forced air and microwave convection drying methods were largely due to changes in the L (lightness) values rather than to changes in the a (redness) and b (yellowness) values. Under the forced-air drying method, a negative linear correlation ($R = -0.75$) between L and drying air temperature was significant at the 0.05 level. As the drying air temperature was increased, the dried samples became darker in color. Relationship between drying air temperature (T , °K) and color parameter L using Arrhenius law (van Boekel 2008), shown as Eq. 2.4, was also significant at the 0.05 level.

$$\ln(L) = 3.285 + 138.0 \left(\frac{1}{T}\right) \quad R^2 = 0.54 \quad (2.4)$$

While there was some negative linear correlation ($R = -0.58$) observed between b and drying air temperature under forced air drying, this was not significant at the 0.05 level. For the color parameter a , no linear correlation was observed with drying air temperature.

Under microwave convection drying at the same 30% power level, L values of dried samples were significantly different ($p < 0.05$) across the four combination settings. The average L values of those dried at the 160°C-30% power setting were significantly lower than those dried using the first two combination settings (130°C-30% power, and 150°C-30% power), indicating significantly darker samples were produced at this higher setting. Values of a , as well as those of b , were not significantly different ($p > 0.05$) across the four microwave convection combination settings.

Darkening of samples as a result of exposure to elevated temperatures during forced air and microwave convection drying is attributed to Maillard reaction. Browning stems from carbonyl compounds and becomes more rapid and intense in the presence of amino compounds (Hodge 1953). During the initial stage of Maillard reaction, colorless products are formed while highly colored products are associated with the advanced stages (Hodge 1953).

Under the microwave drying method, the L , a , and b values were not significantly different ($p > 0.05$) across the four microwave power levels. Reduced color formation during microwave heating is attributed to the lack of hot dry air surrounding the product, which prevents surface dehydration necessary for color formation, and to the shorter exposure time, which prevents completion of other Maillard reactions responsible for browning (Yaylayan and Roberts 2001).

2.4.5 Color and lysine content correlation

The lysine content and *L* values of wheat DDGS samples obtained from forced air, microwave, and microwave-convective drying methods were all positively correlated, with *R* values at 0.74, 0.39 and 0.64, respectively, indicating that higher lysine content tend to be found in the lighter colored wheat DDGS samples. For samples dried under the forced air convection method, lysine-*L* correlation (*R* = 0.74) was found significant at the 0.05 level. For those dried under the microwave and microwave convection methods, the lysine-*L* correlations (*R*=0.39 and *R*=0.64, respectively) were not significant at the 0.05 level. The strength of lysine-*L* correlation could be affected by how advanced Maillard reaction has developed in the dried samples. Hodge (1953) described several reactions that are known to occur during browning in sugar-amine systems and indicated that during initial stages of Maillard reaction, colorless products are formed (Lingnert 1990; Hodge 1953) but some amino acids, like lysine, may already be unavailable. Highly colored polymers are formed during the final stage of Maillard reaction (Hodge 1953; Yaylayan and Roberts 2001). Thus, correlation between lysine content and *L* values at the initial stage of Maillard reaction may not be as strong compared to the final stage.

2.5 Conclusions

The Page model provided a good fit to the experimental data generated from forced air, microwave and microwave convection drying of wheat distillers grain with solubles. Under forced air drying, the model parameters *k* and *n* increased linearly with temperature. Among the three drying methods, microwave-assisted methods significantly decreased the time to dry wheat WDGS. Microwave convection drying gave the shortest time of half response and consumed the lowest energy.

The color parameter *L* and lysine content of wheat DDGS samples decreased with increases in drying air temperature, with higher lysine content tended to be found in the lighter colored samples. Drying air temperature tended to produce stronger correlations with lysine content and the color parameter *L* than microwave power. No significant difference in lysine content and *L* values was observed across the different microwave drying settings (40%, 60%, 80% and 100% power).

The potential of microwave-assisted methods, particularly those that can achieve lower operating temperatures to minimize lysine degradation during drying of wheat distillers grain with solubles will be investigated by the authors in the near future.

Chapter 3

Effect of drying conditions and level of condensed distillers solubles on protein quality and drying characteristics of wheat distillers grain with solubles

Except for the additional entries described below, a similar version of this chapter had been published in the *Drying Technology* journal:

- Mosqueda, M.R., L.G. Tabil and C.Christensen. 2013. Effect of drying conditions and level of condensed distillers solubles on protein quality of wheat distillers dried grain with solubles. *Drying Technology* 31(7): 811-824.

This chapter contains an additional focus on moisture diffusivity that was not found in the published manuscript. The following modifications were introduced:

- a. In subsection 3.3.2 (Drying WDGS) under Materials and Methods,
 - i. A subheading (*3.3.2.1 Page model*) was added to distinctly mark the existing Page model curve fitting procedure from the moisture diffusivity procedure that is to be incorporated.
 - ii. A subsection (*3.3.2.2 Effective moisture diffusivity*) was added to outline moisture diffusivity procedure. Since this procedure includes a new table on estimated slab thickness (Table 3.2) and two new equations (Eq. 3.2 and 3.3), numbering of subsequent tables and equations was adjusted.
- b. In subsection 3.4.1 (Drying the various WDG:CDS blends) under Results and Discussion,

- i. The subsection title was changed from “*Drying the various WDG:CDS blends*” to “*Drying characteristics*” to enable subsequent presentation of moisture diffusivity results.
 - ii. A subheading (*3.4.1.1 Page model*) was added to distinctly mark existing Page model curve fitting results and discussion from the additional moisture diffusivity results.
 - iii. A subsection (*3.4.1.2 Effective moisture diffusivity*) was added to present the additional results. It also included a new figure (Fig. 3.2). Numbering of subsequent subsections was also adjusted.
- c. Additional moisture diffusivity-related entries were also made under Abstract, Introduction, and Conclusions sections.

Contribution of the PhD Candidate

The PhD candidate prepared the literature review, planned and executed the experiments, analyzed the data sets, and wrote the manuscript under the guidance of her research supervisor, Lope G. Tabil. She was assisted by Shivani Trivedi during sample preparation. Amino acid analysis was outsourced to a Saskatoon laboratory because of equipment limitation at the University. She was trained by Angela Hennings of the Feeds Innovation Institute in crude protein and acid detergent insoluble crude protein determination. Lope Tabil and Colleen Christensen provided editorial advice during manuscript preparation.

Contribution of the Paper to the Overall Study

This manuscript examined the effect of drying methods/conditions and level of condensed distillers solubles (CDS) incorporation on the protein quality and moisture diffusivity of wheat distillers dried grain with solubles (DDGS). It partially addressed these two main objectives: (i) evaluation of alternative drying methods that would minimize protein damage, energy consumption, and associated costs; and (ii) assessment of the effect of drying methods/conditions and the blending process on the physical and chemical characteristics of wheat DDGS.

There is currently no study that quantified the effect of the drying methods/conditions and CDS level on the protein quality and moisture diffusivity of wheat DDGS. Information derived from this study would be helpful in feed quality improvement efforts. Ethanol plant managers and quality assurance personnel can use the study results as basis for examining their existing operations, identifying where improvements can be made, and developing the necessary procedures to ensure wheat DDGS can be produced with more consistent and predictable quality. Results on moisture diffusivity would be important in optimizing the drying process for wheat distillers grain with solubles.

3.1 Abstract

Reduced protein quality is one of the concerns currently confronting the supply and utilization of wheat distillers dried grain with solubles (DDGS) as an animal feed ingredient. This study assessed the protein quality of wheat DDGS, expressed as acid detergent insoluble crude protein (ADICP) and lysine content, by blending wet distillers grain (WDG) with varying condensed distillers solubles (CDS) levels and drying using forced air convection, microwave, and

microwave–convection methods. As the CDS level was increased, the protein content of wheat DDGS generated from the three drying methods increased. Interactions of CDS level with drying air temperature, microwave power, and microwave–convection settings had a significant effect ($p < 0.05$) on average acid detergent insoluble crude protein (ADICP) and lysine contents. Higher ADICP and lower lysine contents were observed in samples dried at higher temperature, microwave power, and microwave convection settings. Further, the CDS level significantly affected the color parameters of microwave- and microwave–convection-dried samples and the drying air temperature–CDS level interaction significantly affected the color of forced air convection–dried samples. Significant lysine content–redness, ADICP–lightness color parameter, and ADICP–total color difference correlations were found in forced-air convection, microwave-, and microwave–convection-dried samples, respectively. Microwave and microwave–convection drying achieved desirable protein quality associated with low-temperature drying at much shorter times. Moisture diffusivity increased with drying temperature and microwave power while it decreased as CDS level was increased.

3.2 Introduction

With the significant expansion of the wheat-based fuel ethanol industry in western Canada in recent years, DDGS is increasingly becoming more available in the region. It has been estimated that western Canada’s ethanol plants collectively produce about 460,000 tonnes of wheat DDGS annually (University of Saskatchewan 2009). Utilized primarily as an animal feed ingredient, its supply and utilization, however, are faced with a number of challenges. Reduced protein quality, particularly lysine damage, of wheat DDGS was among the concerns highlighted in a number of animal feed studies (Widyaratne and Zijlstra 2007; Lan et al. 2008; Cozannet et al. 2010;

Nyachoti et al. 2005). Despite its higher crude protein and amino acid content, wheat DDGS not only showed lower lysine content (Widyaratne and Zijlstra 2007) but also lower digestibility values (Widyaratne and Zijlstra 2007; Lan et al. 2008; Cozannet et al. 2010; Nyachoti et al. 2005).

Because lysine is usually the limiting essential amino acid in animal feed ingredients, its loss lowers the nutritive value of a feed material (Fayle and Gerrard 2002), ultimately affecting animal growth performance. This was demonstrated in a study by Cromwell et al. (1993), in which they correlated the physical, chemical, and nutritional characteristics of corn DDGS with the growth performance of swine and poultry. Widyaratne and Zijlstra (2007) also indicated that lysine damage would be a constraint in the utilization of wheat DDGS as feed for monogastric animals. Lysine loss in wheat DDGS could be affected by both ethanol extraction and DDGS production processes. Liquefaction (Cozannet et al. 2010), saccharification (Cozannet et al. 2010), and fermentation (Widyaratne and Zijlstra 2007) were among the ethanol extraction processes identified. In DDGS production, dehydration of thin stillage into condensed distillers solubles (CDS) (Cozannet et al. 2010), addition of CDS to the wet distillers grain (WDG) (Nyachoti et al. 2005), and drying (Nyachoti et al. 2005; Zijlstra and Beltranena 2009) were among those identified to adversely affect lysine content and digestibility (Stein et al. 2006).

Marked losses of lysine during drying and other heat treatment processes have been attributed to the Maillard reaction, which involves the binding of amino groups with the carbonyl compounds of reducing sugars (Widyaratne and Zijlstra 2007; Cozannet et al. 2010; Nyachoti et al. 2005). Among the essential amino acids, lysine is particularly vulnerable because its free ϵ -amino group can readily react with the reducing sugars (Mao et al. 1993). This bound lysine is not normally susceptible to enzymatic decomposition, thus rendering the amino acid nutritionally unavailable

(Mao et al. 1993). Mavromichalis (2001) further indicated that, during the early stage of the Maillard reaction, lysine can still be detected but may no longer be biologically available. In late Maillard reaction stages, a reduction in the amount of chemically analyzed lysine was observed (Mavromichalis 2001). The rate of the Maillard reaction depends upon temperature, time, water activity, and chemical composition, among others (Ames 1990; Owusu-Apenten 2004). Increasing temperature and exposure time tend to increase the rate of the Maillard reaction (Mavromichalis 2001; Ames 1990; Owusu-Apenten 2004). The reaction rate also depends on chemical composition (Evangelisti et al. 1999). In corn DDGS, Kingsly et al. (2010) reported that incorporating higher levels of CDS resulted in a darker product because more reducing sugars are available to react with proteins during the drying process. The Maillard reaction also occurs less readily in substances with high water activity (Ames 1990; Lingnert 1990). When a product is subjected to high temperatures for a considerable time, as in drying processes, its surface dries out, producing a crust with a low water activity, favoring the Maillard reaction (Ames 1990). In addition to lysine, acid detergent insoluble nitrogen (ADIN) or acid detergent insoluble crude protein (ADICP) and color have been reported as indicators of heat-damaged proteins. ADICP is the crude protein recovered from the acid detergent fiber, which primarily consists of cellulose, lignin, and variable silica (Perry et al. 2003). Higher levels of ADICP are suggestive of greater heat damage (Perry et al., 2003; Coblenz et al. 2010). Perry et al. (2003) indicated that forage processing temperatures above 50°C increased the production of artifact lignin via the Maillard reaction and the nitrogen content in the acid detergent fiber (ADF) is used as a basis for estimating this artifact lignin. Color, on the other hand, has been suggested in corn DDGS studies as a quick indicator of lysine content and availability (Batal and Dale 2006; Fastinger et al. 2006) and good nutritional properties (Cromwell et al. 1993). Lighter colored

corn DDGS had higher lysine content and digestibility than darker colored samples (Batal and Dale 2006; Fastinger et al. 2006). The color parameter L was also highly correlated with growth rate and feed-gain, with the darkest colored corn DDGS consumed in the least amount and resulting in the poorest growth rate (Cromwell et al. 1993).

Understanding the effect of various process conditions during ethanol and DDGS production on protein quality, therefore, is imperative to improve the nutritive value and market prospects of wheat DDGS as an attractive alternative to the more expensive, high-protein animal feed ingredients. Though there are already a number of studies that have investigated the effect of CDS level on the physical and chemical characteristics of corn DDGS, there is still very limited detailed information on wheat DDGS. In corn DDGS, Kingsly et al. (2010) and Clementson and Ileleji (2012) conducted plant-scale experiments to examine the effect of CDS on the chemical composition and physical properties using the bulk material and sized fractions, respectively. Ganesan et al. (2008a-c) examined the effect of soluble level and moisture content on the physical, flow, and chemical characteristics at the laboratory scale. Bhadra et al. (2011a; 2011b) studied the drying behavior of corn DDGS and developed a modified Page model to predict the moisture content of corn DDGS at varying drying times, CDS levels, and drying temperatures. They also examined the effect on varying CDS and drying temperature levels on the physical and flow properties of corn DDGS (Bhadra et al. 2012).

In wheat DDGS, previous studies did not consider the influence of CDS level. Tang et al. (2005) and Pronyk et al. (2004) did not consider the effect of CDS level when they investigated the effect of superheated steam drying parameters on starch, protein, β -glucan, and pentosan content of wheat distillers spent grain. Mosqueda and Tabil (2011a) examined the drying characteristics and the relationship between lysine content and color of wheat DDGS produced from forced air

convection, microwave, and microwave–convection methods using wet distillers grains with solubles (WDGS) samples obtained from an ethanol plant. The objective of this study, then, was to examine the effect of drying methods/temperature and the level of CDS incorporation on the protein quality and drying characteristics of wheat distillers grain with solubles. Protein quality was quantified in terms of lysine and ADICP contents. It also aimed to verify whether color can be an indicator of protein quality.

3.3 Materials and Methods

3.3.1 Preparation of WDGS Samples

CDS and WDG (Fig. 3.1a and 3.1b) were obtained from a south Saskatchewan fuel ethanol plant and were stored in tightly sealed bins and pails inside a -18°C freezer until use. These were thawed overnight prior to the mixing runs. Thawed CDS was thoroughly mixed by hand before weighing the amount needed for a mixing run.



Fig. 3.1 Blending of (a) wheat condensed distillers solubles (a) and wet distillers grain (b) at varying proportions using a hand mixer (c).

One-kilogram batches of WDGS were prepared by mixing CDS and WDG (Fig. 3.1c) for 30 min using a Toastmaster 6-speed Hand Mixer (model 1776CAN, Toastmaster Inc., China) at medium

speed (set at speed no. 3). The resulting WDGS was stored in plastic bags inside the -18°C freezer until they were used. Because of personnel and freezer space considerations, only about 10 kg of WDGS were typically prepared for each mixing day. Stocked WDGS was replenished when this inventory ran low.

The level of CDS incorporation ranged from 15 to 45%, by mass. This range was assumed based on the information provided by an ethanol plant personnel. The amount of thin stillage entering the evaporation process was about 30-32% of the material leaving the centrifugation process. The amount of CDS entering the blending process could be below or above these percentage values.

3.3.2 Drying the WDGS

Prior to the drying runs, an appropriate number of bags of WDGS were thawed overnight for equilibration at room temperature (22°C). About 100 g of the laboratory-prepared WDGS was used for each drying run. Three drying methods were used: (1) forced air convection, (2) microwave, and (3) microwave–convection. The use of microwave energy was investigated as a potential alternative drying technology because it offers shorter drying times with no significant loss of product quality compared to conventional hot air drying (Chung and Furutan 1989; Sosyal 2004; Du et al. 2005). Thus, in addition to the potential energy savings, its use could contribute toward minimizing lysine damage during drying and consequently improve the market prospects of wheat DDGS.

In forced air convection drying, an air recirculating drying system was used, which consisted of an airconditioning unit equipped with humidity and temperature sensors, a vane-axial circulating fan, a drying chamber with three wire, scale-mounted trays, and a duct system. This system was

described in detail by Kashaninejad and Tabil (2004) and Kashaninejad et al. (2007). Drying air temperature was set at three levels (40, 80, 120°C) and air velocity and relative humidity were set at 0.7–0.8m·s⁻¹ and below 8%, respectively.

A domestic Panasonic microwave-convection oven (model NNC-980W, Panasonic Canada, Ltd., Mississauga, ON) was used for microwave and microwave–convection drying. Table 3.1 shows its technical specifications. Airflow rate of the domestic microwave oven was not modified and was not measured during drying.

Table 3.1 Panasonic NNC-980W microwave/convection oven technical specifications

Power consumption	
Microwave	12.8 A, 1500 W
Heater	12.5 A, 1500 W
Power output	
Microwave	1100 W
Heater	1400 W
Outside dimensions	376 mm (H) x 606 mm (W) x 491 mm (D)
Oven cavity dimensions	242 mm (H) x 412 mm (W) x 426 mm (D)
Operating frequency	2450 MHz

Source: Matsushita Electric Industrial Co., Ltd. (2000)

Settings used for the microwave drying were at four power levels: 40% (P4), 60% (P6), 80% (P8), and 100% (P10) microwave power (Matsushita Electric Industrial Co. 2000). The power output at these levels was previously determined to be 420, 676, 701, 805 W, respectively (Mosqueda and Tabil 2011a). For microwave–convection drying, the oven’s four nominal combination settings, 130°C–30% power (C1), 150°C–30% power (C2), 160°C–30% power (C3), and 190°C–30% power (C4) (Matsushita Electric Industrial Co. 2000) were used. The power output (30% power) at each of these combinations of settings was previously estimated to be 303, 316, 327, and 332W, respectively (Mosqueda and Tabil 2011a). Separate drying trials

were done to check the air temperature near the product surface using a fiber optic probe connected to a Neoptix fiber optics signal conditioner (Neoptix Inc., Quebec City, QC). In this study, oven temperatures did not reach the stated nominal settings.

The drying air temperature, microwave power, and microwave–convection combination settings employed in the study were limited to the operating ranges of the available equipment in the Department of Chemical and Biological Engineering, University of Saskatchewan. Although the drying conditions used in this study were milder compared to those in wheat ethanol plants, a previous study (Mosqueda and Tabil 2011a) had observed significant differences in product color and lysine content even under these conditions.

For each drying run, the sample was spread in a thin layer on an aluminum foil–lined wire mesh tray during forced air convection drying or on a parchment paper–lined rotating tray for microwave and microwave–convection drying. The sample was dried until the moisture content reached between 8 and 12% (wb). Moisture content was determined using AOAC official method 930.15 (AOAC 2003a). Using the forced air convection method, drying air temperature, relative humidity, air velocity, and sample mass were continuously monitored during the drying process using built-in drying system instrumentation. Drying air conditions were also monitored for 1 h before the sample was placed in the drying chamber. To determine sample mass changes during microwave and microwave–convection drying, the rotating tray containing the sample was removed from the oven every 2 min during the drying period and weighed. Periodic removal of the sample to measure mass loss during microwave drying had been employed in a number of studies (Chung and Furutan 1989; Sosyal 2004; Alibas 2006; Maskan 2000; Maskan 2001). Product temperature was not measured in all three drying methods.

The laboratory-prepared WDGS with varying CDS content was also freeze dried using a Labconco FreeZone Freeze Dry System (Labconco Corp., Kansas City, MO) for comparison with the other samples.

3.3.2.1 *Page model*

Experimental data generated from the drying runs were fitted to the Page model (Eq. 3.1) using SPSS 14.0 for Windows (SPSS Inc., Chicago, IL). The coefficient of determination (R^2) and mean square error (MSE) were used to determine goodness of fit at a significance level of 0.05. It was determined in a previous study by Mosqueda and Tabil (2011a) that the Page model best described the drying behavior of wheat distillers grain with solubles. In Eq. 3.1, MR stands for moisture ratio, t is drying time (min), and k and N are the Page model parameters. The model parameter k is the drying rate constant (min^{-1}).

$$\text{MR} = \exp(-kt^N) \quad (3.1)$$

3.3.2.2 *Effective moisture diffusivity*

Assuming that diffusion was the moisture transfer mechanism during drying, isothermal drying condition, uniform initial moisture distribution, constant slab thickness and moisture diffusivity, and negligible external resistance, and an infinite slab geometry, effective moisture diffusivity (D_{eff}) was estimated using Eq. 3.2 (Srikiatden and Roberts 2007). Assuming further that only the first term of the series dominates, Eq. 3.2 was simplified into a straight line equation (Eq. 3.3) (Srikiatden and Roberts 2007).

$$\text{MR} = \frac{M_t - M_e}{M_i - M_e} = \frac{8}{\pi^2} \sum_{s=0}^{\infty} \frac{1}{(2s+1)^2} \exp\left(- (2s+1)^2 \frac{\pi^2 D_{\text{eff}} t}{4l^2}\right) \quad (3.2)$$

$$\ln(\text{MR}) = \ln\left(\frac{8}{\pi^2}\right) - \frac{\pi^2 D_{\text{eff}} t}{4l^2} \quad (3.3)$$

where MR is the moisture ratio, M_e is the equilibrium moisture content (db), M_i is the initial moisture content (db), M_t is the moisture content (db) at time t (min), D_{eff} is effective moisture diffusivity, and l is the slab thickness (m), with moisture removal occurring from one surface of the material only. In Eqs. 3.2 and 3.3, moisture ratios under microwave and microwave convection drying were calculated using an equilibrium moisture content of zero (Maskan 2000). The slab thickness (l) values, presented in Table 3.2, were estimated using the bulk density of the wet material, the amount of wet material used for each drying run, and the surface area of the sample trays used during drying. These l values were later expressed in terms of meters before using Eq. 3.3. D_{eff} was determined from the slope of the resulting $\ln(\text{MR}) - t$ plot (Eq. 3.3) and later expressed in units of $\text{m}^2 \cdot \text{s}^{-1}$.

Table 3.2 Estimated slab thickness (mm) used in effective moisture diffusivity calculations for the different sample types and drying conditions. Slab thickness is expressed in the form: mean (standard deviation), N = 2

Drying method	Dryer setting	% CDS level, by mass		
		15	30	45
Forced air convection	40°C	2.04 (0.18)	1.67 (0.01)	1.51 (0.01)
	80°C	2.16 (0.01)	1.68 (0.01)	1.53 (0.03)
	120°C	2.15 (0.04)	1.67 (0.03)	1.52 (0.04)
Microwave	P4 (420 W)	1.71 (0.01)	1.31 (0.01)	1.23 (0.03)
	P6 (676 W)	1.71 (0.03)	1.34 (0.02)	1.24 (0.00)
	P8 (701 W)	1.73 (0.01)	1.32 (0.00)	1.22 (0.03)
	P10 (805 W)	1.72 (0.01)	1.33 (0.00)	1.24 (0.00)
Microwave convection	C1 (130°C-30% power)	1.69 (0.02)	1.33 (0.00)	1.19 (0.01)
	C2 (150°C-30% power)	1.70 (0.01)	1.33 (0.00)	1.23 (0.00)
	C3 (160°C-30% power)	1.72 (0.01)	1.34 (0.02)	1.21 (0.00)
	C4 (190°C-30% power)	1.72 (0.03)	1.34 (0.00)	1.21 (0.01)

3.3.3 Crude protein, ADICP and lysine content

Samples obtained from the drying runs were ground using a Thomas-Wiley mill (Thomas Scientific, Swedesboro, NJ) equipped with a 1-mm screen to generate the bulk material. Crude protein content was estimated using the Kjeldahl method, AOAC official method 984.13 (AOAC 2003b). ADF was determined using AOAC official method 973.18 (AOAC, 2003c). ADICP was determined from the residue of ADF analysis using AOAC official method 984.13 (AOAC 2003b) and was expressed as a percentage of crude protein (dry matter basis). A lamb starter feed sample (AAFCO 0728, AAFCO Check Sample Program, Association of American Feed Control Officials, Champaign, IL) was used as the control. Duplicate runs were made from each sample.

For lysine content determination, samples were sent to an external laboratory for amino acid analysis using the Waters AccQ-Tag method (Waters Corp. 1996). This method involves hydrolysis and dilution, derivatization of amino acids, and subsequent separation and analysis using a high performance liquid chromatography system (Waters Corp. 1996). AccQFluor derivatization buffer and AccQ-Fluor reagent (Waters Corporation, Milford, MA) were used during the derivatization step (Waters Corp. 1996). Duplicate runs were made from each sample.

3.3.4 Color

Color of the dried samples was determined using a HunterLab LabScan II colorimeter (Hunter Associates Laboratory Inc., Reston, VA) with a ½” area view and port size setting. Color was expressed in terms of the L, a, and b parameters. The color parameter L ranges from 0 (black) to 100 (white). Positive values of the color parameters a and b represent redness and yellowness, respectively, while their respective negative values represent greenness and blueness.

For samples dried under forced air convection, microwave, and microwave convection methods, color was also expressed using the total color difference (ΔE), computed using Eq. 3.4. Freeze-dried samples were considered as the standard and their color parameters were represented as L_s , a_s , and b_s in Eq. 3.4. The variables L , a , and b in the equation represent the color parameters of samples dried under forced air convection, microwave, and microwave convection.

$$\Delta E = \sqrt{(L - L_s)^2 + (a - a_s)^2 + (b - b_s)^2} \quad (3.4)$$

3.4 Results and Discussion

3.4.1 Drying characteristics

The average initial moisture content of the laboratory-prepared wheat WDGS samples ranged from 67.7% (samples with 15% CDS) to 70.2% (samples with 45% CDS). As expected, the longest drying times were observed under the forced convection method while samples dried under the microwave method produced the shortest drying times.

3.4.1.1 Page model

Table 3.3 shows that the Page model adequately described the drying behavior of wheat WDGS with varying CDS level, with R^2 values ranging from 0.994 to 0.998.

Under forced air convection, the Page model parameter k or the drying rate constant was lowest at 40°C (0.002 to 0.003 min^{-1}) and highest at 120°C (0.012 to 0.017 min^{-1}). This translates to

Table 3.3 Page model parameters of wheat distillers dried grain with solubles produced with varying condensed distillers solubles (CDS) level and dried under forced-air, microwave, and microwave convection methods

Drying method	CDS, % mass	Dryer setting	k ¹	N ²	R ²⁻³	MSE ⁴
Forced-air convection	15	40°C	0.002 ^{aA}	1.402	0.995	0.000
	15	80°C	0.006 ^{bA}	1.497	0.995	0.000
	15	120°C	0.012 ^{cA}	1.552	0.998	0.000
	30	40°C	0.002 ^{aB,aA}	1.393	0.996	0.001
	30	80°C	0.006 ^{bB,bA}	1.497	0.996	0.000
	30	120°C	0.014 ^{cB,cA}	1.538	0.998	0.000
	45	40°C	0.003 ^{aC,aA}	1.390	0.997	0.000
	45	80°C	0.007 ^{bC,bA}	1.475	0.998	0.000
	45	120°C	0.017 ^{cC}	1.481	0.998	0.000
Microwave ⁵	15	P4	0.008 ^{dA}	2.138	0.998	0.000
	15	P6	0.064 ^{eA}	1.698	0.996	0.001
	15	P8	0.080 ^{fA}	1.639	0.996	0.001
	15	P10	0.094 ^{gA, fA}	1.598	0.997	0.001
	30	P4	0.018 ^{dB, dA}	1.853	0.998	0.001
	30	P6	0.049 ^{eB, eA}	1.870	0.998	0.001
	30	P8	0.080 ^{fB, fA}	1.767	0.998	0.001
	30	P10	0.083 ^{gB, gA}	1.738	0.998	0.001
	45	P4	0.015 ^{dC, dA}	1.991	0.994	0.001
	45	P6	0.047 ^{eC, eA}	1.963	0.995	0.001
	45	P8	0.053 ^{fC, gC}	2.062	0.996	0.001
	45	P10	0.038 ^{gC, eC}	2.166	0.994	0.001
Microwave convection ⁶	15	C1	0.016 ^{hA}	1.764	0.996	0.001
	15	C2	0.014 ^{hA}	1.809	0.996	0.000
	15	C3	0.016 ^{hA}	1.774	0.996	0.000
	15	C4	0.014 ^{hA}	1.778	0.996	0.001
	30	C1	0.010 ^{hA}	1.936	0.996	0.001
	30	C2	0.013 ^{hA}	1.865	0.996	0.001
	30	C3	0.013 ^{hA}	1.837	0.996	0.001
	30	C4	0.011 ^{hA}	1.902	0.996	0.001
	45	C1	0.010 ^{hA}	1.966	0.995	0.001
	45	C2	0.009 ^{hA}	2.004	0.995	0.001
	45	C3	0.009 ^{hA}	1.981	0.994	0.001
	45	C4	0.014 ^{hA}	1.914	0.994	0.001

¹ drying rate constant. Tukey's test at 5% significance level for the same CDS level at varying drying temperatures (a, b, c), microwave power (d,e,f,g), microwave convection setting (h,i,j,k) and for the same drying temperature at varying CDS levels (A,B,C). Values followed by the same set of letters are significantly different; ² Page model parameter; ³ coefficient of determination; ⁴ mean square error; ⁵P4 - 420 W; P6 - 676 W; P8 - 701 W; P10 - 805 W; ⁶ C1 - 130°C-30% power (303 W); C2 - 150°C-30% power (316 W); C3 - 160°C-30% power (327 W); C4 - 190°C-30% power (332 W)

longer drying times at 40°C (2.5 to 3.4 h) compared at 120°C (33 to 36 min). The k value ranges obtained in this study (0.002 to 0.017 min⁻¹) are comparable to those reported by Mosqueda and Tabil (2011a) (k = 0.003 – 0.012 min⁻¹) in drying plant-sourced wheat WDGS samples under similar drying conditions. In drying corn WDGS, Bhadra et al. (2011a; 2011b) showed that a modified Page model, incorporating both CDS level and drying air temperature, adequately described co-product's drying behavior. Their reported k value (0.00148 min⁻¹) could not be compared to those obtained in this study because of inherent differences between the two studies with respect to drying model, raw material composition, sample mass, and drying conditions used.

Analysis of variance showed that interaction between drying air temperature and CDS level had a significant effect on the values of the Page model parameter k. This interaction effect, however, was only manifested in samples dried at 120°C, wherein those with 45% CDS had significantly higher k value than those with 15% and 30% CDS. For samples dried at the lower temperatures (40°C and at 80°C), CDS level did not have a significant effect on the k values. Further, when the samples were analyzed by CDS level, it was consistently observed that those dried at the higher drying air temperature had significantly higher k values than those dried at the lower temperature (Table 3.3). At each CDS level, those dried at 120°C had significantly higher k value than those dried at 40°C and at 80°C. Similarly, those dried at 80°C had significantly higher k values than those dried at 40°C.

Under microwave drying, the k values ranged from 0.008 to 0.094 min⁻¹ (Table 3.3). Drying times at 40% power (P4 or 420 W) setting ranged from 14 to 18 min while those at 100% microwave power (P10, 801 W) averaged about 7 to 8 min for all blends. Mosqueda and Tabil

(2011a) reported k values ($0.009 - 0.064 \text{ min}^{-1}$) for plant-sourced wheat WDGS that were within the observed range for laboratory-prepared samples. Result from the analysis of variance showed the interaction between microwave power and CDS level was significant. Posthoc multiple comparisons (Table 3.3) showed that similar trends as what was observed in the previous study of Mosqueda and Tabil (2011a). Samples dried at higher microwave power levels had significantly higher k values than those dried at the lower power levels (Table 3.3). This was consistently observed in samples with 15%, 30%, and 45% CDS level. When samples were grouped according to microwave power level, CDS level did not have a significant effect on the k values when drying was done at the lower power levels (P4 or 420 W; P6 or 676 W). However, when drying was conducted at the higher microwave power levels (P8 or 701 W; P10 or 805 W), samples with 15% CDS showed significantly higher k values than those with 45% CDS (Table 3.3).

Under the microwave convection method, the values of the Page model parameter k ranged from 0.009 to 0.016 min^{-1} . The k values for the lowest nominal setting (C1, 130°C -30% power) ranged from 0.01 for samples with 45% CDS content to 0.016 min^{-1} in samples with 15% CDS while those for the highest setting (C4, 190°C -30% power), drying rates also ranged from 0.011 to 0.016 min^{-1} . Drying times were also similar. C1 samples achieved the moisture content target range between 17-20 minutes while C4 samples dried between 15-18 min. These k value ranges, however, were lower than what was reported by Mosqueda and Tabil (2011a) ($k = 0.071 - 0.095 \text{ min}^{-1}$) for the plant-sourced wheat WDGS because of sample mass differences. This study used a sample mass of 100 g while the previous study used 25 g for each drying run.

3.4.1.2 *Effective moisture diffusivity*

Figure 3.2 shows the effective moisture diffusivity (D_{eff}) of the wheat DDGS samples, produced at varying CDS level and dried under forced-air convection-, microwave-, and microwave convection methods. Forced-air convection samples gave the lowest D_{eff} values while the microwave samples produced the highest values.

Regardless of the drying method used, Fig. 3.2 shows that D_{eff} linearly decreased as CDS level increased ($R^2 = 0.72 - 0.90$, $P < 0.05$). This could be attributed to differences in porosity of the samples since a porous structure presents less resistance to moisture movement. The influence of porosity on moisture diffusivity was also demonstrated in the study of Xiong et al. (1991), where higher moisture diffusivity values were obtained in porous puffed pasta than in the less porous regular pasta. Laboratory-scale investigations showed that increases in CDS level were associated with decreases in porosity (Tables 4.5 and 5.6). Difference in porosity of the wet samples was also observed while spreading the material in thin layers on the trays in preparation for drying. This was reflected in estimated thickness of the material in Table 3.2, where larger L values were obtained in samples with lower CDS content (loose/less dense material) than in samples with higher CDS level (denser material). Variation in D_{eff} could also be due to differences in chemical composition since increases in CDS level resulted to increases in protein and ash content and decreases in fiber content (Tables 4.4 and 5.4). This, however, needs to be further investigated so that the effect of porosity and that of composition on D_{eff} can be differentiated.

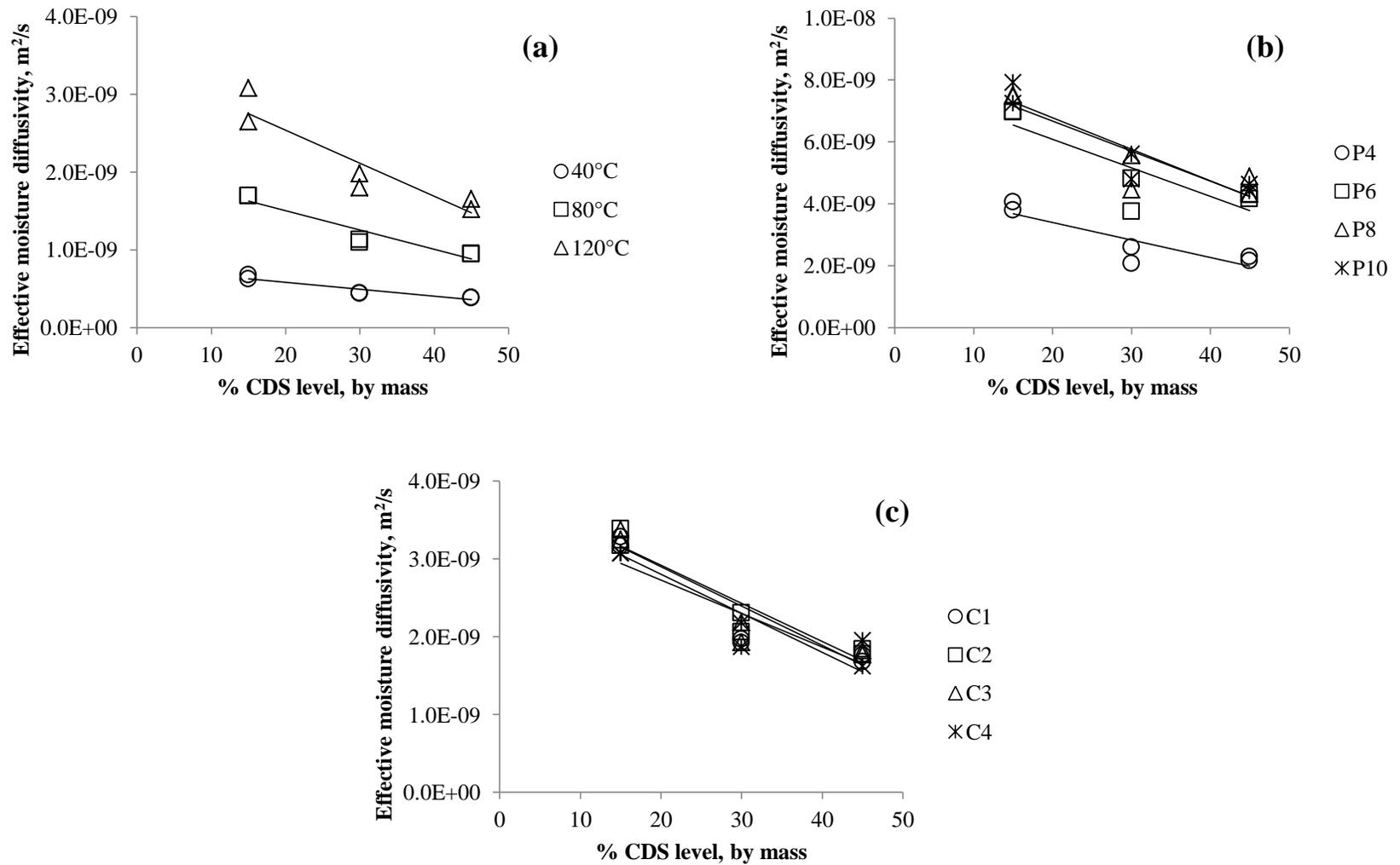


Fig. 3.2 Effective moisture diffusivity of wheat distillers grain with solubles produced at varying levels of condensed distillers solubles (CDS) and dried under (a) forced-air convection, (b) microwave, and (c) microwave convection methods. P4 - 420 W; P6 - 676 W; P8 - 701 W; P10 - 805 W; C1 - 130°C-30% power (303 W); C2 - 150°C-30% power (316 W); C3 - 160°C-30% power (327 W); C4 - 190°C-30% power (332 W).

Aside from CDS level, drying temperature, CDS level-drying temperature, and microwave power significantly affected D_{eff} . At each CDS level, D_{eff} values linearly increased with drying temperature ($R^2 = 0.98 - 0.99$, $P < 0.05$) and microwave power ($R^2 = 0.82 - 0.92$, $P < 0.05$). Under the forced-air convection, variation in D_{eff} values due to drying temperature accounted about 75% of the ANOVA total sums of squares, suggesting the stronger influence of drying temperature than CDS level (18% of total sums of squares). CDS level-drying temperature interaction and random error accounted the remaining 5% and 2% of the observed variation. Under microwave drying, lower microwave power level (P4 or 420 W) produced a significantly lower D_{eff} value than the P6 (676 W), P8 (701 W), and P10 (805 W) levels. The latter three power levels (P6, P8, P10) produced statistically similar D_{eff} values. Under microwave convection method, dryer setting did not significantly affect the D_{eff} values. This is because drying was done at constant microwave power level (303 – 332 W) and variations in product temperature brought about by the microwave convection settings may have not differed greatly to cause significant differences in the D_{eff} values.

3.4.2 Protein content

Table 3.4 shows the average crude protein, ADICP, and lysine contents of the plant-sourced DDGS and the freeze-dried DDGS samples with 15%, 30%, and 45% CDS. For the freeze-dried samples (Table 3.4), average crude protein content increased as CDS level in the blend was increased. Regression analysis showed that the linear model (Eq. 3.5) was significant ($p < 0.05$) and explained about 97% of the total variation observed between average protein content (% dry matter) of the freeze-dried samples and CDS level (% WDGS mass).

$$\text{Protein} = 0.157 * \text{CDS} + 26.437 \quad (3.5)$$

Table 3.4 Average crude protein, acid detergent insoluble crude protein (ADICP), lysine content (% dry matter) and color parameters of laboratory-prepared and ethanol plant-sourced wheat distillers dried grain with solubles (DDGS) samples. Wheat DDGS samples were prepared at varying levels of condensed distillers solubles (CDS). Values in parentheses represent standard deviation (N=2)

Sample	Average content, % (dry basis)			Color parameters ²		
	Protein	ADICP ¹	Lysine	L	a	b
15% CDS ³	29.02 (0.24)	6.95 (0.09)	1.03 (0.02)	53.0 (0.7)	5.6 (0.2)	16.5 (0.0)
30% CDS ³	30.70 (0.11)	6.19 (0.47)	1.02 (0.01)	51.5 (1.0)	5.9 (0.2)	16.5 (0.2)
45% CDS ³	33.74 (0.00)	5.66 (0.37)	1.09 (0.00)	48.5 (1.0)	6.8 (0.2)	16.7 (0.0)
Plant sample	38.01 (0.14)	18.37 (1.71)	0.76 (0.14)	35.3 (0.6)	10.2 (0.3)	15.3 (0.5)

¹ADICP – acid detergent insoluble crude protein; ²Color parameters L, a, and b represent lightness, redness and yellowness, respectively; ³Freeze-dried laboratory prepared samples

These results, although different from those reported by Kingsly et al. (2010) and Ganesan et al. (2008a) for corn DDGS, were expected for wheat DDGS. In a previous study, Mosqueda and Tabil (2011b) found higher protein content in CDS compared to the WDG fraction. They did not, however, obtain the amino acid profiles of these fractions. Ojowi et al. (1996; 1997) also reported higher protein content of thin stillage, where CDS was derived, than the WDG component. Results from these wheat-based studies are presented in Table 3.5 along with the chemical composition of the corn-based WDG and CDS fractions found in literature.

Table 3.5 Chemical composition of corn- and wheat-based wet distillers grain (WDG), condensed distillers solubles (CDS), and thin stillage fractions (% dry matter). Values in parentheses represent standard deviation (N=2)

Chemical constituent ¹	Corn-based		Wheat-based			
	WDG ^{2,3}	CDS ^{2,4}	WDG ⁵	WDG ⁶	CDS ⁶	Thin stillage ^{7,8}
Protein	34.4 – 35.0	22.4 – 23.0	26.4	28.84 (0.60)	45.82 (0.39)	45.7 – 48.5
Ash	2.0 – 2.35	6.6 – 11.6	2.7	2.90 (0.02)	9.30 (0.02)	8.0 – 8.3
Crude fat	10.9 – 14.0	21.6 – 34.4	6.6	6.71(0.04)	2.21 (0.04)	9.63 – 13.6
NDF	39.0	3.6 – 4.9	74.9	77.53 (0.24)	18.14 (0.29)	34.0 – 34.5
ADF	11.9 – 17.2	2.92	24.1	18.39 (0.39)	6.66 (0.08)	3.4 – 4.0

¹NDF – neutral detergent fiber, ADF – acid detergent fiber; ²Cao et al., 2009; ³Kim et al., 2008; ⁴Fron et al., 1996; ; ⁵Ojowi et al., 1997; ⁶Mosqueda and Tabil, 2011b; ⁷Ojowi et al., 1996 ⁸Mustafa et al., 1999.

Table 3.6 shows the average protein content of samples generated from forced air convection-, microwave-, and microwave convection-dried samples with varying CDS level. In all three sets of samples, average crude protein content also showed similar increasing trends as the amount of CDS in the blends increased. Regression analyses show that a positive linear relationship was able to explain about 95%, 97%, and 81% of the total variation observed between CDS level (% WDGS mass) and average protein content (% dry matter) in forced air convection-, microwave-, and microwave convection-dried samples. These relationships (Eqs. 3.6 – 3.8) were found significant at the 0.05 level:

$$\text{Forced-air convection:} \quad \text{Protein} = 0.116 * \text{CDS} + 28.515 \quad (3.6)$$

$$\text{Microwave:} \quad \text{Protein} = 0.133 * \text{CDS} + 28.132 \quad (3.7)$$

$$\text{Microwave convection:} \quad \text{Protein} = 0.110 * \text{CDS} + 28.968 \quad (3.8)$$

Table 3.7 shows how the observed mean differences in protein were affected by the factors and factorial interactions. In forced air convection-dried samples, CDS level and drying air temperature were found significant. Post hoc multiple comparisons of observed means (Table 3.6) further showed that samples with 45% CDS level had significantly higher protein content than those with 15% and 30% CDS level. Similarly, samples containing 30% CDS had significantly higher protein content ($p < 0.05$) than those with 15% CDS. In microwave-dried samples, both microwave power and CDS level caused significant differences in protein content (Table 3.7). Samples with 45% CDS had significantly higher protein content ($p < 0.05$) than those with 15% and 30% CDS level (Table 3.7). Similarly, those with 30% CDS also had significantly higher protein content than those with 15% CDS. Those dried at the lowest microwave power

Table 3.6 Protein, acid detergent insoluble crude protein (ADICP), lysine content and total color difference (ΔE) of wheat DDGS with varying levels of condensed distillers solubles (CDS) and dried under three methods. Values in parentheses are standard deviations

Drying method	CDS, % mass	Dryer setting	Chemical constituents ¹ , % dry matter			ΔE^2
			Protein	ADICP	Lysine	
Forced-air convection	15	40°C	30.29 (0.25) ^{aA}	8.95(0.77) ^{aA,bA,cA}	1.12(0.00) ^{aA}	7.35 (1.92) ^{aA}
	15	80°C	30.04 (0.09) ^{aA}	7.87(0.01) ^{bA}	1.05(0.01) ^{bA}	2.74 (0.75) ^{bA}
	15	120°C	30.51 (0.17) ^{cA}	10.67 (0.59) ^{cA,cC}	1.08(0.01) ^{cA}	7.08 (0.36) ^{aA,cA}
	30	40°C	31.67 (0.24) ^{aB}	7.81 (1.28) ^{aB,aA}	1.10(0.00) ^{aB,aA}	3.12 (1.56) ^{aB,cB}
	30	80°C	31.97 (0.15) ^{aB}	7.10 (0.90) ^{aB,bA}	1.08 (0.02) ^{aB,bB}	1.97 (0.42) ^{aB,bA}
	30	120°C	32.28 (0.21) ^{cB}	12.59 (0.66) ^{cB,cA}	1.05 (0.00) ^{cB}	5.13 (1.57) ^{cB,cA}
	45	40°C	33.41 (0.10) ^{aC}	7.17 (0.10) ^{aC,cC,cA}	1.14 (0.00) ^{aC}	2.59 (0.81) ^{aC,aB}
	45	80°C	33.61 (0.09) ^{aC}	6.15 (0.41) ^{aC,bA}	1.12 (0.01) ^{aC,bC}	4.14 (1.18) ^{aC,bA}
	45	120°C	34.31 (0.10) ^{cC}	8.91 (0.48) ^{cC}	0.96 (0.01) ^{cC}	4.48 (1.59) ^{aC,cA}
Microwave ³	15	P4	30.53 (0.03) ^{dA}	8.31 (0.25) ^{dA}	1.14 (0.03) ^{dA}	7.85 (0.82) ^{dA}
	15	P6	30.19 (0.15) ^{dA,eA}	10.29 (0.47) ^{eA}	1.04 (0.01) ^{eA}	8.13 (0.18) ^{dA}
	15	P8	29.95 (0.07) ^{eA}	8.69 (0.22) ^{fA,dA}	1.07 (0.00) ^{eA,fA}	8.72 (0.71) ^{dA}
	15	P10	29.83 (0.13) ^{eA}	9.08 (0.31) ^{eA,dA,fA}	1.06 (0.00) ^{eA,eC}	7.29 (0.91) ^{dA}
	30	P4	32.57 (0.12) ^{dB}	8.04 (0.61) ^{fB,gB,dA}	1.08 (0.02) ^{dB,eB,gB}	5.13 (1.34) ^{dB}
	30	P6	31.97 (0.15) ^{dB,eB}	7.41 (0.25) ^{fB,gB}	1.10 (0.04) ^{eB}	6.88 (1.04) ^{dB}
	30	P8	32.00 (0.02) ^{eB}	8.83 (1.25) ^{fB,fA}	1.04 (0.03) ^{eB,gB,fA}	5.59 (1.03) ^{dB}
	30	P10	31.87 (0.35) ^{eB}	7.13 (0.09) ^{gB,eA}	1.03 (0.00) ^{gB,eC}	6.32 (0.71) ^{dB}
	45	P4	34.11 (0.16) ^{dC}	6.39 (0.41) ^{dC}	1.07 (0.02) ^{dC,dB}	3.73 (1.94) ^{dC}
	45	P6	34.20 (0.18) ^{dC,eC}	5.19 (0.02) ^{dC}	1.08 (0.00) ^{dC,eA,eB}	5.31 (2.31) ^{dC}
	45	P8	33.93 (0.08) ^{eC}	5.21 (0.17) ^{dC}	1.07 (0.00) ^{dC,fA}	4.69 (0.56) ^{dC}
45	P10	34.19 (0.27) ^{eC}	8.70 (0.73) ^{gC}	1.02 (0.01) ^{dC,eC}	3.64 (0.88) ^{dC}	
Microwave convection ⁴	15	C1	30.91 (0.30) ^{hA}	8.23 (0.06) ^{hA}	1.04 (0.02) ^{hA,kA}	7.24 (0.37) ^{hA}
	15	C2	30.30 (0.09) ^{hA}	9.85 (0.70) ^{iA,kA}	1.03 (0.01) ^{iA,kA}	7.40 (1.45) ^{hA}
	15	C3	30.47 (0.19) ^{hA}	9.78 (0.02) ^{jA,iA}	1.16 (0.02) ^{jA}	8.52 (1.58) ^{hA}
	15	C4	31.81 (0.31) ^{kA}	9.39 (0.08) ^{kA,jA}	1.08 (0.00) ^{jA,kA}	7.96 (0.70) ^{hA}
	30	C1	32.11 (0.04) ^{hB}	8.73 (0.22) ^{hB,hA}	1.01 (0.00) ^{hB,hA}	5.02 (1.69) ^{hB}
	30	C2	31.53 (0.42) ^{hB,kB}	6.21 (0.01) ^{iB}	1.10 (0.00) ^{hB,iA}	4.84 (1.37) ^{hB,iB}
	30	C3	32.16 (0.34) ^{hB}	7.93 (0.38) ^{hB,jB}	1.10 (0.07) ^{hB,jB,jA}	5.71 (0.88) ^{hB,jB}
	30	C4	31.29 (0.48) ^{kB,kA}	8.28 (0.28) ^{kB,jB}	1.02 (0.02) ^{hB,kA}	5.44 (1.56) ^{hB,kB}
	45	C1	33.36 (0.07) ^{hC,kC}	8.81 (0.24) ^{hC,hA}	1.02 (0.00) ^{hC,hA}	5.72 (1.87) ^{hC,hB}
	45	C2	34.73 (0.01) ^{iC}	6.16 (0.35) ^{iC,iB,kC}	1.07 (0.02) ^{hC,iA}	4.23 (1.38) ^{hC,iB}
	45	C3	34.57 (0.23) ^{iC}	5.93 (0.31) ^{jC,iC}	1.06 (0.01) ^{hC,jB}	2.47 (2.05) ^{hC,jB}
	45	C4	34.07 (0.27) ^{iC,kC}	5.30 (0.26) ^{kC}	1.06 (0.00) ^{hC,kA}	3.99 (1.17) ^{hC,kB}

Tukey's test at 5% significance level for the same CDS level at varying drying temperatures (a, b, c), microwave power (d,e,f,g), microwave convection setting (h,i,j,k) and for the same drying temperature at varying CDS levels (A,B,C). Values followed by the same set of letters are not significantly different.

¹ N=2; ² N=3; ³ P4 - 420 W; P6 - 676 W; P8 - 701 W; P10 - 805 W; ⁴ C1 - 130°C-30% power (303 W); C2 - 150°C-30% power (316 W); C3 - 160°C-30% power (327 W); C4 - 190°C-30% power (332 W)

setting (P4, 420 W) showed relatively higher protein content than those dried at P8 (701 W), and at P10 (805 W).

Table 3.7 The analysis of variance p-values obtained from testing mean differences, protein, lysine and acid detergent insoluble crude protein (ADICP) contents and total color difference (ΔE) of wheat DDGS samples. The samples were produced at varying levels of condensed distillers solubles (CDS) and dried under three methods. Significance level was set at 0.05

Drying Method	Source of variation	Protein content	ADICP content	Lysine content	ΔE
Forced-air convection	Drying air temperature	0.000	0.000	0.000	0.001
	CDS level	0.000	0.002	0.116	0.002
	Temperature * CDS level	0.091	0.040	0.000	0.007
Microwave	Microwave power	0.002	0.081	0.003	0.147
	CDS level	0.000	0.000	0.251	0.000
	Microwave power * CDS level	0.062	0.000	0.010	0.621
Microwave convection	Combination setting	0.242	0.000	0.018	0.871
	CDS level	0.000	0.000	0.172	0.000
	Combination setting * CDS level	0.000	0.000	0.100	0.198

Finally, in microwave convection-dried samples, the interaction effect of the microwave-convection combination setting and CDS level on protein content was significant (Table 3.7). Samples with 15% and 45% CDS had significantly higher protein content when dried at higher combination settings (Table 3.5). For samples with 30% CDS content, those dried under C1 (30% microwave power-130°C) and C3 (30% microwave power, 160°C) settings showed significantly higher protein content than at C4, the highest combination setting (30% microwave power-190°C).

In summary, the observed differences in the protein content of wheat DDGS samples were largely attributed to the level of CDS incorporation. Although drying conditions employed in this

study had also significantly affected protein content of samples, their effect was not as pronounced compared to CDS level. Strong linear relationships between CDS level and protein content were significant in all three sets of samples.

For the wheat DDGS sample sourced directly from a Saskatchewan ethanol plant, the average protein content was at 38.0%, dry basis (Table 3.4). This value was close to what was reported by Widyaratne and Zijlstra (2007) but was higher compared to the values obtained from the laboratory-prepared samples in this study (Tables 3.4 and 3.6). These differences between the protein content of the laboratory-prepared samples and the plant-sourced sample indicate that the source ethanol plant could have incorporated higher CDS levels than what were used in this study.

3.4.3 ADICP content

The average ADICP content of freeze-dried samples ranged from 5.69 – 6.95% of total crude protein and decreased as CDS level was increased (Table 3.4). The linear model (Eq. 3.9) was significant and explained about 81% of the total variation observed between ADICP content (% crude protein, dry matter basis) and CDS level (% WDGS mass). Decrease in ADICP content with increase in CDS level was expected because the wheat-based CDS component had lower ADF content (Table 3.5), from which the ADICP was derived.

$$\text{ADICP} = -0.429 * \text{CDS} + 7.555 \quad (3.9)$$

The ADICP content of samples dried under forced convection, microwave- and microwave convection methods (Table 3.6), however, did not exhibit similar strong linear trends as that observed in freeze-dried samples (Table 3.4). Lower coefficients of determination (R^2) were

obtained from the linear relationships between CDS level and ADICP content in forced air convection- ($R^2 = 0.14$), microwave- ($R^2 = 0.52$), and microwave convection-dried ($R^2 = 0.54$) samples. Only the linear relationships in microwave (Eq. 3.10) and microwave convection (Eq. 3.11) -dried samples were significant.

$$\text{Microwave:} \quad \text{ADICP} = -0.091 * \text{CDS} + 10.488 \quad (3.10)$$

$$\text{Microwave convection:} \quad \text{ADICP} = -0.092 * \text{CDS} + 10.644 \quad (3.11)$$

Analysis of variance results (Table 3.7) indicate that the interaction effects of CDS level with drying air temperature, microwave power level, and with microwave-convection combination settings significantly affected ADICP values. For samples dried under forced air convection, all blends dried at 120°C had significantly higher ADICP compared to those dried at 80°C (Table 3.6). Also, samples with 30% CDS had significantly higher ADICP content when dried at 120°C than at 40°C. In microwave-dried samples, those with 45% CDS content and dried at the highest microwave power level (801 W) showed significantly higher ADICP content compared to those dried at the lower microwave power settings (Table 3.6). Also, samples with 15% CDS that were dried at 60% power (676 W) had significantly higher ADICP compared to those dried at 40% power (420 W) and at 80% power (701 W). At 30% CDS, samples dried at 80% power (701 W) had significantly higher ADICP than those dried at 100% power (805 W). For samples dried under the microwave convection method, those with 45% CDS content and dried using the lowest microwave convection setting (30% power-130°C) registered significantly higher ADICP compared to those dried at higher settings (Table 3.6). At 30% CDS, samples dried using C1 (30% power-130°C) and C3 (30% power-160°C) settings were significantly higher in ADICP compared to those dried at the C2 (30% power-150°C) setting. At 15% CDS, samples dried at

the higher settings had significantly higher ADICP content compared to those dried using the lowest setting.

With respect to the commercial wheat DDGS sample, it was previously indicated that the ethanol plant could be incorporating higher CDS levels than what were used in the study, as seen from the higher protein content of the sample compared to those blended in the laboratory. Higher CDS level, however, did not translate to lower ADICP content in the plant-sourced sample. Its average ADICP content was 18.4% (dry basis) (Table 3.4), higher than what was observed in the laboratory-prepared samples (Tables 3.4 and 3.6). Higher ADICP content could be due to the higher drying air temperatures used in the ethanol plant, which increased the production of insoluble, heat damaged protein aggregates via the Maillard reaction.

These results suggest that unlike protein content, where CDS level was a major source of variation, ADICP content was significantly affected by both CDS level and drying conditions. This is seen from the lower CDS-ADICP R^2 values compared to the CDS-protein relationship and from the significant factorial interaction effects that were consistently observed across the three drying methods. Further, microwave- and microwave convection-dried samples showed significant, slightly stronger relationships with CDS level compared to forced air convection-dried samples.

3.4.4 Lysine content

Average lysine content of the freeze-dried samples ranged from 1.02 to 1.09% (dry basis), as shown in Table 3.4. Observed mean differences in lysine content were found to be significant. Samples with 45% CDS had significantly higher lysine content compared to those with 15% and 30% CDS. This was expected because higher CDS levels translate to higher protein content, and

thus, higher lysine content. The linear model (Eq. 3.12), which shows lysine content of freeze-dried samples (Lys, % dry matter) as a function of CDS content (% WDGS mass), was significant only at the 0.10 level.

$$\text{Lys} = 0.002 * \text{CDS} + 0.989 \quad (3.12)$$

Table 3.6 shows the lysine content of wheat DDGS samples dried under forced air convection, microwave, and microwave convection methods. Linear relationship between CDS level and lysine content was not significant in all three sets of samples. Table 3.7, however, shows that interaction between CDS level and drying conditions (drying air temperature, microwave power, and microwave convection setting) had significant effect on lysine content of all three sets of samples.

Under forced air convection, samples with 15%, 30%, and 45% CDS all showed significantly higher lysine content when dried at 40°C than at 120°C (Table 3.6). Among these, those with 45% CDS showed both the lowest and highest lysine contents when dried at 40°C and at 120°C, respectively. Those dried at 80°C also showed significantly higher lysine content compared to those dried at 120°C. Average lysine content in samples containing 30% and 45% CDS was not significantly different when dried at 40°C and at 80°C.

In microwave-dried samples, those dried at lower microwave power settings tend to have higher average lysine content (Table 3.6). At 15% CDS, samples dried using the lowest microwave power setting (P4, 420W) showed higher lysine content compared to those dried using the other 3 higher power settings. Samples dried at the P6 (676 W) power setting had significantly higher lysine content compared to those dried at the P8 (701 W) and P10 (805W) settings. At 45% CDS

content, samples dried using the P6 (676 W) power level showed significantly higher lysine content than the ones dried at P10 (805 W).

In microwave convection-dried samples, significant differences in average lysine content were only found in samples with 15% and 30% CDS. No significant difference in lysine content was found among samples with 45% CDS content. For samples with 15% CDS content, drying at the C3 (30% power-165°C) resulted to samples with significantly higher lysine content compared to those dried under C1 (30% power – 130°C), C2 (30% power – 150°C) and C4 (30% power – 190°C). At 30% CDS, samples dried at the C2 (30% power – 150°C) and C3 (30% power-165°C) settings had significantly higher lysine content compared to the other 2 combination settings.

The plant-sourced DDGS sample gave an average lysine content of 0.76% (dry basis) (Table 3.4), close to the 0.72% (dry basis) lysine content reported by Widyarante and Zijlstra (2007). The laboratory-prepared samples had much higher lysine content of 1.01 – 1.16% (dry basis) than the plant-sourced sample. Higher protein content in the plant-sourced sample did not translate to higher lysine content. The use of elevated temperatures during the drying process in the ethanol plant had affected the sample's lysine content.

3.4.5 Color

Table 3.4 shows the color parameters of the freeze-dried samples at varying CDS levels. Dominant wavelengths were between 580.17 – 581.22 nm, confirming the brown color of the samples. The following linear models (Eqs. 3.13 and 3.14) depict the significant relationship between CDS level (C, % mass) and the color parameters L (lightness) and a (redness).

$$L = -0.152 * CDS + 55.535 \quad R^2 = 0.87 \quad (3.13)$$

$$a = 0.038 * CDS + 4.943 \quad R^2 = 0.85 \quad (3.14)$$

Samples with 15% CDS were significantly lighter (higher L values) and less red (lower a values) compared to those with 45% CDS level. Mean differences in yellowness (color parameter b) were not significant. These observed differences across the CDS levels are attributed to differences in the CDS and WDG color parameters. Freeze-dried CDS were darker and redder ($L = 45.7$, $a = 5.3$, $b = 16.9$) compared to the WDG sample ($L = 52$, $a = 3.2$, $b = 15.6$) (Mosqueda and Tabil 2011b). Thus, higher CDS level in the blends resulted to darker samples.

Further, wheat DDGS samples generated from freeze drying were also significantly lighter, less red, and more yellow compared to the sample obtained from a Saskatchewan wheat ethanol plant (Table 3.6). Observed differences are attributed to the incorporation of higher amount of CDS during blending and use of high drying air temperatures in the ethanol plant.

Table 3.6 shows the total color difference (ΔE) values of the samples dried under the three methods. In general, as CDS level was increased, ΔE tended to decrease. Eqs. 3.15 to 3.17 show the significant linear relationships between CDS level (% WDGS mass) and ΔE of samples generated under the three drying methods. Microwave- ($R^2 = 0.63$) and microwave convection-dried ($R^2 = 0.53$) samples showed higher R^2 values compared to the forced-air convection dried samples ($R^2 = 0.15$).

$$\text{Forced-air convection:} \quad \Delta E = -0.066 * CDS + 6.281 \quad (3.15)$$

$$\text{Microwave:} \quad \Delta E = -0.122 * CDS + 9.763 \quad (3.16)$$

$$\text{Microwave convection: } \Delta E = -0.123 * \text{CDS} + 9.394 \quad (3.17)$$

Under forced-air convection dried samples, interaction between CDS and drying air temperature had a significant effect on ΔE (Table 3.7). Samples with 15% CDS that were dried at 40°C and at 120°C had significantly higher ΔE values compared to those dried at 80°C (Table 3.6). There was no significant difference in the mean ΔE values between those dried at 40°C and at 120°C (Table 3.6). This suggests that drying at low temperature (40°C) and longer exposure time (3 h) produced a similar effect on color as drying at a higher temperature (120°C) and shorter exposure time (30 min). For samples with 30% CDS, drying at 120°C produced significantly higher ΔE values than those dried at 80°C. For samples with 45% CDS, mean differences in ΔE values across the three temperature levels were not significantly different.

In microwave and microwave convection-dried samples, only CDS level had a significant effect on ΔE (Table 3.7). Both sets of samples with 15% CDS had significantly higher ΔE than those with 30% and 45% CDS (Table 3.6). Mean ΔE difference between samples with 30% and 45% CDS was significant in microwave-dried samples but was not significant in microwave convection-dried samples (Table 3.6).

With respect to the individual color parameters, L values of forced-air convection dried samples were significantly affected by the CDS level-drying air temperature interaction, just like the ΔE values. For example, samples with 15% CDS and dried at 40°C and 120°C were significantly darker than those dried at 80°C. Similarly, those with 30% CDS and dried at 120°C was significantly darker than those dried at 80°C. Further, drying air temperature significantly affected redness (color parameter a) of the forced-air convection dried samples. Those dried at

120°C were significantly redder compared to those dried at 40°C and at 80°C. Both drying air temperature and CDS level significantly affected the yellowness parameter (b).

Similar to what was observed in the ΔE values, CDS level had a significant effect on the L, a, and b values of microwave convection-dried samples as well as on the L values of microwave-dried samples. Microwave- and microwave convection-dried samples with 30% CDS level, for example, were significantly lighter (higher L values) than those with 45% CDS.

3.4.6 Optimum conditions

Table 3.8 shows that blending CDS at 45% (by mass) and drying at lower temperature (80°C), lower microwave power level (60% power or 676W), and lower microwave-convection setting (C2 or 150°C, 30% power level) produced the highest protein and lysine contents and lowest ADICP content among the treatment combinations investigated. The drying conditions that maximized the color parameter L (lightness) and minimized redness (color parameter a) and yellowness (color parameter b) are also presented. These tabular results highlight the importance of blending higher amount of CDS but using lower drying air temperature, microwave power, and microwave-convection settings during drying to minimize the incidence of heat-damaged, dark-colored wheat DDGS.

3.4.7 Linear correlations between properties

Table 3.9 shows the significant linear property correlations that were derived from the forced-air convection-, microwave-, and microwave convection-dried samples. Some of these correlations were already observed in the previous sections. These include: (1) positive relationship between CDS level and protein content in all three sets of samples; (2) negative relationship between

Table 3.8 Optimum treatment combinations, in terms of protein, lysine, and acid detergent insoluble crude protein (ADICP) contents, and color for wheat distillers dried grain with solubles (DDGS) samples. Wheat DDGS samples were produced with varying levels of condensed distillers solubles (CDS) and dried under 3 methods

Parameter	Forced air convection	Microwave	Microwave convection
1. Protein quality			
CDS level	45%	45%	45%
Nominal setting of dryer	80°C, <8% RH	P6 (676 W)	C2 ²
Protein, % dry basis	33.61	34.20	34.73
Lysine, % dry basis	1.12	1.08	1.07
ADICP, % of protein dry basis	5.87	5.19	6.16
2. Color¹			
CDS level	45%	15%	45%
Nominal setting of dryer	40°C, <8% RH	P8 (701 W)	C4 ³
L	46.66	44.48	44.97
a	6.35	6.61	6.53
b	15.10	15.1	14.79

¹color parameters L, a, and b represent lightness, redness, and yellowness, respectively; ²C2 - 150°C-30% power (316 W); ³C4 - 190°C-30% power (332 W)

Table 3.9 Significant linear correlations (p < 0.05) found in forced air convection-, microwave-, and microwave convection-dried samples of wheat-based distillers dried grain with solubles (DDGS) prepared with varying levels of condensed distillers solubles (CDS). Correlations were based on the mean values of the properties

Forced-air convection dried samples		Microwave-dried samples		Microwave convection-dried samples	
Relationship	R	Relationship	R	Relationship	R
T x a	0.842	CDS x a	0.641	CDS x ΔE	-0.878
T x Lys	-0.721	CDS x ΔE	-0.637	CDS x Protein	0.909
CDS x Protein	0.980	CDS x Protein	0.990	CDS x ADICP	-0.730
L x b	0.914	CDS x ADICP	-0.740	L x b	0.764
a x Lys	-0.712	L x b	0.652	a x b	0.811
		L x ADICP	0.636	ΔE x Protein	-0.809
		a x Protein	0.615	ΔE x ADICP	0.852
		ΔE x Protein	-0.654	Protein x ADICP	-0.727
		Protein x ADICP	-0.718		
		Lys x MW power	-0.637		

ADICP - acid detergent insoluble crude protein; a - redness color parameter; b - yellowness color parameter; ΔE - total color difference; L - lightness color parameter; Lys - lysine content; MW - microwave; R - correlation coefficient; T - drying air temperature;

CDS level and ADICP content and between CDS level and ΔE in microwave- and microwave convection-dried samples, respectively; (3) negative relationship between lysine content and drying air temperature, and between lysine content and microwave power; and (4) positive relationship between redness (color parameter a) and drying air temperature.

The other significant correlations observed could be derived from the relationships of a particular property with other properties. For example, negative correlations between ΔE and protein content and between protein and ADICP content of microwave and microwave convection-dried samples were derived from the individual relationships of CDS level with protein, with ADICP, and with the total color difference, ΔE . As CDS level increased, protein content increased while ADICP and ΔE decreased. Thus, protein and ΔE , as well as protein and ADICP, were negatively correlated. The same explanation is offered for the positive relationship between redness and protein content in microwave-dried samples. As CDS level was increased, protein content and redness of microwave-dried samples increased, thus, explaining the positive relationship between redness and protein content.

3.4.7.1 Lysine content and color

Table 3.9 further shows significant negative correlation between lysine content and the color parameter a in forced air convection-dried samples. The linear equation (Eq. 3.18) was significant and was able to explain about 51% of the total variation observed between redness (color parameter a) and lysine content (Lys, % dry basis).

$$\text{Lys} = -0.134a + 1.959 \quad R^2 = 0.51 \quad (3.18)$$

No consistent trend was found between lysine content and ΔE . Although a significant negative correlation existed between the redness parameter and lysine content (Table 3.9), this was not detected when color was expressed in terms of ΔE . Differences in the lightness parameter, rather than in the redness or yellowness parameter, accounted for a major proportion of the ΔE values. There were a number of negative correlations found between lysine content and ΔE /color parameter L but these were not statistically significant. These include samples with 45% CDS and dried under forced-air convection and microwave convection methods, those with 30% CDS and dried under forced-air convection and microwave methods, and those with 15% CDS and dried under microwave method.

Strength of the color and lysine content correlations could be affected by the extent of development of Maillard reactions during the drying process as well as by the chemical composition of the samples. During the initial stages of Maillard reaction, colorless products are formed (Hodge 1953) but some amino acids, like lysine, may already be unavailable but may or may not be detected in chemical analyses (Mavromichalis 2001). At later stages of the reaction, lysine can be destroyed, manifested in its marked decrease during chemical analysis (Mavromichalis 2001), and highly colored polymers are also formed (Hodge 1953; Yaylayan and Roberts 2001). Thus, during the initial stages of Maillard reaction, correlations between lysine and color may not be as strong compared to the advanced stages. Further, it was previously shown in this study that blends with higher CDS content tended to have higher protein content compared to blends with lower CDS content. With higher protein content, more lysine would be available to react with the reducing sugars present during the drying process.

3.4.7.2 ADICP content and color

Table 3.9 also shows significant positive correlation between lightness parameter (L) and ADICP and between total color difference (ΔE) and ADICP (% of total crude protein, dry basis) in microwave- and microwave convection-dried samples, respectively. The equivalent linear relationships are presented as Eq. 3.19 and 3.20, respectively.

$$\text{ADICP} = 1.094L - 41.50 \quad R^2 = 0.40 \quad (3.19)$$

$$\text{ADICP} = 0.735\Delta E + 3.64 \quad R^2 = 0.73 \quad (3.20)$$

Higher levels of ADICP are suggestive of heat damage and samples, therefore, were expected to be darker. This was not depicted in microwave-dried samples, where the color parameter L was positively correlated with ADICP (Table 3.9 and slope of Eq. 3.20). Reduced color formation in microwave heating/drying systems had been attributed to two factors: (1) the lack of hot dry air surrounding the product, thus preventing surface dehydration crucial for color formation by Maillard reaction; and (2) shorter exposure time, which could prevent the completion of slower or complex Maillard reactions responsible for color formation (Yaylayan and Roberts 2001). Samples from forced-air convection and microwave convection drying, on the other hand, consistently showed negative correlations between L and ADICP, although these were not statistically significant.

The positive linear relationship shown in Eq. 3.20 for microwave convection-dried samples results from relationships between CDS level and ADICP (Eq. 3.11) and between ΔE and CDS level (Eq. 3.17). As CDS level increased, ADICP content and ΔE decreased. Thus, ADICP and ΔE , in turn, are positively correlated.

3.5 Conclusions

This study examined the effect of condensed distillers solubles (CDS) level and drying methods on the protein quality of wheat distillers dried grain with solubles (DDGS) and verified whether wheat DDGS color was indicative of its protein quality. Protein quality was expressed in terms of acid detergent insoluble crude protein (ADICP), and lysine contents.

Page model adequately described the drying behavior of wheat WDGS with varying CDS level under forced-air convection, microwave, and microwave convection methods. Drying air temperature, microwave power, and microwave convection combination settings, in general, showed stronger influence on the drying rate constant than CDS level.

Effective moisture diffusivity linearly decreased as CDS level was increased. This could be due to differences in porosity and chemical composition. Porosity decreased as CDS level in the blend increased. A less porous structure presents less resistance to moisture movement. Effective moisture diffusivity linearly increased with drying temperature and microwave power level.

Increasing the level of CDS incorporation during the blending process increased protein and lysine content, decreased ADICP content, and darkened the product. Use of higher air temperature, microwave power, and microwave-convection setting during drying resulted to higher ADICP in all samples. Drying at higher air temperature and microwave power level also resulted to lower lysine content. Higher drying air temperature also increased the redness of the wheat DDGS. On the other hand, CDS level showed a stronger influence on color than the microwave power and microwave-convection setting used during drying.

Significant relationships between protein quality and color were observed in forced-air convection-, microwave- and microwave convection-dried samples. Lysine content decreased when redness of forced-air convection dried samples increased. ADICP content increased when total color difference of microwave convection-dried samples decreased. In microwave-dried samples, ADICP content increased when the lightness color parameter increased.

The potential of microwave-assisted methods need to be further explored, from the standpoint of minimizing both protein damage and energy consumption during drying. This study illustrated that, with the right selection of settings, microwave power and microwave convection drying can achieve desirable protein quality associated with low temperature drying at much shorter times.

Although the use of low temperature settings is an essential requisite to maintain protein quality, it is not yet a feasible industrial approach for drying bulk quantities of high moisture feedstock. The drying methods used in the study are also not available on an industrial scale.

Chapter 4

Effect of condensed distillers solubles and drying temperature on the physico-chemical characteristics of laboratory-prepared wheat distillers grain with solubles

A similar version of this chapter has been published in the International Journal of Agricultural and Biological Engineering:

- Mosqueda, M.R., L.G. Tabil, and K. Muthukumarrapan. 2013. Effect of condensed distillers solubles and drying temperature on the physico-chemical characteristics of laboratory-prepared wheat distillers grain with solubles. *International Journal of Agricultural and Biological Engineering* 6(2): 73 – 84.

Contribution of the PhD Candidate

The PhD candidate did the literature review, planning and execution of experiments, data analysis, and manuscript preparation under the guidance of her research supervisor, Lope G. Tabil. She was assisted by Shivani Trivedi and Dallas Nelson, undergraduate student research assistants, in some aspects of sample preparation and data collection, respectively. She was trained by Irene Northey of the Department of Animal and Poultry Science and Angela Hennings of the Feeds Innovation Institute in proximate analysis and by Yijing Wang, a graduate student of South Dakota State University, in using the powder characteristics tester. Professor Kasiviswanath Muthukumarappan of South Dakota State University assisted her in accessing the bioprocessing laboratory of the USDA North Central Laboratory for flow properties determination. He also provided editorial advice during manuscript preparation.

Contribution of the Paper to the Overall Study

This paper addresses the second main objective of the PhD research project: assessment of the effect of drying conditions and condensed distillers solubles (CDS): wet distillers grain (WDG) blending proportion on the physico-chemical characteristics of wheat distillers dried grain with solubles (DDGS). While wheat CDS:WDG blending proportion had been mentioned in animal feed-related studies as one of the possible causes of DDGS variability, there were no studies found that quantified their effect on the product's characteristics. Most of the related studies found focused on corn DDGS. Since there are inherent compositional differences between the two grains, varying the drying and blending parameters during DDGS production might not produce similar effect on their respective DDGS physical and chemical characteristics. This paper provides a comprehensive look at the effect of drying temperature and CDS:WDG blending proportion (also referred to as CDS level in this report) on five sets of properties: (i) physical attributes; (ii) flow properties; (iii) compression characteristics; (iv) frictional properties, and (v) thermal properties. Information derived from this study would be useful to ethanol plant managers and quality assurance officers, personnel involved in storage and handling, including transportation to market destinations, and to animal feed processors and nutritionists.

4.1 Abstract

Samples of wheat distillers grain with solubles were prepared at 15%, 30%, and 45% CDS level, by mass, and dried under 40°C, 80°C, and 120°C to examine the effects of CDS level and drying temperature on their chemical, physical, flow, compression, thermal, and frictional properties. As CDS level increased, protein and ash contents increased while fat and fiber contents

decreased. Fat and acid detergent fiber contents were also markedly affected by drying temperature. While CDS level, drying temperature, and their interaction significantly affected a number of the physical properties, results suggest that CDS level had a stronger influence. Samples with high CDS level, for example, were significantly finer, denser, less flowable, and less dispersible than those with lower CDS. These samples also had significantly higher thermal diffusivity and coefficient of internal friction and produced pellets with higher failure stresses than those with lower CDS. Their pellet density increased with CDS level and was also significantly affected by drying temperature. Further, the samples were classified as fairly flowable and floodable and their compression characteristics were adequately described by the Kawakita-Ludde model.

4.2 Introduction

Product variability is one of the challenges that currently confront wheat DDGS production in western Canada (University of Saskatchewan 2010). Nuez-Ortin (2010), for example, found significant nutrient variability in wheat DDGS samples obtained from two Saskatchewan plants and stressed the importance of product consistency not only in formulating more accurate feed rations but also in improving the market prospects of wheat DDGS. Blending of CDS and WDG has been identified as among the major causes of variation in the physico-chemical characteristics in both corn and wheat DDGS. While a number of laboratory- (Ganesan et al. 2008a, 2008c; Bhadra et al. 2009b, 2009c, 2011a, 2012) and plant- (Kingsly et al. 2010; Clementson and Ileleji 2012) scale investigations had studied its effect on the physico-chemical characteristics of corn DDGS, there is still very limited information on how CDS:WDG blending proportion affect wheat DDGS properties.

In a laboratory-scale investigation, Ganesan et al. (2008a) reported that soluble level and moisture content had significant effects on protein content, color, aerated and packed bulk densities, compressibility, dispersibility, flowability, and floodability of corn DDGS. As soluble level increased, protein content, and the color parameter L decreased while the color parameter increased. They also reported significant differences in particle size as soluble level was varied but they did not observe distinct trends. In their study of the flow properties of laboratory-scale produced corn DDGS using the Jenike shear tester, they also reported significant soluble and moisture content effects but did not observe any definitive trend (Ganesan et al. 2008c). These two studies defined “solubles” as the “non-water portion of CDS that passed through a filter media”. Bhadra et al. (2009b; 2009c; 2011a; 2012) also investigated the effect of CDS level (10%, 15%, 25% w.b.), drying temperatures (100°C, 200°C, 300°C), and cooling temperatures (-12°C, 25°C, 35°C) on the drying kinetics, glass transition temperature, and on physical and flow characteristics of corn DDGS produced in the laboratory-scale. They reported significant main and interaction effects on most of the physical and flow properties (Bhadra et al. 2009b). Compared to CDS level, drying temperature was seen to be the more important factor that affected drying rate (Bhadra et al. 2011a) and flowability (Bhadra et al. 2009b; 2011a). Higher drying temperature increased drying rate and resulted to better flowability. Cooling temperature levels did not produce significant differences on five important flow properties (Bhadra et al. 2009b).

Kingsly et al. (2010) and Clementson and Ileleji (2012) also conducted plant-scale investigations on this subject. Both these corn DDGS studies reported increase in particle size, bulk density, and particle density as CDS level was increased. They attributed the increase in particle size to the agglomeration of smaller particles with CDS acting as a binding agent (Kingsly et al. 2010;

Clementson and Ileleji 2012). Kingsly et al. (2010) further reported that as CDS level increased, the color parameter, fat, ash, sugars, and glycerol content, increased while the color parameter L, crude protein, acid detergent fiber (ADF), and neutral detergent fiber (NDF), on the other hand, decreased. Amino acids lysine, methionine, threonine and tryptophan also decreased, indicating higher concentration of amino acids in the WDG than in the CDS component (Kingsly et al. (2010). Clementson and Ileleji (2012) also reported that particle porosity, pore volume and effective bulk porosity decreased as CDS level increased. They did not find significant correlation between CDS level and the shape functions.

Inherent compositional differences between corn and wheat kernels and subsequently between their corresponding CDS and WDG fractions (Table 4.1) necessitate separate investigations for wheat DDGS. Corn-based CDS, for example, had lower protein and fiber but higher ash and fat content compared to the WDG fraction. In contrast, wheat-based thin stillage, from which the CDS component is derived, showed higher protein and ash but lower fat content compared to the WDG fraction (Ojowi et al. 1996; 1997). These compositional differences could translate to differences in physico-chemical characteristics. The overall objective of this study, therefore, was to examine the effect of CDS:WDG blending proportion on the physico-chemical characteristics of laboratory-prepared wheat DDGS.

Table 4.1 Comparative chemical composition of corn and wheat grain, wet distillers grain(WDG), thin stillage, and condensed distillers solubles (CDS), % dry matter basis

Sample	Crude protein	Ash	Crude fat	NDF ^a	ADF ^b
Corn-based					
Grain ^c	10.13	1.73	4.59	14.47	3.66
WDG ^{d,e}	34.4-35.0	2.0-2.35	10.9-14.0	39.0	11.9-17.2
CDS ^{d,f}	22.4-23.0	6.6-11.6	21.6-34.4	3.6-4.9	2.92
Wheat-based					
Grain ^c	14.28	2.12	1.91	17.22	3.68
WDG ^g	26.4	2.7	6.6	74.9	24.1
Thin stillage ^{h,i}	45.7-48.5	8.0-8.3	9.63-13.6	34.0-34.5	3.4-4.0
Wet distillers solubles ^j	32.6	8.9	5.7	2.3	

Note: ^aNDF means neutral detergent fiber; ^bADF means acid detergent fiber; ^cNuez-Ortin 2010; ^dCao et al. 2009; ^eKim et al. 2008; ^fFron et al.1996; ^gOjowi et al. 1997; ^hOjowi et al. 1996; ⁱMustafa et al. 1999; ^jPedersen and Lindberg 2010.

4.3 Materials and Methods

4.3.1 Preparation of WDGS samples

CDS and WDG were obtained from a south Saskatchewan fuel ethanol plant, placed in tightly sealed bins, and stored in a -16°C freezer. Five hundred grams to one kilogram batches of wet distiller's grain with solubles (WDGS) were prepared by mixing thawed CDS and WDG at three ratios, by mass: 15:85, 30:70, and 45:55 using a 6-speed Toastmaster hand mixer (model Toastmaster Inc., China) for 15 to 30 min at medium speed. These blends are subsequently referred to in this paper as 15%, 30%, and 45% CDS, respectively. Prepared WDGS were placed in sealed plastic bags and stored in the -16°C freezer until these were used.

4.3.2 Drying the WDGS

Bags of frozen WDGS were thawed overnight and were placed in the sample preparation room for equilibration with room temperature (22-24°C) prior to the thin layer drying runs. Samples

were dried using the forced air convection method until moisture content reached 8% (w.b.). The drying system consisted of an air conditioning unit equipped with humidity and temperature sensors, a vane-axial circulating fan, a drying chamber with three wires, scale-mounted trays, and a duct system. Kashaninejad and Tabil (2004) and Kashaninejad et al. (2007) provided a detailed description of the drying system. Drying air temperature was set at three levels (40°C, 80°C, 120°C) while air velocity and relative humidity were set at 0.7-0.8 m/s and below 8%, respectively. The temperature, relative humidity, air velocity and weight monitoring devices of the drying system were connected to a computer installed with LabView 8.2 (National Instruments, Austin, TX) software for data capture. Although the temperature levels used in this study were considerably lower (40-120°C) than those employed in the source ethanol plant (dryer inlet and outlet temperatures of 225-275°C and 110-130°C, respectively) because of laboratory equipment constraints, a previous study had indicated that protein quality changes were already detected at these levels (Mosqueda et al. 2013a). Thus, changes in other physico-chemical characteristics could also be discernible at these levels.

4.3.3 Chemical composition

The chemical composition of both wet and dried samples was determined. Wet samples (WDGS, CDS, and WDG) were freeze-dried using the Labconco FreeZone Freeze Dry System (Labconco Corp., Kansas City, MO) prior to the proximate analysis runs. All dried samples were ground using a Thomas-Wiley knife mill (Thomas Scientific, Swedesboro, NJ) equipped with a 1.0 mm screen.

Moisture content was determined using the AOAC Official Method 920.36 (AOAC 2003a). Crude protein was estimated using the Kjeldahl method, AOAC Official Method 984.13 (AOAC

2003b). Crude fat was determined using the Goldfisch fat extractor (Labconco Corporation, Kansas City, MO), following the AOAC Official Method 920.39 (AOAC 2003d), with anhydrous diethyl ether as extraction solvent. Crude ash was determined using AOAC Official Method 942.05 (AOAC 2003e), while ADF and NDF were estimated through AOAC Official Method 973.18 (AOAC 2003c) and through the method laid out by van Soest et al. (1991), respectively. A lamb starter feed sample (AAFCO 0728) was used as a check sample. The proximate analysis was conducted in duplicate runs. Figure 4.1 shows the fat and fiber extraction laboratory setup.



Fig. 4.1 Laboratory setup for fat (a) and fiber (b) extraction.

4.3.4 Physical properties

Dried samples were passed through a Thomas-Wiley mill (Thomas Scientific, Swedesboro, NJ), in batches of 150-200 g for two minutes to generate the bulk material. The mill was equipped with a 2.68 mm screen, a size about four times larger than the observed geometric mean diameter of commercial wheat DDGS samples. While grinding the samples may not be reflective of the ethanol plant-generated bulk material, use of the same sample amount range during grinding,

grinding time, and screen size, still enabled evaluation of the effect of CDS level on wheat DDGS physical properties.

Density, particle size and size distribution, flow, compression, frictional, and thermal properties of forced-air convection-dried samples were determined. Property measurement was conducted in duplicate runs, unless otherwise stated. Moisture content of samples ranged about 8%-9% (w.b.).

4.3.4.1 Particle size, density, and color

Sieve sizes of 12, 20, 30, 40, 50, 60, 70, 80, 100, 140, 200, and 270 and a Ro-tap sieve shaker (Tyler Manufacturing, Mentor, OH) were used for particle size analysis, following ANSI/ASAE S319.4 (ASAE, 2008). The calculated geometric mean diameter was used to represent particle size in this study.

To determine bulk density, the sample was placed on a funnel and was allowed to freely flow into a 0.5 L steel cup (SWA951, Superior Scale Co. Ltd., Winnipeg, MB). The cup contents were leveled using a steel rod and weighed. Bulk density was calculated by dividing sample mass contained in the cup with the cup volume. Particle density was determined using a gas multipycnometer (QuantaChrome, Boynton Beach, FL). Bulk porosity (ε), expressed as a percentage, was determined as a function of bulk (ρ_b) and particle densities (ρ_p) using Eq. 4.1 below.

$$\varepsilon = 1 - \frac{\rho_b}{\rho_p} \quad (4.1)$$

4.3.4.2 *Flow properties*

The Carr flowability and floodability indices were determined by measuring seven properties using a Hosokawa Micron Powder Tester PT-R (Hosokawa Micron Corp., Osaka, Japan), shown in Fig. 4.2. Each property measurement was assigned an index value, ranging from 0 to 25, depending on the point score classification system developed by Carr (1965).

The flowability index, which ranged from 0 (very, very poor flowability) to 100 (excellent flowability), was derived from the sum of the individual index values of compressibility, angle of repose, angle of spatula, and uniformity coefficient. The floodability index, which also ranged from 0 (will not flood) to 100 (very floodable), was derived in a similar manner, using the index values of flowability, angle of fall, angle of difference, and dispersibility. Detailed description of each flow property and its measurement was presented by Carr (1965), Hosokawa Micron Corp. (n.d.), and Ganesan et al. (2008a). The Hosokawa Micron Powder Tester had been used in determining the flow properties of corn DDGS (Ganesan et al. 2008a; 2008c; Bhadra 2009b) and in other materials (Hosokawa Micron Corp. n.d.) with average particle sizes larger than wheat DDGS.

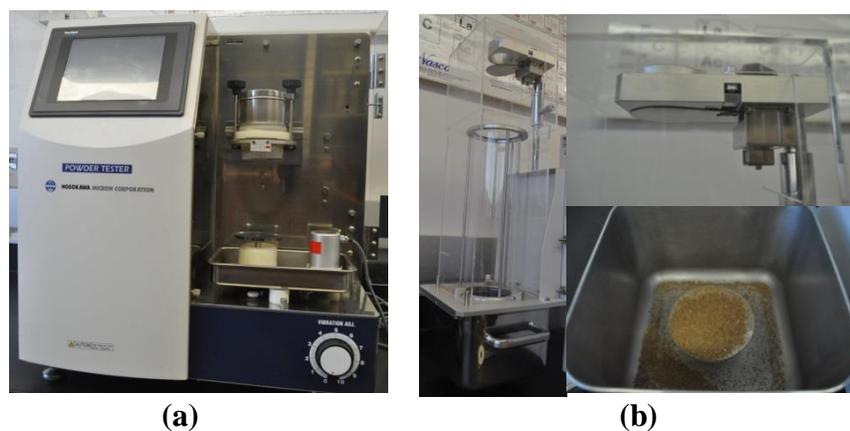


Fig. 4.2 The Hosokawa Micron Powder Tester PT-R (a), with the setup for dispersibility (b).

4.3.4.3 *Thermal properties*

Thermal conductivity and thermal diffusivity of dried samples at ambient room condition were determined using a KD 2 Thermal Properties Analyzer (Decagon Devices, Inc., Pullman, WA). Distilled water and 1.5% agar solution (w/v) were used as check samples. Bulk density was determined immediately before the thermal properties measurement, using the same procedure previously outlined. At low bulk densities, the volume of pores is greater, thus, the thermal conductivity would be lower because air has very low thermal conductivity. At higher bulk densities, porosity is decreased, thus, resulting in higher thermal conductivities.

4.3.4.4 *Compression characteristics*

An Instron Model 3366 testing machine (Instron, Norwood, MA) (Fig. 4.3a), fitted with 10 kN load cell and a 6.30 mm plunger, was used to compress 1 g samples placed inside a cylindrical die (6.35 mm diameter and 135.34 mm length) that was constantly heated at 90°C . The die and plunger assembly is shown as Fig. 4.3b. Crosshead speed was set at 30 mm/min. When the preset compressive load of 4 400 N was reached, the plunger remained in its place for 1 min before ejecting the pellet. The test was conducted in five replicates. Force, displacement, and time data were directly logged into the computer. Experimental data generated were fitted to two models: Jones (Eq. 4.2) (Mani et al. 2004; Emami and Tabil 2008) and Kawakita and Ludde (Eq. 4.3) (Emami and Tabil 2008). The most suitable model was chosen using R^2 and MSE as criteria.

$$\ln \rho = m \cdot \ln P + r \quad (4.2)$$

$$\frac{P}{C} = \frac{1}{d \cdot f} + \frac{P}{d} \quad (4.3)$$

where, $C = \frac{V_0 - V}{V_0}$; ρ is bulk density; P is the applied compressive pressure; m and r are the model parameters; C is the degree of volume reduction; V_0 is the volume of compact at zero pressure; V is volume of compact at pressure P (MPa); d and f are the model parameters. Model parameter d is equal to the initial porosity of the sample while the reciprocal of the model parameter f (f^{-1}) is related to failure stress (MPa) of the pelleted sample.

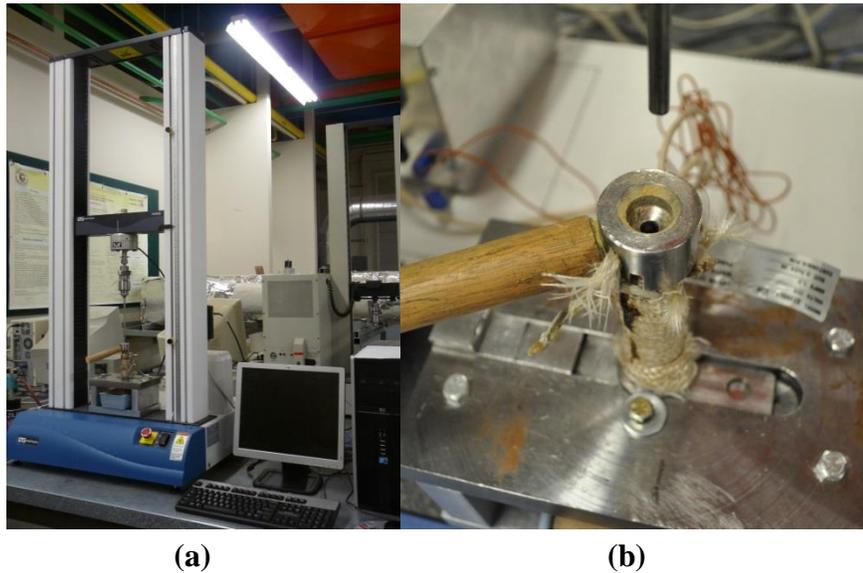


Fig. 4.3 The Instron model 3366 (a) and the cylindrical die and plunger assembly (b) used in producing wheat DDGS pellets.

Pellet density was determined by measuring the mass, length, and diameter of the resulting pellet immediately after it was extruded from the die. Specific energy consumption during compression and extrusion was estimated by calculating the area under the corresponding force-displacement curve using the trapezoidal rule and dividing it by the pellet mass.

4.3.4.5 Frictional properties

The coefficient of internal friction and cohesion were determined using the Wykeham Farrance shear box apparatus (Wykeham Farrance International Ltd., Slough, U.K.), as described in Mani et al. (2004) and Emami and Tabil (2008). Maximum shear stress was determined at four normal loads (200, 400, 600, and 800 N) (Emami and Tabil 2008). Values for the angle of internal friction and cohesion were estimated through regression analysis of experimental data using Equation (4.4) (Peleg et al. 1973), where τ is the shear stress (Pa), σ is the normal stress (Pa), θ is the angle of internal friction and C_c is the cohesion (Pa).

$$\tau = \sigma \tan \theta + C_c \quad (4.4)$$

4.3.5 Statistical analysis

The general linear model univariate procedure in SPSS 14.0 for Windows (SPSS Inc., Chicago, IL) was used to test the effect of CDS level and drying temperature. Tukey's test was used to further evaluate statistically significant main effects and interactions. Linear regression analysis was conducted to determine the relationship between CDS level and the chemical constituents of freeze-dried DDGS samples. All tests were conducted at the 0.05 significance level.

4.4 Results and Discussion

4.4.1 Chemical composition

4.4.1.1 Freeze-dried samples

Table 4.2 shows the proximate composition of freeze-dried wheat WDG, CDS, and DDGS samples at varying CDS level. The CDS component had higher crude protein and ash content

Table 4.2 Chemical composition¹ of freeze-dried, wheat-based wet distillers grain (WDG), condensed distillers solubles (CDS), and distillers dried grain with solubles (DDGS) samples. Values in parentheses represent standard deviation ($N = 2$)

Sample ²	Moisture/%, w.b.		Dry matter ³ /%				
	Initial	Freeze-dried	Protein	Ash	Fat	NDF	ADF
WDG	66.82 (0.24)	16.24 (0.29)	28.84 (0.60) ^a	2.90 (0.02) ^a	6.71 (0.04) ^a	77.53 (0.24) ^a	18.39 (0.39) ^a
CDS	75.52 (0.26)	18.98 (1.93)	45.82 (0.39) ^b	9.30 (0.02) ^b	2.21 (0.04) ^b	18.14 (0.29) ^b	6.66 (0.08) ^b
DDGS							
15% CDS	67.71 (0.26)	6.81 (0.31)	29.02 (0.24) ^a	3.56 (0.03) ^a	5.72 (0.13) ^a	60.45 (0.38) ^a	18.61 (0.03) ^a
30% CDS	69.60 (0.40)	8.08 (0.23)	30.70 (0.11) ^b	4.05 (0.01) ^b	5.59 (0.04) ^{a,b}	55.00 (0.03) ^b	17.48 (0.13) ^b
45% CDS	71.44 (1.04)	11.14 (0.25)	33.74 (0.00) ^c	4.88 (0.02) ^c	5.35 (0.01) ^b	51.62 (0.16) ^c	15.58 (0.45) ^c
Plant-sourced	13.32 (0.23)	-	38.81 (0.14)	7.10 (0.01)	4.89 (0.18)	46.55 (0.80)	17.45 (0.27)

Note: ¹ Values with the same superscript letter, within each sample category, are not significantly different at the 0.05 level;

² Except for the plant-sourced wheat DDGS sample, all other samples were freeze-dried prior to proximate analysis;

³ NDF means neutral detergent fiber; ADF means acid detergent fiber.

but lower fat and fiber content compared to WDG. Ojowi et al. (1996; 1997) also reported similar trends, wherein thin stillage, from where the CDS component was derived, had higher crude protein and lower NDF and ADF than the WDG fraction (Table 4.1). Differences in the CDS and WDG composition are attributed to differences in the composition of the wheat kernel components that comprised these two fractions. The bran, which is high in fiber, predominantly comprised the WDG fraction while endosperm and germ particles made up the CDS fraction. The chemical constitution of wheat DDGS was not similar to those reported for corn DDGS (Ganesan et al. 2008a; Kingsly et al. 2010; Clementson and Ileleji 2012; Cao et al. 2009) due to the basic compositional differences between corn and wheat grains and between their respective CDS and WDG fractions (Table 4.1).

The laboratory-prepared DDGS samples with 15%, 30%, and 45% CDS exhibited the expected trends based on the CDS and WDG composition. As CDS level in the blend increased, crude protein and ash content increased while fat, NDF, and ADF content decreased (Table 4.3). The corresponding coefficients of determination (R^2) reflected in Table 4.3 indicate that the 85% - 98% of the variability in the chemical constituents of freeze-dried samples can be explained by CDS level.

Table 4.2 also shows the chemical composition of the plant-sourced wheat DDGS sample. The sample had higher protein and ash but lower fat and NDF content than the laboratory-prepared samples. This suggests that the plant-sourced sample was produced at a higher CDS level than what was used in the laboratory-prepared samples. The sample was obtained from the same ethanol plant on the same production date as the CDS and WDG samples.

Table 4.3 Significant linear relationships ($P = 0.05$) between selected chemical constituents and condensed distillers solubles (CDS) in wheat distillers dried grain with solubles (DDGS). Samples were produced at 15%, 30%, and 45% CDS and dried under freeze drying and forced air convection methods. Values inside the parentheses are coefficients of determination (R^2)

Chemical constituent	Freeze-dried samples	Forced air convection samples
Protein	0.16CDS + 26.44 (0.97)	0.12CDS + 28.52 (0.95)
Ash	0.04CDS + 2.84 (0.98)	0.05CDS + 2.48 (0.98)
Fat	- 0.01CDS + 5.92 (0.85)	-0.03CDS + 6.79 (0.47)
Neutral detergent fiber	-0.29CDS + 64.52 (0.98)	-0.38CDS + 68.16 (0.80)
Acid detergent fiber	-0.10CDS + 20.26 (0.96)	-0.10CDS + 20.98 (0.56)

4.4.1.2 *Forced-air convection-dried samples*

Table 4.4 shows the chemical constituents of forced air convection-dried samples. The chemical constituents followed similar trends exhibited by the freeze-dried samples with respect to CDS level (Table 4.3). The protein and ash content increased while fat, NDF, and ADF decreased as CDS level increased. While the R^2 values for protein, ash and NDF content of these samples were comparable to those found in freeze-dried samples, their fat and ADF contents, however showed much lower R^2 values (Table 4.3). This indicates that other factors aside from CDS level had affected fat and ADF content.

Fat and ADF content showed significant difference due to CDS level, drying air temperature, and their interaction (Table 4.4). In terms of fat content, 15% CDS samples dried under 80°C showed significantly higher fat content than those dried under 40°C and 120°C. Among 30% CDS samples, those dried under 40°C and 80°C had significantly higher fat content than those dried under 120°C. In samples with 45% CDS, those dried under 40°C contained significantly higher fat content than those dried under 80°C and 120°C. With respect to ADF content,

samples at all CDS levels that were dried under 120°C showed significantly higher ADF than those dried under 40°C and 80°C. Those with 15% and 45% CDS also showed significantly higher ADF content when these were dried under 80°C than under 40°C.

Samples dried under 120°C showed lower fat and higher ADF content compared to those dried under lower temperatures. Lower fat content of the 120°C -dried samples is attributed to losses due to lipid oxidation because of elevated drying temperature. Increase in ADF under higher drying air temperature is attributed to the formation of artifact lignin as a result of Maillard reaction (Perry et al. 2003). The effect of drying air temperature on ADF content was further highlighted when it was incorporated into the original ADF-CDS linear model (Table 4.3). The R^2 value of the model improved from 0.56 to 0.94 when drying air temperature was added. These increases in ADF indicate that protein quality may have been affected because of Maillard reaction, although changes in protein content were not as markedly manifested as those seen in fat content.

Table 4.4 Proximate composition¹ of forced air convection-dried wheat distillers dried grain with solubles (DDGS) samples with varying condensed distillers solubles (CDS) level. Values in parentheses represent standard deviation ($N = 2$)

CDS, mass %	Drying temp, °C	Moisture, % dry basis	Dry matter ² , %				
			Protein	Ash	Fat	NDF	ADF
15	40	7.78 (0.18)	30.3 (0.3) ^{aA}	3.3 (0.0) ^{aA}	5.9 (0.0) ^{aA}	65.1 (0.1) ^{aA}	18.9 (0.3) ^{aA}
	80	8.84 (0.03)	30.0 (0.1) ^{aA}	3.4 (0.0) ^{bA}	6.9 (0.1) ^{bA}	61.1 (0.0) ^{bA}	18.8 (0.0) ^{aA}
	120	8.91 (0.02)	30.5 (0.2) ^{cA}	3.2 (0.0) ^{aA}	6.1 (0.1) ^{aA}	62.9 (0.4) ^{aA,bA}	20.7 (0.3) ^{cA}
30	40	9.15 (0.19)	31.7 (0.2) ^{aB}	4.2 (0.0) ^{aB}	6.3 (0.0) ^{aB}	57.2 (1.3) ^{aB}	16.4 (0.2) ^{aB}
	80	8.62 (0.05)	32.0 (0.2) ^{aB}	3.9 (0.0) ^{bB}	6.5 (0.0) ^{aB}	51.9 (2.1) ^{bB}	18.0 (0.0) ^{bB}
	120	9.60 (0.08)	32.3 (0.2) ^{cB}	4.0 (0.1) ^{bB}	5.6 (0.0) ^{cB}	58.0 (0.2) ^{aB}	19.4 (0.2) ^{cB}
45	40	9.65 (0.02)	33.4 (0.1) ^{aC}	4.9 (0.0) ^{aC}	5.7 (0.2) ^{aC,aA}	50.9 (0.4) ^{aC}	15.3 (0.5) ^{aC}
	80	7.81 (0.00)	33.6 (0.1) ^{aC}	4.9 (0.0) ^{aC}	5.2 (0.0) ^{bC}	49.9 (0.9) ^{aC,bB}	16.1 (0.4) ^{bC}
	120	7.89 (0.04)	34.3(0.1) ^{cC}	4.9 (0.0) ^{aC}	5.3 (0.1) ^{bC}	54.2 (0.7) ^{cC}	18.0 (0.2) ^{cC}

Note: ¹ Tukey's test at 5% significance level for the same CDS level at varying drying temperatures (a, b, c) and for the same drying temperature at varying CDS levels (A,B,C). Values followed by the same set of letters are not significantly different; ² NDF means neutral detergent fiber; ADF means acid detergent fiber.

Overall, the level of CDS incorporation significantly affected the chemical composition of wheat DDGS. As CDS level increased, protein and ash content increased while fat and fiber content decreased, regardless of the drying methods employed. The influence of drying temperature on chemical composition was markedly manifested in the samples' fat and ADF content. At the same CDS level, drying under 120°C produced samples with significantly lower fat and higher ADF content than drying under lower temperatures. These results highlight the importance of selecting the appropriate CDS:WDG blending proportion and drying condition that will maximize its nutritive value, and thus, its market potential as an alternative feed ingredient.

4.4.2 Physical properties

4.4.2.1 *Particle size and size distribution*

Table 4.5 and Fig. 4.4 present the average particle size and the size distribution of laboratory-prepared wheat DDGS samples, respectively, while Table 4.6 shows the results of the corresponding analysis of variance. Average particle size ranged from 0.52 mm to 0.79 mm (Table 4.5). While CDS level, drying air temperature, and their interaction significantly affected particle size, the CDS level-associated sums of squares, which comprised 74% of the total sums of squares (Table 4.6), suggested the stronger influence of CDS level. This is further illustrated in Fig. 4.4, where samples with 45% CDS tended to have finer particles than those with 15% CDS. Decrease in particle size with increased CDS level is attributed to the increased presence of finer, endosperm- and germ-derived solids and decreased amount of the fibrous, bran-related particles. The effect of CDS level on particle size was still discernible even though these samples were ground after drying to generate the bulk material. The use of a screen size that was

four times larger than the geometric mean diameter of the plant-sourced sample had helped curb the adverse effect of grinding.

Table 4.5 Structural properties¹ of wheat distillers dried grains solubles (DDGS) samples, produced at varying condensed distillers solubles (CDS) level and dried under forced air convection method. Values in parentheses represent standard deviation ($N = 2$).

CDS, mass %	Drying temp., °C	Moisture, w.b. %	Particle size/mm	Bulk density, $\text{kg}\cdot\text{m}^{-3}$	Particle density, $\text{kg}\cdot\text{m}^{-3}$	Porosity, %
15	40	8.32 (0.05)	0.79 (0.00) ^{aA}	317 (2) ^{aA}	1346 (8) ^{aA}	76.4
	80	9.23 (0.41)	0.78 (0.02) ^{aA}	347 (1) ^{bA}	1336 (5) ^{aA}	74.0
	120	8.14 (0.22)	0.79 (0.02) ^{aA}	333 (2) ^{cA}	1315 (1) ^{aA}	74.7
30	40	8.93 (1.55)	0.76 (0.01) ^{aB,aA}	381 (5) ^{aB}	1337 (2) ^{aA}	71.5
	80	8.80 (0.22)	0.67 (0.02) ^{bB}	404 (7) ^{bB}	1340 (6) ^{aA}	69.8
	120	8.97 (0.06)	0.55 (0.02) ^{cB}	388 (2) ^{aB}	1338 (3) ^{aA}	71.0
45	40	8.75 (0.31)	0.58 (0.02) ^{aC}	422 (7) ^{aC}	1359 (1) ^{aC}	68.9
	80	8.32 (0.06)	0.52 (0.00) ^{bC}	440 (2) ^{bC}	1365 (1) ^{aC}	69.2
	120	8.51 (0.05)	0.60 (0.02) ^{cC}	420 (2) ^{aC}	1357 (1) ^{aC}	69.0

Note: ¹ Tukey's test at 5% significance level for the same CDS level at varying drying temperature (a, b, c) and for the same drying temperature at varying CDS level (A, B, C). Values followed by the same set of letters are not significantly different.

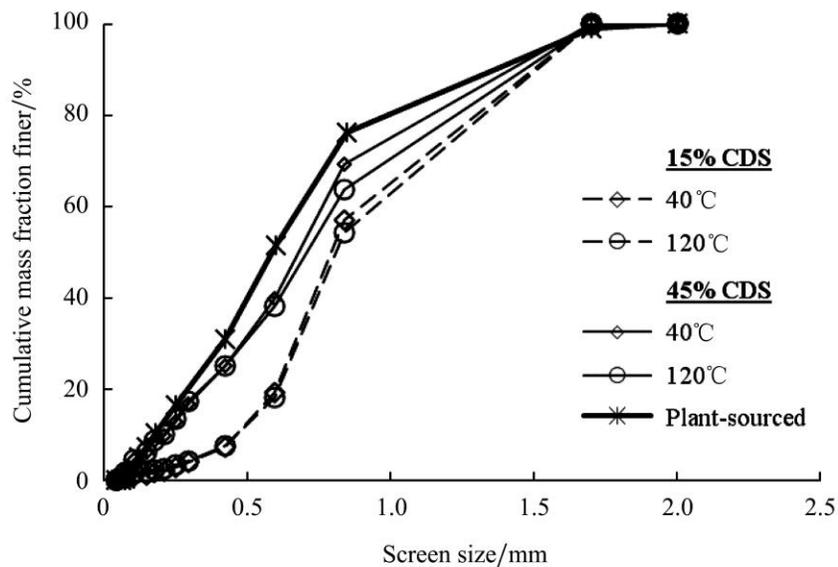


Fig. 4.4 Average particle size distribution of plant-sourced (9% moisture, w.b.) and laboratory-prepared (8%-9% moisture, w.b.) wheat distillers dried grain with solubles (DDGS) samples ($N = 2$).

Table 4.6 Analysis of variance (ANOVA) results for selected physical properties of wheat distillers dried grain with solubles (DDGS). The samples with 15%, 30%, and 45% condensed distillers solubles (CDS) were dried under 40°C, 80°C, and 120°C forced-air convection

Property	Total sums of squares, distribution/%				P-values		
	CDS level	Drying temp. (T)	CDS × T	Residual	CDS level	T	CDS × T
1. Physical attributes							
Particle size	72.6	6.5	19.9	1.0	0.000	0.000	0.000
Bulk density	92.7	6.1	5.2	0.6	0.000	0.000	0.025
Particle density	61.8	11.4	16.2	10.7	0.000	0.038	0.059
2. Flow properties							
Compressibility	61.6	14.8	18.6	5.1	0.000	0.002	0.004
Angle of repose	47.7	1.4	22.0	28.9	0.013	0.808	0.231
Ave. angle of spatula	57.4	22.0	11.1	9.5	0.000	0.005	0.104
Uniformity coefficient	81.8	1.6	16.2	0.4	0.000	0.000	0.000
Angle of fall	5.6	8.1	60.0	26.3	0.422	0.298	0.020
Angle of difference	29.1	12.5	32.1	26.3	0.035	0.173	0.096
Dispersibility	71.5	6.4	20.2	1.9	0.000	0.001	0.000
Overall flowability index	58.6	21.8	11.3	8.3	0.000	0.003	0.075
Overall floodability index	18.1	14.7	23.8	43.3	0.268	0.208	0.361
3. Compression characteristics							
Pellet density	50.4	12.0	6.6	31.0	0.000	0.003	0.129
Specific compression energy	19.8	12.8	25.3	42.1	0.001	0.008	0.002
Specific extrusion energy	5.0	12.7	26.1	56.1	0.219	0.025	0.007
Kawakita-Ludde model d	53.8	15.4	7.7	23.1	0.000	0.000	0.052
Kawakita-Ludde model f ⁻¹	50.7	12.6	10.3	26.4	0.000	0.001	0.016
4. Thermal properties							
Conductivity	20.6	11.8	14.7	52.9	0.228	0.405	0.656
Diffusivity	53.1	5.4	19.0	22.4	0.004	0.377	0.193
5. Frictional properties							
Coef. of internal friction	52.6	0.0	42.1	5.3	0.001	0.850	0.001
Cohesion	25.5	11.7	47.2	15.6	0.054	0.078	0.015

Figure 4.4 also shows the particle size distribution of the plant-sourced DDGS sample with moisture content similar to those of the laboratory-prepared samples. The plant sample has finer particles compared to the laboratory-prepared ones, further supporting the observation that

increased CDS level in the blend tended to produce finer particles. It was previously suggested that the plant-sourced sample may have been produced at a CDS level higher than 45% (by mass), considering its chemical composition relative to what was found in the CDS and WDG fractions (Table 4.2). This sample was obtained from an ethanol plant that employed a ring dryer for DDGS production. Under this drying system, the WDGS is dispersed and conveyed through the dryer in a hot air stream at high speed. Thus, incidence of particle agglomeration was not a concern compared to rotary drum drying. Plant-scale studies (Kingsly et al. 2010; Clementson and Ileleji 2012) on corn DDGS indicated that particle size increased with CDS level due to agglomeration during rotary drum drying.

4.4.2.2 *Density and porosity*

Like particle size, CDS level, drying air temperature, and their factorial interaction had significant effect on bulk density values (Tables 4.5 and 4.6). The sums of squares associated with CDS level comprised about 93% of the total variance (Table 4.6), once again suggesting the dominant influence of CDS level. Under each drying air temperature, samples with 45% CDS had significantly higher bulk density values than those with 15% and 30% CDS. Similarly, those with 30% CDS had significantly higher bulk density than those with 15% CDS. Increased CDS level in the blend translates to increased presence of finer but heavier solids and decreased amount of the larger, more fibrous particles. These finer particles would easily move through and fill the inter-particle spaces, consequently leading to a heavier bulk mass and lower porosity. Particle density also increased with CDS level but was not significantly affected by drying air temperature.

Based on the initial moisture content of the wet CDS and WDG fractions (Table 4.2), solids content of CDS was about 24.5% while that of WDG fraction was about 33.2%. The CDS fraction, however, was heavier compared to the WDG fraction. This was also seen in the bulk density of the wet WDGS samples ($685 - 965 \text{ kg}\cdot\text{m}^{-3}$), which increased as CDS level was increased.

In corn DDGS, Ganesan et al. (2008a) reported that solubles level, along with moisture content, had significant effect on aerated and tapped bulk densities but did not observe a specific trend.

4.4.2.3 *Flow properties*

Table 4.7 shows the various flow property measurements made from the wheat DDGS samples. The overall flowability index, which is the sum of the component indices of compressibility, angle of repose, average angle of spatula, and uniformity coefficient (Carr 1965), ranged from 70.5 to 77.5. With this value range, the wheat DDGS samples were classified as having a “fair” degree of flowability (Carr 1965). Materials under this category would sometimes require vibration to assure flow (Carr 1965). In general, the overall flowability index was significantly affected by CDS level and drying air temperature, although the stronger influence of CDS was still suggested by the associated sums of squares results (Table 4.6). Samples with higher CDS level had lower flowability. Those dried under 120°C also had significantly lower flowability index than those dried under 40°C and 80°C . The two flowability index component properties, compressibility and uniformity coefficient, were significantly affected by CDS level, drying air temperature, and their interaction (Tables 4.6 and 4.7). Similar to density and size, the ANOVA sums of squares associated with these two properties (Table 4.6) also suggest a stronger influence of CDS level. Samples with higher CDS level tended to be more compressible and

Table 4.7 Flow properties¹ of wheat distillers dried grain with solubles (DDGS) samples, produced from varying condensed distillers solubles (CDS) level and dried under forced air convection method

CDS, mass %	Drying temp., °C	Moisture, % w.b.	Compressibility %	Angle of Repose, °	Ave angle of Spatula, °	Uniformity coefficient	Angle of Fall, °	Angle of Difference, °	Dispersibility, %	Overall index	
										Flowability	Floodability
15	40	8.49 (0.04)	15.0 (0.3) ^{aA}	44.4 (2.0) ^{aA}	41.5 (3.1) ^{aA}	2.5 (0.0) ^{aA}	36.9 (2.6) ^{aA}	7.6 (0.6) ^{aA}	29.4 (0.9) ^{aA}	77.0 (0.0) ^{aA}	66.1 (1.2) ^{aA}
	80	9.40 (0.24)	15.3 (0.0) ^{aA,bA}	41.5 (1.4) ^{aA,bA}	43.4 (0.3) ^{aA,bA}	2.3 (0.0) ^{bA}	34.4 (0.7) ^{aA,bA}	7.2 (0.8) ^{aA,bA}	25.7 (0.6) ^{bA}	77.5 (0.7) ^{aA}	64.1 (1.2) ^{aA}
	120	8.00 (0.20)	16.1 (0.7) ^{aA}	43.7 (2.1) ^{aA,cA}	49.8 (0.1) ^{cA,bA}	2.9 (0.0) ^{cA}	34.9 (2.4) ^{aA,cA}	8.8 (4.5) ^{aA,cA}	20.7 (2.0) ^{cA}	73.3 (0.4) ^{cA}	65.3 (3.2) ^{aA}
30	40	8.37 (0.05)	15.2 (0.6) ^{aB,aA}	45.8 (0.8) ^{aB}	49.8 (1.6) ^{aB}	3.3 (0.0) ^{aB}	37.4 (0.2) ^{aB,aA}	8.5 (0.6) ^{aB}	23.1 (1.4) ^{aB}	73.5 (0.0) ^{aB}	65.8 (1.1) ^{aA}
	80	8.86 (0.38)	15.9 (0.7) ^{aB,bA}	46.6 (1.7) ^{aB,bB}	51.5 (1.8) ^{aB,bB}	3.6 (0.1) ^{bB}	37.4 (2.1) ^{aB,bA}	9.2 (0.4) ^{aB,bB}	17.7 (0.4) ^{bB}	72.3 (1.8) ^{aB,bB}	62.8 (0.4) ^{aA}
	120	8.51 (0.31)	18.5 (0.6) ^{cB}	46.3 (1.6) ^{aB,cB}	51.2 (0.1) ^{cB,bB}	2.9 (0.0) ^{cB,cA}	32.4 (0.1) ^{cB,cA}	13.9 (1.4) ^{aB,cB}	15.4 (1.4) ^{cB,bB}	70.5 (2.1) ^{cB}	66.5 (2.1) ^{aA}
45	40	8.54 (0.02)	18.0 (0.4) ^{aC}	43.7 (0.6) ^{aC,aA,aB}	49.9 (3.0) ^{aC,aB}	4.2 (0.1) ^{aC}	32.5 (0.9) ^{aC}	11.3 (0.4) ^{aC,aA,aB}	10.0 (1.1) ^{aC}	73.0 (0.0) ^{aC,aB}	63.8 (1.8) ^{aA}
	80	7.89 (0.10)	19.4 (0.4) ^{aC}	45.6 (0.1) ^{aC,bA,bB}	51.5 (0.9) ^{aC,bC,bB}	3.8 (0.1) ^{bC}	35.4 (0.9) ^{aC,bA}	10.3 (0.8) ^{aC,bA,bB}	13.6 (1.0) ^{bC}	71.3 (0.4) ^{aC,bB}	64.0 (1.4) ^{aA}
	120	8.45 (0.19)	18.2 (0.4) ^{aC,cB}	45.2 (0.7) ^{aC,cA,cB}	53.6 (1.0) ^{cC,bC,cB}	3.7 (0.1) ^{cC}	35.9 (1.1) ^{aC,cA}	9.4 (0.4) ^{aC,cA,cB}	15.4 (0.8) ^{cC,bC,cB}	71.8 (0.4) ^{cC,cB}	62.8 (0.4) ^{aA}

Note: ¹ Tukey's test at 5% significance level for the same CDS level at varying drying temperatures (a, b, c) and for the same drying temperature at varying CDS levels (A,B,C). Values followed by the same set of letters are not significantly different.

more differentiated in size than those with lower CDS level. Increasing the CDS level in the blend increased the presence of finer solids and decreased the amount of fibrous particles, thus, leading to slightly more differentiated particle sizes.

Aside from easily penetrating the void spaces in the bulk material, which translates to higher bulk density and higher compressibility, these finer particles also promote increased intergranular friction because of the greater surface area of contact. This is seen in the angle of repose and average angle of spatula values. Samples with 30% CDS had significantly higher angles of repose than those with 15% CDS. Similarly, those with higher CDS (30%, 45%) level had significantly higher average angle of spatula compared to those with lower CDS (15%). The angle of repose was significantly affected by CDS level while the angle of spatula was affected by variations in both CDS level and drying air temperature. The 120°C -dried sample had significantly higher angle of spatula than the 40°C -dried sample.

Under the Carr classification system (Carr 1965), the 45% CDS samples' angle of repose and average angle of spatula (Table 4.7) were considered on the borderline between the free-flowing and non-free flowing material. Their compressibility index (18.0%-19.4%) was also near the borderline (20%-21%). In terms of the uniformity coefficient, the samples were classified as having “excellent” flowability.

With respect to the overall floodability index, the values ranged from 62.8 to 66.5, classifying the wheat DDGS samples under the “floodable” category. Materials under this category require the use of a rotary seal to prevent flushing (Carr 1965). This means, for example, that in designing feed systems, adequate measures should be installed to ensure that these materials can be metered out under control.

The evaluation of the floodability index involved flowability index, angle of fall, angle of difference, and dispersibility. Dispersibility showed similar trends with flowability index with respect to the effect of CDS level. Samples with higher CDS level, which have higher bulk density values, were significantly less dispersible than those with lower CDS level. Although drying air temperature and its interaction with CDS level also significantly affected dispersibility, the associated sums of squares (Table 4.6) still suggests a stronger influence of CDS level.

4.4.2.4 *Compression characteristics*

Table 4.8 shows the pellet density, the specific energy consumption during the compression and extrusion processes, and the parameters of the Kawakita-Ludde model, which adequately described the compression characteristics of wheat DDGS samples.

Pellet density approached to about 86%-90% of particle density (Table 4.5) and was significantly affected by CDS level and drying air temperature (Table 4.6). Samples with 30% and 45% CDS had significantly higher pellet density than those with 15% CDS. Samples dried under 40°C and 80°C had significantly higher pellet density than those dried under 120°C. The ANOVA sums of squares associated with CDS level, however, comprised about 49% of the total observed variation (Table 4.5), suggesting a relatively strong influence of CDS level.

In terms of specific energy expenditure, about 9.9-17.2 MJ·t⁻¹ was consumed during compression, representing about 95%-99% of the total specific energy expenditure. The rest of the energy was consumed for extruding the pellet from the cylindrical die. While specific energy consumption during both processes was affected by one or both main factors and the CDS level × drying temperature interaction (Table 4.6), no consistent pattern was observed. Sizeable

Table 4.8 Compression characteristics¹ of wheat distillers dried grain with solubles (DDGS), produced at varying condensed distillers solubles (CDS) level and dried under forced air convection method. Values in parentheses represent standard deviation ($N = 5$)

CDS, mass %	Drying temp., °C	Moisture, % w.b.	Pellet density, kg·m ⁻³	Specific energy consumption, MJ·t ⁻¹		Kawakita-Ludde model parameters ²			
				Compression	Extrusion	d	f^1 /MPa	R^2	MSE
15	40	8.83 (0.26)	1168 (11) ^{aA}	10.63 (0.39) ^{aA}	0.30 (0.02) ^{aA}	0.73 (0.00) ^{aA}	0.49 (0.04) ^{aA}	0.967	0.000
	80	9.60 (0.28)	1175 (7) ^{aA,bA}	10.31 (0.16) ^{aA,bA}	0.45 (0.06) ^{bA}	0.70 (0.00) ^{bA}	0.53 (0.07) ^{aA}	0.970	0.000
	120	8.04 (0.11)	1162 (13) ^{cA,bA}	14.99 (0.66) ^{cA}	0.23 (0.02) ^{aA,cA,aC}	0.75 (0.00) ^{aA}	1.46 (0.12) ^{cA}	0.994	0.000
30	40	9.01 (0.41)	1206 (12) ^{aB}	12.71 (2.69) ^{aB,aA}	0.31 (0.14) ^{aB,aA}	0.70 (0.02) ^{aB}	1.01 (0.48) ^{aB,aA}	0.983	0.000
	80	9.03 (0.12)	1199 (16) ^{aB,bB}	12.80 (2.70) ^{aB,bA}	0.31 (0.15) ^{aB,bB}	0.68 (0.02) ^{bB}	1.42 (0.76) ^{aB}	0.985	0.000
	120	8.96 (0.14)	1187 (12) ^{cB,bB}	12.12 (0.08) ^{aB}	0.20 (0.08) ^{aB,cA}	0.69 (0.02) ^{aB,cB}	1.20 (0.48) ^{aB}	0.987	0.000
45	40	8.98 (0.07)	1210 (13) ^{aC,aB}	12.71 (1.76) ^{aC,aA}	0.32 (0.06) ^{aC,aA}	0.69 (0.01) ^{aC}	1.55 (0.43) ^{aC,aB}	0.981	0.000
	80	8.66 (0.27)	1190 (7) ^{aC,bC,bB}	15.47 (1.32) ^{bC}	0.27 (0.02) ^{aC,bB}	0.68 (0.01) ^{bC}	2.38 (0.41) ^{bC}	0.989	0.000
	120	8.74 (0.02)	1187 (11) ^{cC,bC,cB}	14.98 (1.12) ^{cC,bC,cA}	0.34 (0.08) ^{cC}	0.70 (0.01) ^{aC,cB}	2.24 (0.37) ^{bC}	0.984	0.000

Note: ¹Tukey's test at 5% significance level for the same CDS level at varying drying temperature (a, b, c) and for the same drying temperature at varying CDS level (A, B, C). Values followed by the same set of letters are not significantly different; ² The Kawakita-Ludde model parameters d and f^1 relate to initial porosity and failure stress, respectively.

residual sums of squares vis-à-vis the total sums of squares (Table 4.6), derived from both compression and extrusion specific energy consumption data sets, suggest that there could still be other factors that may have contributed to the observed variation.

The Kawakita-Ludde model adequately described the compression characteristics of wheat DDGS samples. Values of the model parameter d , which represent initial porosity (Mani et al. 2004; Emami and Tabil 2008) of the sample, were 95%-101% of the bulk porosity values (Table 4.5) and were significantly affected by CDS level and drying air temperature.

Similar to what was observed in the density-derived bulk porosity values (Table 4.5), samples with higher CDS level had significantly lower d values than those with lower CDS. Those dried under 40°C and 120°C also showed significantly higher d values than those dried under 80°C. On the other hand, the model parameter f^{-1} , which relates to failure stress, was significantly affected by CDS level, drying air temperature, and their interaction. Samples with 30% and 45% CDS consistently showed significantly higher f^{-1} values compared to those with 15% CDS across the temperature levels. This indicates that pellets with higher CDS level would be harder to break than those with lower CDS. The 120°C -dried samples also had significantly higher failure stress than the 40°C -dried samples.

Increased pellet density and failure stress of samples with increased CDS could be attributed to the increased amount of finer particles in the blend as well as to the chemical composition. With increased CDS level, there is increased presence of finer particles that would easily fill the voids between larger particles during compression. These finer particles also have greater surface area for contact, thus, enhancing binding. Higher CDS level also translates to higher protein and ash but lower fat and fiber content. Heat applied during densification, as well as those resulting from

friction, may have also altered the state of the chemical constituents and could promote better binding characteristics. For example, protein could have been denatured, and combined with the sugars present, positively affecting the strength of the pellets, as a result of Maillard reaction (Thomas and van der Poel 1996).

4.4.2.5 *Thermal properties*

Table 4.9 presents the thermal properties of wheat DDGS at 23°C and at the specified bulk densities. Average thermal conductivity of wheat DDGS samples was not significantly different across CDS and drying air temperature levels (Table 4.6). Thermal diffusivity, on the other hand, was significantly affected by CDS level and its values ranged from 1.35×10^{-7} to $1.65 \times 10^{-7} \text{ m}^2 \cdot \text{s}^{-1}$. Samples with 15% and 30% CDS had significantly higher thermal diffusivity values than those with 45% CDS. This is attributed to differences in density and porosity. Since air has a much higher thermal diffusivity compared to that of water (Kostaropoulos and Saravacos 1997), more porous materials, like the 15% CDS samples, would have higher thermal diffusivity than the less porous, 45% CDS samples at the same moisture content.

4.4.2.6 *Frictional properties*

The coefficient of internal friction and cohesion values for wheat DDGS are also presented in Table 4.9. The friction coefficient ranged from 0.63 to 0.74, equivalent to about 32°-35° angle of internal friction, close to the values reported for chickpea (Emami and Tabil 2008) and wheat (Teunou et al. 1999) flours. It was significantly affected by CDS level, drying air temperature, and by the CDS level \times drying temperature interaction, with the associated ANOVA sums of squares results suggesting a strong influence of CDS level (Table 4.6). The higher CDS samples, dried at 40°C and 120°C, presented significantly higher coefficient of friction than those with

Table 4.9 Thermal and frictional properties¹ of wheat distillers dried grain with solubles (DDGS), produced at varying condensed distillers solubles (CDS) level and dried under forced air convection method. Values in parentheses represent standard deviation ($N = 2$)

CDS, mass %	Drying temp., °C	Moisture, % w.b.	Thermal properties (at 23°C)			Frictional properties ²			Standard error of estimate
			Bulk density, $\text{kg}\cdot\text{m}^{-3}$	Conductivity, $\text{W}\cdot\text{m}^{-1}\cdot\text{°C}^{-1}$	Diffusivity ($\times 10^{-7}$), $\text{m}^2\cdot\text{s}^{-1}$	Coef. of internal friction	Cohesion, kPa	Coef. of determination, R^2	
15	40	8.83 (0.26)	330.6 (0.3)	0.05 (0.01) ^{aA}	1.65 (0.01) ^{aA}	0.63 (0.01) ^{aA}	2.96 (0.52) ^{aA}	0.999	0.38
	80	9.60 (0.28)	364.8 (0.6)	0.05 (0.00) ^{aA}	1.55 (0.01) ^{aA}	na	na		
	120	8.04 (0.11)	341.4 (3.1)	0.05 (0.01) ^{aA}	1.60 (0.01) ^{aA}	0.66 (0.02) ^{cA}	2.86 (1.00) ^{aA,cA}	0.999	0.53
30	40	9.01 (0.41)	397.3 (1.6)	0.05 (0.00) ^{aA}	1.55 (0.01) ^{aB,aA}	0.71 (0.01) ^{aB}	2.74 (0.02) ^{aB,aA}	0.996	1.09
	80	9.03 (0.12)	416.0 (0.3)	0.05 (0.00) ^{aA}	1.45 (0.01) ^{aB,aA}	na	na		
	120	8.96 (0.14)	389.6 (0.3)	0.05 (0.01) ^{aA}	1.65 (0.01) ^{aB,aA}	0.64 (0.01) ^{cB,cA}	4.93 (0.18) ^{cB}	0.997	0.95
45	40	8.98 (0.07)	427.6 (2.8)	0.05 (0.01) ^{aA}	1.35 (0.01) ^{aC}	0.69 (0.01) ^{aC,aB}	3.15 (0.08) ^{aC,aA}	0.997	1.03
	80	8.66 (0.27)	426.8 (0.8)	0.05 (0.00) ^{aA}	1.45 (0.01) ^{aC}	na	na		
	120	8.74 (0.02)	425.0 (1.1)	0.05 (0.00) ^{aA}	1.40 (0.00) ^{aC}	0.74 (0.00) ^{cC}	2.79 (0.06) ^{aC,cA}	0.999	0.52

Note: ¹ Tukey's test at 5% significance level for the same CDS level at varying drying temperature (a, b, c) and for the same drying temperature at varying CDS level (A,B,C). Values followed by the same set of letters are not significantly different. ² na – data were not available

lower CDS. Decreased particle size with increased CDS level may have contributed to higher values of the friction coefficient because this provides greater surface area of contact.

Cohesion estimates ranged from 2.7 kPa to 4.9 kPa and were significantly affected by the CDS level \times drying temperature interaction (Table 4.6). Only a few pairs of samples, however, showed significant difference. In samples dried under 120°C, those with 15% and 30% CDS, as well as those with 30% and 45% CDS, were significantly different. No consistent trend, however, was observed. Samples with 30% CDS and dried under 120°C showed significantly higher cohesion value than those dried under 40°C. The rest of the pairs were statistically similar.

4.4.2.7 *Influence of drying air temperature*

Although CDS level was seen as having a stronger influence on some of the physical properties investigated, there were consistent patterns observed with respect to the effect of drying temperature. The 40°C- and 120°C-dried samples, regardless of CDS level, showed significantly lower bulk density than those dried under 80°C. This was consistent with the trend of the Kawakita-Ludde parameter d values, where both 40°C- and 120°C-dried samples showed higher initial porosity than the 80°C sample. Between the 40°C - and 120°C -dried samples, the former had significantly higher uniformity coefficient, lower angle of spatula, higher flowability index, and higher pellet density.

Variability due to drying temperature could be attributed to the structural changes that occur during the drying process. Nowak and Lewicki (2005) reported microstructure differences between convectively-dried and infrared-dried apple tissues and indicated that drying rate could

be the probable cause of the microstructure differences. Higher drying rates, for example, by the higher drying temperature (120°C) used in the study, could result in larger shrinkage stresses and greater tissue damage (Nowak and Lewicki 2005). Significant differences in the structural properties (such as bulk density and porosity) could, consequently, affect flow, compression, thermal, and frictional properties.

4.5 Conclusions

The aim of this study was to assess the effect of CDS and drying temperature on the physico-chemical characteristics of wheat DDGS.

As CDS level in the blend was increased, protein and ash increased while fat and fiber decreased. Fat and ADF content were also markedly affected by drying air temperature. Compared to drying temperature, CDS level was seen as having a stronger influence on the most of the physical properties investigated. Wheat DDGS samples with higher CDS content were denser, smaller in size, less flowable, and less dispersible. These also had significantly lower thermal diffusivity, higher angle of internal friction, and produced pellets with higher density and failure stresses.

These results contribute toward better understanding of wheat DDGS variability, highlighting the importance of selecting the appropriate CDS:WDG blending proportion and drying conditions to maximize its nutritive value as an animal feed ingredient and improve the efficiency of related handling and processing operations.

Chapter 5

Physico-chemical characteristics of microwave- and microwave convection-dried wheat distillers grain with solubles

A similar version of this chapter has been published in the Journal of Microwave Power and Electromagnetic Energy:

- Mosqueda, M.R., L.G. Tabil, and V. Meda. 2013. Physico-chemical characteristics of microwave-dried wheat distillers grain with solubles. *Journal of Microwave Power and Electromagnetic Energy* 47(3): 155 - 176.

Contribution of the PhD Candidate

Literature review, planning and execution of the experiments, data analysis, and manuscript preparation were done by the PhD candidate under the guidance of her research supervisor, Lope G. Tabil. Edward Robertson and Shivani Trivedi, undergraduate research assistants, assisted in data collection and sample preparation, respectively. Editorial advice during manuscript preparation was provided by Lope Tabil and Venkatesh Meda.

Contribution of the Paper to the Overall Study

This paper focused on evaluating the effect of microwave-based drying and varying blending proportions of wet distillers grain (WDG) and condensed distillers solubles (CDS) on the physico-chemical characteristics of wheat distillers grain with solubles. It addressed one of the

main objectives of the PhD research project: assessment of the effect of drying methods/conditions and condensed distillers solubles (CDS): wet distillers grain (WDG) blending proportion on the physical and chemical characteristics of wheat distillers dried grain with solubles (DDGS). Presently, there is very limited information on how one of the identified causes (CDS:WDG blending) of wheat DDGS variation affected its physical and chemical characteristics. While there are already some studies on corn DDGS that explored this aspect, inherent compositional differences between corn and wheat kernels and their respective WDG and CDS streams, as well as differences in drying methods used, necessitate separate investigations for wheat DDGS. Properties investigated include: (i) chemical composition, (ii) physical attributes, such as particle size, density, and color; (iii) flow properties; (iv) compression characteristics; (v) frictional properties; and (vi) thermal properties. This paper also examined if alternative drying technologies, such as microwave-based methods and conditions, affected the physico-chemical characteristics of wheat DDGS. Aside from researchers, information derived from this study would be useful to ethanol plant managers and quality assurance officers who are considering alternative drying technologies for wheat WDGs, microwave dryer design engineers and marketing personnel, and animal feed processors.

5.1 Abstract

Laboratory-prepared samples of wheat distillers grain with solubles with varying CDS content were dried under varying microwave power and microwave convection settings using a domestic microwave oven to examine their effect on the chemical, structural, color, flow, compression, thermal, and frictional properties of wheat DDGS. As CDS level increased, protein and ash content increased while fat and fiber content decreased in wheat-based DDGS. Fat content was

also markedly affected by the microwave oven drying conditions. While CDS level, microwave power or microwave convection setting, and/or their interactions significantly affected a number of physical properties, results indicated that CDS level had a stronger influence compared to the other factors. DDGS samples with high CDS levels were significantly denser, finer but more differentiated in size, less flowable, and less dispersible. These also produced denser and stronger pellets.

5.2 Introduction

Product variability, energy-intensive drying process and reduced protein quality are among the challenges that currently confront wheat DDGS production from ethanol processing plants in western Canada (University of Saskatchewan 2010). Several animal feeds studies, for example, reported low lysine content, availability and digestibility in wheat DDGS, despite its high crude protein content (Nyachoti et al. 2005; Widyaratne and Zijlstra 2007; Lan et al. 2008; Cozannet et al. 2010). Loss of lysine, an essential amino acid in animal diet, lowers the nutritive value of a feed ingredient (Fayle and Gerrard 2002) and limits the utilization of wheat DDGS as feed ingredient for monogastric animals (Widyaratne and Zijlstra 2007). Also, Nuez-Ortin (2010) found significant nutrient variability in wheat DDGS samples obtained from two Saskatchewan plants and stressed the importance of product consistency not only for more accurate animal feed formulations but also in improving the market prospects of wheat DDGS.

High temperature drying and the blending of CDS and WDG had been identified as among the major causes of variation in the physico-chemical characteristics and reduced protein quality in both corn (Batal and Dale 2006; Fastinger et al. 2006; Ileleji and Rosentrater 2008) and wheat (Nyachoti et al. 2005; Widyaratne and Zijlstra 2007; Lan et al. 2008; Cozannet et al. 2010; Nuez-

Ortin 2010) DDGS. The effect of CDS and WDG blending proportions on the physical and/or chemical properties of corn DDGS had been examined by Ganesan et al. (2008a; 2008c), Bhadra et al.(2009b; 2009c; 2011a; 2012), Kingsly et al. (2010), and Clementson and Ileleji (2012). Bhadra et al. (2009b; 2009c; 2011a; 2012) evaluated both varying CDS and temperature levels while the rest evaluated DDGS characteristics at relatively uniform temperatures. Ganesan et al. (2008a; 2008c) and Bhadra et al. (2009b; 2009c; 2011a; 2012) conducted laboratory-scale investigations while the Kingsly et al. (2010) and Clementson and Ileleji (2012) did plant-scale experiments.

Compared to corn DDGS, there is still limited detailed information on the effect of varied blending proportions of CDS and WDG on the physical and chemical characteristics of wheat DDGS. Investigations on this aspect are necessary considering the inherent compositional differences between the corn- and wheat-based CDS and WDG fractions. These compositional differences, summarized in Table 5.1, could translate to differences in physico-chemical characteristics.

Table 5.1 Comparative chemical composition of corn and wheat grain, wet distillers grain (WDG), and thin stillage/condensed distillers solubles (CDS), % dry matter

Sample	Crude protein	Ash	Crude fat	Neutral detergent fiber	Acid detergent fiber
Corn-based					
Grain ¹	10.13	1.73	4.59	14.47	3.66
WDG ^{2,3}	34.4 – 35.0	2.0 – 2.35	10.9 – 14.0	39.0	11.9 – 17.2
CDS ^{2,4}	22.4 – 23.0	6.6 – 11.6	21.6 – 34.4	3.6 – 4.9	2.92
Wheat-based					
Grain ¹	14.28	2.12	1.91	17.22	3.68
WDG ⁵	26.4	2.7	6.6	74.9	24.1
Thin stillage ^{6,7}	45.7 – 48.5	8.0 – 8.3	9.63 – 13.6	34.0 – 34.5	3.4 – 4.0
Wet distillers solubles ⁸	32.6	8.9	5.7	2.3	

¹Nuez-Ortin 2010; ²Cao et al. 2009; ³Kim et al. 2008; ⁴Fron et al. 1996; ⁵Ojowi et al. 1997; ⁶Ojowi et al. 1996; ⁷Mustafa et al. 1996; ⁸Pedersen and Lindberg 2010

Further, there had also been studies investigating the potential of alternative drying technologies, such as the use of superheated steam (Tang et al. 2005; Pronyk et al. 2004) and microwave energy (Mosqueda and Tabil 2011a), to reduce energy consumption and minimize the negative impact of high temperature drying on nutrient composition of wheat DDGS. These studies were focused on commercial wheat DDGS and evaluated the effect of drying parameters on a limited number of product characteristics. Tang et al. (2005) and Pronyk et al. (2004) reported that except for starch, the other nutrient components (β -glucan, pentosan and protein) of wheat distillers spent grain were not significantly affected with varying steam temperature and drying time. Mosqueda and Tabil (2011a) examined the effect of three drying methods (forced-air convection, microwave, and microwave convection) on lysine content and color of wheat DDGS. Microwave-assisted methods significantly lowered the drying time and showed the potential of minimizing decreases in lysine content. In another study, they also examined the effect of CDS level and drying methods on protein quality of wheat DDGS. Mosqueda et al. (2013a) reported higher acid detergent insoluble crude protein and lower lysine when samples were dried at higher drying air temperature, microwave power, and microwave convection settings. Microwave drying produced similar optimum protein quality as forced-air convection drying at lower temperature but was achieved at a much shorter time.

Investigations quantifying the combined effect of varied CDS:WDG mixing ratios and drying parameters on the physical and chemical characteristics of wheat DDGS, however, are still few, particularly using microwave-based drying methods. Such studies could contribute toward a better understanding of the underlying causes of product quality variation and in evaluating the potential of the alternative drying technologies. The objective of this study, therefore, was to

examine the effect of varied CDS:WDG blending proportions, microwave power levels, and microwave convection settings on the physico-chemical characteristics of wheat DDGS.

5.3 Materials and Methods

CDS and WDG were obtained from a south Saskatchewan fuel ethanol plant, placed in tightly sealed bins, and stored in a -16°C freezer. Five hundred gram to one kilogram batches of wet distiller's grain with solubles (WDGS) were prepared by mixing thawed CDS and WDG at three ratios, by mass: 15:85, 30:70, and 45:55 using a 6-speed Toastmaster hand mixer (model 1776CAN, Toastmaster Inc., China) for 15 to 30 minutes at medium speed. These blends are subsequently referred to in this paper as 15%, 30%, and 45% CDS. Prepared WDGS were placed in sealed plastic bags and stored in the -16°C freezer until these were used.

5.3.1 Drying the WDGS

Bags of frozen WDGS were thawed overnight and were placed in the sample preparation room for equilibration with room temperature (22-24°C) prior to the thin layer drying runs. A Panasonic® microwave/convection oven (model NNC980W, Panasonic Canada, Ltd., Mississauga, ON) was used for both microwave and microwave convection drying. Its technical specifications are presented in Mosqueda et al. (2013a). Four (4) power levels were used for microwave drying: 40% (P4), 60% (P6), 80% (P8), and 100% (P10) microwave power, previously estimated to be 420, 676, 701 and 805 W, respectively (Mosqueda and Tabil 2011a). In microwave convection drying, the oven's four (4) nominal combination settings, 130°C-30% power (C1), 150°C-30% power (C2), 160°C-30% power (C3), and 190°C-30% power (C4) (Matsushita Electric Industrial Co. Ltd. 2000) were all used. The power output at each of these

combination settings were also previously estimated to be 303, 316, 327, and 332 W, respectively (Mosqueda and Tabil 2011a). Figure 5.1 shows the sample material before and after drying.

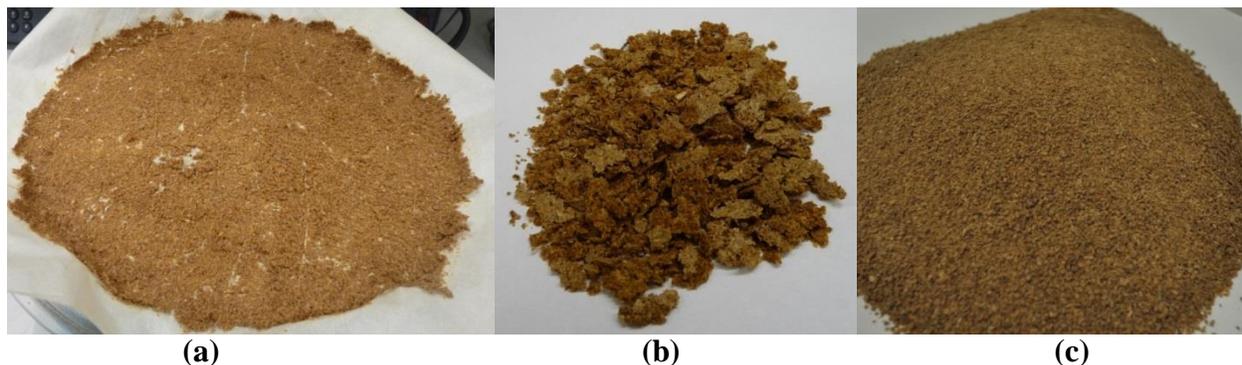


Fig. 5.1. Sample preparation: (a) wet distillers grain with solubles before drying, (b) material after drying, (c) bulk material after grinding.

5.3.2 Chemical composition

The chemical composition of both wet and dried samples was determined. Wet samples were (WDGS, CDS, and WDG) freeze-dried using the Labconco FreeZone Freeze Dry System (Labconco Corp., Kansas City, MO) prior to the proximate analysis runs. All dried samples were ground using a Thomas-Wiley knife mill (Thomas Scientific, Swedesboro, NJ) equipped with a 1 mm screen.

Moisture content was determined using the AOAC Official Method 920.36 (AOAC 2003a). Crude protein was estimated using the Kjeldahl method, AOAC Official Method 984.13 (AOAC 2003b). Crude fat was determined using the Goldfish fat extractor (Labconco Corporation, Kansas City, MO), following the AOAC Official Method 920.39 (AOAC 2003d), with anhydrous diethyl ether as extraction solvent. Crude ash was determined using AOAC Official Method 942.05 (AOAC 2003e), while neutral detergent fiber (NDF) and acid detergent fiber

(ADF) were estimated through AOAC Official Method 973.18 (AOAC 2003c) and through the method laid out by van Soest et al. (1991), respectively. A lamb starter feed material, AAFCO 0728 (Association of American Feed Control Officials (AAFCO) Check Sample Program, AAFCO, Champaign, IL), was used as a check sample. The proximate analysis was conducted in duplicate runs.

The proximate composition of these laboratory-prepared samples was also compared with a wheat DDGS sample that was sourced from the same ethanol plant where the CDS and WDG samples were obtained. All three materials (CDS, WDG, and the plant-sourced DDGS) had the same production date.

5.3.3 Physical properties

Because of raw material constraints, only two levels of microwave (P4, P10) and microwave convection (C1, C4) drying settings were used in generating samples for physical property measurement. Dried samples were passed through a Thomas-Wiley mill (Thomas Scientific, Swedesboro, NJ), in batches of 150-200 g for 2 minutes to generate the bulk material. The mill was equipped with a 2.68 mm screen, a size about four times larger than the observed geometric mean diameter of commercial wheat DDGS samples. This screen size was used to help curb the adverse effect of grinding on the sample's physical characteristics. Moisture content of samples was about 8-9% (wet basis).

Density, particle size and size distribution, color, flow, compression, frictional, and thermal properties of the dried samples were determined. Duplicate measurements were made for each property, unless otherwise stated.

5.3.3.1 Physical attributes

Sieve sizes 12, 20, 30, 40, 50, 60, 70, 80, 100, 140, 200, and 270 and a sieve shaker were used for particle size analysis, following ANSI/ASAE S319.4 (ASAE 2008). The calculated geometric mean diameter was used to represent particle size in this study.

To determine bulk density, the sample was placed on a funnel (Fig. 5.1a) and was allowed to freely flow into a 0.5 L steel cup (SWA951, Superior Scale Co. Ltd., Winnipeg, MB). The cup contents were leveled using a steel rod and weighed. Bulk density was calculated by dividing sample mass contained in the cup with the cup volume. Particle density was determined using a gas multipycnometer (QuantaChrome, Boynton Beach, FL), shown in Fig. 5.1b. Bulk porosity (ϵ), expressed as a percentage, was determined as a function of bulk (ρ_b) and particle densities (ρ_p) using Eq. 5.1 below.

$$\epsilon = 1 - \frac{\rho_b}{\rho_p} \quad (5.1)$$

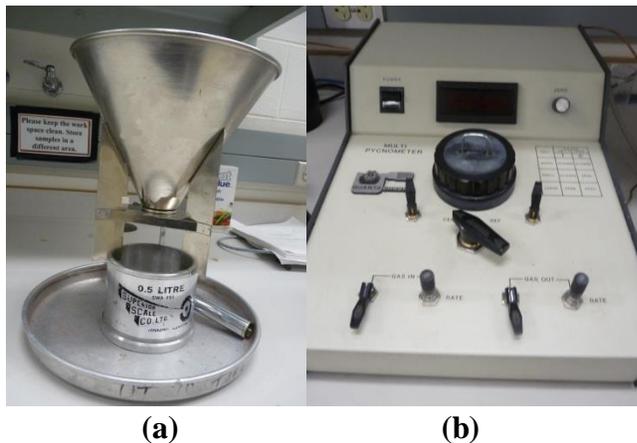


Fig. 5.2 Bulk density apparatus (a) and a gas multipycnometer (b).

5.3.3.2 *Color*

Color was determined using a HunterLab LabScan II colorimeter (Hunter Associates Laboratory Inc., Reston, VA) with a ½” area view and port size setting and expressed in terms of the Hunter Lab values. The color parameter L represents lightness and ranges from 0 (black) to 100 (white). Positive values of the color parameters a and b represent redness and yellowness, respectively, while their respective negative values represent greenness and blueness.

5.3.3.3 *Flow properties*

The Carr flowability and floodability indices were determined by measuring seven properties using a Hosokawa Micron Powder Tester PT-R (Hokosawa Micron Corp., Osaka, Japan). Each property measurement was assigned an index value, ranging from 0 to 25, depending on the point score classification system developed by Carr (1965). The flowability index, which ranged from 0 (very, very poor flowability) to 100 (excellent flowability), was derived from the sum of the individual index values of compressibility, angle of repose, angle of spatula, and uniformity coefficient. The floodability index, which also ranged from 0 (will not flood) to 100 (very floodable), was derived in a similar manner, using the index values of flowability, angle of fall, angle of difference, and dispersibility. Detailed description of each flow property and its measurement was presented by Carr (1965), Hokosawa Micron Corp. (n.d.) and Ganesan et al. (2008a). The Hosokawa Micron Powder Tester had been used in determining the flow properties of corn DDGS (Ganesan et al. 2008a; Ganesan et al. 2008c; Bhadra et al. 2009a; 2009b) and in other materials (Hokosawa Micron Corp. n.d.) with average particle sizes larger than the wheat DDGS.

5.3.3.4 *Compression characteristics*

An Instron Model 3366 testing machine (Instron, Norwood, MA), fitted with 10 kN load cell and a 6.30 mm plunger, was used to compress 1 g samples placed inside a cylindrical die (6.35 mm diameter and 135.34 mm length) that was constantly heated at 90°C. Crosshead speed was set at 30 mm·min⁻³. When the preset compressive load of 4400 N was reached, the plunger remained in its place for 1 min before ejecting the pellet. The test was conducted in 5 replicates. Force, displacement, and time data were directly logged into the computer. Experimental data generated were fitted to 2 models: 1) Jones (Eq. 5.2) (Mani et al. 2004; Emami and Tabil 2008); and 2) Kawakita and Ludde (Eq. 5.3) (Emami and Tabil 2008). The suitable model was chosen using coefficient of determination (R²) and mean square error (MSE) as criteria.

$$\ln\rho = m \ln P + r \quad (5.2)$$

$$\frac{P}{C} = \frac{1}{d \cdot f} + \frac{P}{d} \quad (5.3)$$

$$\text{where: } C = \frac{V_0 - V}{V_0} \quad (5.4)$$

In Eq. 5.2, ρ is bulk density, P is the applied compressive pressure, while m and r are the model parameters. In Eq. 5.3, C is the degree of volume reduction, V_0 is the volume of compact at zero pressure, V is volume of compact at pressure P , while d and f are the model parameters. Model parameter d is equal to the initial porosity of the sample while f^{-1} is related to failure stress of the pelleted sample (Mani et al. 2004; Emami and Tabil 2008).

Pellet density was determined by measuring the mass, length and diameter of the resulting pellet immediately after it was extruded from the die. Specific energy consumption during compression

and extrusion was estimated by calculating the area under the corresponding force-displacement curve using the trapezoidal rule and dividing it by the pellet mass.

5.3.3.5 *Thermal properties*

Thermal conductivity and thermal diffusivity of dried samples at ambient room condition were determined using a KD 2 Thermal Properties Analyzer (Decagon Devices, Inc., Pullman, WA). Distilled water and 1.5% agar solution were used as check samples. Because bulk density can affect thermal property measurement, it was measured immediately before measurements for thermal properties were obtained.

5.3.3.6 *Frictional properties*

The coefficient of internal friction and cohesion were determined using the Wykeham Farrance shear box apparatus (Wykeham Farrance International Ltd., Slough, U.K.), as described in Mani et al. (2004) and Emami and Tabil (2008). Maximum shear stress was determined at four normal loads (200, 400, 600, and 800 N) (Emami and Tabil 2008). Values for the angle of internal friction and cohesion were estimated through regression analysis of experimental data using Eq. 5.5 (Peleg et al., 1973), where τ is the shear stress (Pa), σ is the normal stress (Pa), θ is the angle of internal friction and C_c is the cohesion (Pa).

$$\tau = \tan \theta \sigma + C_c \tag{5.5}$$

5.3.4 **Statistical analysis**

The general linear model univariate procedure in SPSS 14.0 for Windows (SPSS Inc., Chicago, IL) was used to test the effect of CDS level, microwave power, and microwave convection

settings. Tukey's test was used to further evaluate statistically significant main effects and interactions. Linear regression analyses were conducted the same statistical package and MS Excel 2010 (Microsoft Corp., Redmond, WA). All tests were conducted at the 0.05 significance level.

5.4 Results and Discussions

5.4.1 Chemical composition

5.4.1.1 *Freeze-dried samples*

Table 5.2 shows the proximate composition of freeze-dried wheat WDS, CDS, and laboratory-prepared DDGS samples at varying CDS level. The CDS component had higher crude protein and ash content but lower fat and fiber content compared to WDG. Ojowi et al. (1996; 1997) also reported similar trends, wherein the wheat-based thin stillage, from where the CDS component was derived, had higher crude protein and lower neutral detergent fiber (NDF) and acid detergent fiber (ADF) than the WDG fraction (Table 5.1). Differences in the CDS and WDG composition are attributed to differences in the composition of the wheat kernel components that comprised these two fractions. The bran, which is high in fiber, predominantly comprised the WDG fraction while endosperm and germ particles made up the CDS fraction. The chemical constitution of wheat DDGS was not similar to those reported for corn DDGS (Ganesan et al. 2008a; Kingsly et al. 2010; Clementson and Ileleji 2012; Cao et al. 2009) due to the basic compositional differences between corn and wheat grains and between their respective CDS and WDG fractions (Table 5.1). Table 5.2 also shows that the plant-sourced sample has higher protein and ash but lower fat and NDF content than the laboratory-prepared samples. This

Table 5.2 Chemical composition¹ of wheat-based wet distillers grain (WDG), condensed distillers solubles (CDS), and distillers dried grain with solubles (DDGS) samples. Values in parentheses represent standard deviation (N = 2)

Sample ²	% Moisture, wet basis		% dry matter ³				
	Initial	Freeze-dried	Protein	Ash	Fat	Neutral detergent fiber	Acid detergent fiber
WDG	66.82 (0.24)	16.24 (0.29)	28.84 (0.60)	2.90 (0.02)	6.71(0.04)	77.53 (0.24)	18.39(0.39)
CDS	75.52 (0.26)	18.98 (1.93)	45.82 (0.39)	9.30 (0.02)	2.21 (0.04)	18.14 (0.29)	6.66 (0.08)
DDGS							
15% CDS	67.71 (0.26)	6.81 (0.31)	29.02 (0.24) ^a	3.56 (0.03) ^a	5.72 (0.13) ^a	60.45 (0.38) ^a	18.61 (0.03) ^a
30% CDS	69.60 (0.40)	8.08 (0.23)	30.70 (0.11) ^b	4.05 (0.01) ^b	5.59 (0.04) ^{a,b}	55.00 (0.03) ^b	17.48 (0.13) ^b
45% CDS	71.44 (1.04)	11.14 (0.25)	33.74 (0.00) ^c	4.88 (0.02) ^c	5.35 (0.01) ^b	51.62 (0.16) ^c	15.58 (0.45) ^c
Plant-sourced	13.32 (0.23)	-	38.81 (0.14)	7.10 (0.01)	4.89 (0.18)	46.55 (0.80)	17.45 (0.27)

¹Values with the same superscript letter under each column are not significantly different at P = 0.05.

² Except for the plant-sourced wheat DDGS sample, all other samples were freeze-dried prior to proximate analysis

suggests that the plant sample was produced at a higher CDS level than was used in the laboratory-prepared samples.

Table 5.3 show the linear relationships between CDS level (% mass) and selected chemical constituents (% dry matter) of the freeze-dried samples. These relationships reflected the expected trends for wheat DDGS considering the CDS and WDG chemical composition. As CDS level in the blend increased, crude protein and ash content increased while fat, NDF, and ADF content decreased. The corresponding coefficients of determination (R^2) indicate that the 85-98% of the variations in the chemical constituents can be accounted for by variations in CDS level.

5.4.1.2 *Microwave- and microwave convection-dried samples*

Table 5.3 also compared the CDS level-chemical constituent linear relationships derived from freeze-dried samples with those from microwave- and microwave convection-dried samples. Both sample types exhibited similar trends as the freeze-dried samples. Protein and ash content increased while fat, NDF, and ADF decreased as CDS level in the blend increased.

Some of these linear relationships, however, indicate that other factors, aside from CDS level, have affected the chemical constituents. This is particularly observed in fat content, where most of the coefficients of determination (R^2) were considerably lower than those derived from freeze-dried samples. Processing conditions, such as use of higher temperature settings at constant microwave power (C2, C3, C4), longer exposure to lower microwave power levels (P4, P6), and shorter exposure to higher microwave power levels (P8, P10), may have contributed to lipid oxidation and adversely affected fat content.

Table 5.3 Linear relationships between chemical constituents and condensed distillers solubles (CDS) level in freeze-, microwave-, and microwave convection-dried wheat distillers dried grain with solubles (DDGS). Samples under each drying method were produced at 15%, 30% and 45% CDS. Relationships are significant at P = 0.05 unless otherwise indicated

Chemical constituent	Freeze-dried sample		Microwave-dried samples			Microwave convection-dried samples		
	Equation	R ²	Setting ¹	Equation	R ²	Setting ²	Equation	R ²
Protein	0.16 CDS + 26.44	0.97	P4	0.12 CDS + 28.83	0.99	C1	0.08 CDS + 29.68	0.98
			P6	0.13 CDS + 28.12	0.99	C2	0.15 CDS + 27.76	0.93
			P8	0.13 CDS + 27.98	1.00	C3	0.14 CDS + 28.31	0.98
			P10	0.14 CDS + 27.60	0.99	C4	0.08 CDS + 30.13 ^{ns}	0.56
Ash	0.04 CDS + 2.84	0.98	P4	0.05 CDS + 2.60	0.96	C1	0.05 CDS + 2.58	0.93
			P6	0.05 CDS + 2.56	0.99	C2	0.04 CDS + 2.78	0.93
			P8	0.05 CDS + 2.55	0.99	C3	0.04 CDS + 3.01	0.90
			P10	0.05 CDS + 2.70	0.97	C4	0.04 CDS + 2.72	0.90
Fat	- 0.01 CDS + 5.92	0.85	P4	-0.03 CDS + 7.09 ^{ns}	0.59	C1	-0.05 CDS + 7.86	0.70
			P6	-0.02 CDS + 6.77 ^{ns}	0.56	C2	-0.01 CDS + 6.57 ^{ns}	0.12
			P8	-0.03 CDS + 7.17	0.81	C3	-0.01 CDS + 6.24 ^{ns}	0.26
			P10	-0.04 CDS + 7.53	0.71	C4	-0.02 CDS + 6.68 ^{ns}	0.46
Neutral detergent fiber	-0.29 CDS + 64.52	0.98	P4	-0.36 CDS + 67.53	0.97	C1	-0.49 CDS + 74.11	0.91
			P6	-0.58 CDS + 75.84	1.00	C2	-0.32 CDS + 68.21	0.98
			P8	-0.41 CDS + 69.73	1.00	C3	-0.44 CDS + 73.80	0.91
			P10	-0.36 CDS + 68.62	0.98	C4	-0.42 CDS + 73.22	0.98
Acid detergent fiber	-0.10 CDS + 20.26	0.96	P4	-0.21 CDS + 23.45	0.95	C1	-0.12 CDS + 20.20	0.72
			P6	-0.23 CDS + 24.06	0.89	C2	-0.19 CDS + 21.69	0.83
			P8	-0.20 CDS + 22.98	0.94	C3	-0.12 CDS + 19.97	0.83
			P10	-0.12 CDS + 20.61 ^{ns}	0.41	C4	-0.22 CDS + 23.11	0.79

¹P4 – 40% power (420 W); P6 – 60% power (676 W); P8 – 80% power (701 W); P10 – 100% power (805 W)

²C1 - 130°C, 30% power (303 W); C2 -150°C, 30% power (316 W); C3 - 160°C, 30% power (326 W); and C4 -190°C, 30% power (332 W)

^{ns} The linear relationship was not significant at P = 0.05

Table 5.4 shows the proximate composition of microwave- and microwave convection-dried samples in detail while Table 5.5 presents the results of the analysis variance (ANOVA) conducted for each of the chemical constituents investigated. While interaction between CDS level and dryer settings had significant effect on most of the chemical constituents, CDS level seemed to have the stronger influence compared to the dryer settings in both sample types (Table 5.5). Except for fat content of microwave convection-dried samples, the sums of squares associated with CDS level accounted between 85 to 99% of the total sums of squares derived from the chemical composition data sets in both sample types. Table 5.4 also shows several statistically similar samples with respect to variations due to microwave power or microwave convection settings at the same CDS level. This was not the case with respect to variations due to CDS level at each of the dryer settings. Samples with varying CDS level at the same dryer setting have significantly different chemical composition values. Except for fat content, the strong relationships between CDS level and the chemical constituents were also illustrated more clearly in Table 5.3.

5.4.2 Physical properties

Table 5.6 presents the selected physical attributes (particle size, bulk and particle densities, color) of the laboratory-prepared wheat DDGS. The previously presented Table 5.5 shows the corresponding analysis of variance (ANOVA) results for these properties. CDS level at each of the dryer settings. Samples with varying CDS level at the same dryer setting have significantly different chemical composition values. Except for fat content, the strong relationships between CDS level and the chemical constituents were also illustrated more clearly in Table 5.3.

Table 5.4 Proximate composition¹ of microwave- and microwave convection-dried wheat distillers dried grain with solubles (DDGS)with varying condensed distillers solubles (CDS) level. Values in parentheses represent standard deviation (N = 2)

CDS, % mass	Drying		Moisture, % dry basis	% dry matter ³				
	Method	Setting ²		Protein	Ash	Fat	Neutral detergent fiber	Acid detergent fiber
15	Microwave (MW)	P4	8.53 (0.07)	30.5 (0.0) ^{Aa}	3.3 (0.1) ^{Aa}	6.9 (0.1) ^{Aa}	62.2 (1.5) ^{Aa}	20.6 (0.3) ^{Aa, Ad}
		P6	7.96 (0.17)	30.3 (0.2) ^{Aa, Ab}	3.2 (0.0) ^{Aa}	6.7 (0.1) ^{Aa}	67.8 (0.5) ^{Ab}	21.4 (0.1) ^{Aa}
		P8	8.21 (0.10)	30.0 (0.1) ^{Ab}	3.3 (0.1) ^{Aa}	6.8 (0.2) ^{Aa}	63.5 (0.0) ^{Aa}	19.6 (0.2) ^{Ac}
		P10	8.90 (0.16)	29.8(0.1) ^{Ab}	3.3 (0.0) ^{Aa}	7.2 (0.1) ^{Ad}	63.4 (0.4) ^{Aa}	20.1(0.0) ^{Ac,Ad}
30	MW	P4	14.92 (0.09)	32.6 (0.1) ^{Ba}	4.3 (0.0) ^{Ba}	5.9 (0.0) ^{Ba}	56.9 (0.6) ^{Ba}	16.4 (0.5) ^{Aa}
		P6	7.90 (0.08)	32.0 (0.2) ^{Ba,Bb}	4.1 (0.0) ^{Bb}	6.0 (0.1) ^{Ba,Bb}	58.0 (0.8) ^{Ba}	16.0 (0.4) ^{Aa}
		P8	14.09 (0.08)	32.0 (0.0) ^{Bb}	4.2 (0.0) ^{Bb}	5.9 (0.1) ^{Ba,Bc}	57.9 (0.2) ^{Ba}	17.8 (0.6) ^{Ac}
		P10	14.54 (0.05)	31.9 (0.4) ^{Bb}	4.2 (0.1) ^{Bb}	6.0 (0.1) ^{Ba,Bd}	57.4 (1.3) ^{Ba}	14.6 (0.2) ^{Ad}
45	MW	P4	11.81 (0.10)	34.1 (0.2) ^{Ca}	4.8 (0.0) ^{Ca}	6.0 (0.1) ^{Ca, Cc, Aa}	51.5 (0.9) ^{Ca, Cb}	14.2 (0.4) ^{Aa}
		P6	10.22 (0.01)	34.2 (0.2) ^{Ca, Cb}	4.7 (0.0) ^{Ca}	6.1 (0.1) ^{Ca, Bb}	50.0 (0.2) ^{Cb}	14.5 (0.8) ^{Aa}
		P8	10.80 (0.08)	33.9 (0.1) ^{Cb}	4.8 (0.1) ^{Ca}	5.8 (0.0) ^{Cc, Bc}	51.3 (0.5) ^{Ca}	13.6 (0.1) ^{Aa}
		P10	8.44 (0.13)	34.2 (0.3) ^{Cb}	4.8 (0.1) ^{Ca}	6.0 (0.1) ^{Ca, Cc, Bd}	52.6 (0.8) ^{Ca, Cb}	16.5 (0.0) ^{Ad}
15	Microwave convection (MWC)	C1	8.82 (0.01)	30.9 (0.3) ^{Ae}	3.3 (0.0) ^{Ae}	7.4 (0.2) ^{Ae}	67.9 (1.1) ^{Ae}	19.0 (0.4) ^{Ae}
		C2	8.97 (0.09)	30.4 (0.1) ^{Ae}	3.2 (0.0) ^{Af}	6.3 (0.4) ^{Af}	63.5 (0.2) ^{Af}	19.1 (0.6) ^{Ae, Ah}
		C3	10.47 (0.07)	30.5 (0.2) ^{Ae}	3.3 (0.0) ^{Ag}	6.2 (0.1) ^{Af, Ag}	68.1 (1.5) ^{Ae}	18.6 (0.4) ^{Ae}
		C4	14.37 (0.01)	31.8 (0.3) ^{Ah}	3.5 (0.0) ^{Ag}	6.6 (0.1) ^{Af}	66.6 (0.7) ^{Ae}	20.7 (0.3) ^{Ah}
30	MWC	C1	15.30 (0.25)	32.1 (0.0) ^{Be}	4.3 (0.0) ^{Be}	5.7 (0.1) ^{Be}	57.0 (1.1) ^{Be}	15.4 (0.3) ^{Be}
		C2	13.79 (0.02)	31.5 (0.4) ^{Be, Bf}	4.2 (0.0) ^{Be}	5.9 (0.1) ^{Be, Bf}	58.8 (0.2) ^{Bf}	14.8 (0.1) ^{Be, Bf}
		C3	13.94 (0.26)	32.2 (0.3) ^{Bg}	4.3 (0.0) ^{Be}	5.9 (0.1) ^{Be, Ag}	58.7 (0.6) ^{Be}	15.6 (0.8) ^{Be, Bg}
		C4	14.31 (0.03)	31.3 (0.5) ^{Bf, Ah}	4.4 (0.0) ^{Bh}	5.7 (0.1) ^{Be, Bh}	61.2 (1.0) ^{Be}	14.6 (0.3) ^{Be, Bh}
45	MWC	C1	8.74 (0.03)	33.4 (0.1) ^{Ce}	4.6 (0.0) ^{Ce}	5.8 (0.0) ^{Ce, Be}	53.1 (1.0) ^{Ce}	15.4 (0.2) ^{Ce, Be}
		C2	13.38 (0.04)	34.7 (0.0) ^{Cf}	4.6 (0.0) ^{Cf}	6.3 (0.0) ^{Cf, Bf}	53.7 (1.3) ^{Ce}	14.0 (0.9) ^{Cf, Bf}
		C3	12.00 (0.22)	34.6 (0.2) ^{Cf}	4.6 (0.0) ^{Cf}	6.0 (0.0) ^{Ce, Cf, Ag}	55.0 (1.8) ^{Ce}	15.0 (0.0) ^{Ce, Cf, Bg}
		C4	9.11 (0.03)	34.1 (0.3) ^{Ce, Cf}	4.6 (0.0) ^{Ce, Cf}	6.0 (0.1) ^{Ce, Cf, Bh}	54.0 (1.0) ^{Ce}	14.1 (0.9) ^{Ce, Cf, Bh}

¹Tukey's test at 5% significance level for the same CDS level at varying microwave power levels (a, b,c,d) or at two microwave convection settings (e,f,g,h) and for the same microwave power or microwave convection setting at varying CDS level (A,B,C). Values followed by same set of letters are not significantly different; ²P4 – 40% power (420 W); P6 – 60% power (676 W); P8 – 80% power (701 W); P10 – 100% power (805 W); C1 - 130°C, 30% power (303 W); C2 -150°C, 30% power (316 W); C3 - 160°C, 30% power (326 W); and C4 -190°C, 30% power (332 W).

Table 5.5 P-values from analysis of variance (ANOVA) runs for physico-chemical properties of wheat distillers dried grain with solubles (DDGS), produced at varying condensed distillers solubles (CDS) level and dried under microwave (MW) and microwave convection (MWC) methods

Property	Microwave-dried samples			Microwave convection-dried samples		
	CDS level	MW power	Interaction	CDS level	MWC setting	Interaction
1. Chemical composition						
Protein	0.000	0.002	0.060	0.000	0.242	0.000
Ash	0.000	0.001	0.048	0.000	0.000	0.000
Fat	0.000	0.010	0.006	0.000	0.041	0.001
Neutral detergent fiber	0.000	0.024	0.001	0.000	0.014	0.008
Acid detergent fiber	0.000	0.650	0.000	0.000	0.400	0.007
2. Physical attributes						
Particle size	0.000	0.493	0.000	0.000	0.107	0.340
Bulk density	0.000	0.001	0.000	0.000	0.000	0.002
Particle density	0.000	0.001	0.618	0.000	0.489	0.004
3. Color						
L (lightness)	0.002	0.032	0.200	0.315	0.018	0.114
a (redness)	0.001	0.001	0.228	0.000	0.218	0.432
b (yellowness)	0.001	0.001	0.277	0.008	0.070	0.120
4. Flow properties						
Compressibility	0.002	0.205	0.313	0.038	0.547	0.187
Angle of repose	0.003	0.805	0.195	0.041	0.167	0.539
Ave. angle of spatula	0.017	0.010	0.035	0.041	0.049	0.085
Uniformity coefficient	0.000	0.669	0.122	0.000	0.390	0.764
Angle of fall	0.449	0.010	0.273	0.075	0.437	0.110
Angle of difference	0.023	0.020	0.056	0.045	0.500	0.009
Dispersibility	0.000	0.000	0.003	0.005	0.002	0.102
5. Compression characteristics						
Pellet density	0.000	0.145	0.612	0.000	0.055	0.184
Specific compression energy	0.000	0.000	0.000	0.000	0.000	0.000
Specific extrusion energy	0.517	0.755	0.042	0.000	0.021	0.125
Kawakita-Ludde model d	0.000	0.000	0.008	0.000	0.001	0.005
Kawakita-Ludde model f^{-1}	0.000	0.009	0.000	0.000	0.005	0.001
6. Thermal properties						
Conductivity	0.014	1.000	0.296	0.125	0.024	0.124
Diffusivity	0.000	0.356	0.422	0.007	0.585	0.003
7. Frictional properties						
Coefficient of internal friction	0.001	0.435	0.543	0.089	0.037	0.399
Cohesion	0.186	0.245	0.136	0.056	0.871	0.901

Table 5.6 Particle size, density, porosity, and color ¹ of wheat dried distillers grains solubles (DDGS) samples, produced at varying condensed distillers solubles (CDS) level and dried under microwave (MW) and microwave convection (MWC) methods. Values in parentheses represent standard deviation (N = 2)

CDS, % by mass	Drying parameters		Moisture, % wet basis	Particle size, mm	Bulk density, kg/m ³	Particle density, kg/m ³	Porosity, %	Color parameters ³		
	Method	Setting ²						L	a	b
15	MW	P4	8.33 (0.47)	0.82 (0.01) ^{Aa}	329 (4) ^{Aa}	1321 (1) ^{Aa}	75.1	42.3 (1.1) ^{Aa}	6.6 (0.1) ^{Aa}	14.8 (0.3) ^{Aa}
	MW	P10	8.92 (0.13)	0.88 (0.02) ^{Ab}	312 (4) ^{Ab}	1314 (1) ^{Ab}	76.3	42.4 (0.4) ^{Ab}	6.8 (0.1) ^{Ab}	15.1 (0.1) ^{Ab}
30	MW	P4	9.32 (0.09)	0.72 (0.00) ^{Ba}	351 (3) ^{Ba}	1358 (3) ^{Ba}	74.2	43.5 (0.9) ^{Ba}	6.8 (0.0) ^{Ba,Aa}	15.4 (0.4) ^{Ba}
	MW	P10	8.98 (0.12)	0.78 (0.02) ^{Bb}	334 (8) ^{Bb}	1347 (5) ^{Bb}	75.2	44.2 (0.8) ^{Bb}	6.9 (0.1) ^{Bb,Ab}	15.9 (0.4) ^{Bb}
45	MW	P4	8.31 (0.14)	0.59 (0.03) ^{Ca}	419 (2) ^{Ca}	1383 (1) ^{Ca}	69.6	43.3 (0.3) ^{Ca,Ba}	6.8 (0.2) ^{Ca}	15.1 (0.3) ^{Ca,Ba}
	MW	P10	8.45 (0.26)	0.50 (0.01) ^{Cb}	428 (2) ^{Cb}	1374 (2) ^{Cb}	68.5	45.0 (0.5) ^{Cb,Bb}	7.1 (0.1) ^{Cb}	16.0 (0.2) ^{Cb,Bb}
15	MWC	C1	9.25 (0.24)	0.80 (0.02) ^{Ac,Ad}	341 (5) ^{Ac}	1321 (1) ^{Ac}	74.2	44.3 (0.5) ^{Ac}	6.4 (0.1) ^{Ac}	15.1 (0.3) ^{Ad,Ac}
	MWC	C4	9.57 (0.27)	0.78 (0.01) ^{Ad}	345 (5) ^{Ac}	1325 (1) ^{Ad}	74.0	43.7 (0.4) ^{Ad}	6.4 (0.1) ^{Ac}	15.0 (0.2) ^{Ad}
30	MWC	C1	8.77 (0.11)	0.77 (0.00) ^{Ac,Bc,Cc}	367 (7) ^{Bc}	1351 (2) ^{Bc}	72.8	44.7 (0.2) ^{Ac}	6.7 (0.1) ^{Bc}	15.8 (0.1) ^{Ac,Bc,Bd,Cc}
	MWC	C4	9.09 (0.19)	0.76 (0.02) ^{Ad,Bc,Cd}	371 (4) ^{Bc}	1346 (0) ^{Bd}	72.4	42.6 (1.6) ^{Ad}	6.7 (0.0) ^{Bc}	15.0 (0.6) ^{Bc,Ad}
45	MWC	C1	9.02 (0.23)	0.64 (0.03) ^{Cc}	391 (3) ^{Cc}	1356 (0) ^{Cc}	71.2	44.4 (0.2) ^{Ac}	6.9 (0.1) ^{Cc}	15.7 (0.2) ^{Cc,Cd}
	MWC	C4	9.18 (0.35)	0.59 (0.01) ^{Cc,Cd}	420 (6) ^{Cd}	1357 (0) ^{Cc}	69.0	44.2 (0.4) ^{Ad}	7.0 (0.1) ^{Cc}	15.7 (0.3) ^{Cd,Bd}

¹Tukey's test at 5% significance level for the same CDS level at two microwave power levels (a, b) or at two microwave convection settings (c,d) and for the same microwave power or microwave convection setting at varying CDS level (A,B,C). Values followed by same set of letters are not significantly different;

²P4 – 40% power (420 W); P10 – 100% power (805 W); C1 - 130°C, 30% power (303 W); C4 -190°C, 30% power (332 W).

³The color parameters Hunter L, a, and b represent lightness, redness, and yellowness, respectively;

5.4.2.1 *Particle size and size distribution*

Average particle size ranged from 0.50 mm to 0.88 mm (Table 5.6). In microwave-dried samples, CDS level, microwave power, and their interaction had significant effect on particle size (Table 5.5). At the same CDS level, average particle sizes of samples dried under the two microwave power levels were significantly different although no consistent trend was observed. At the same microwave power level, however, samples with lower CDS level consistently showed larger particle sizes than those with higher CDS. Samples with 15% CDS, for example, had larger particle size than those with 30% and 45% CDS. Similarly, those with 30% CDS had significantly larger particle size than those with 45% CDS. In microwave convection-dried samples, only CDS level had a significant effect on particle size (Table 5.5). Samples with 15% CDS had significantly larger particle sizes than those with 45% CDS. Figure 5.3 illustrates this trend for both microwave- and microwave convection-dried samples. Decrease in size with increased CDS level is attributed to the increased presence of finer, endosperm- and germ-derived solids and decreased amount of the fibrous, bran-related particles. The effect of CDS level on particle size was still discernible even though these samples were ground after drying to generate the bulk material. The use of a screen size that was four times larger than the geometric mean diameter of the plant-sourced sample had helped curb the adverse effect of grinding.

Figure 5.3 also shows the particle size distribution of the plant-sourced wheat DDGS sample in comparison to those of the laboratory-prepared microwave- and microwave convection-dried samples. The plant-sourced sample tended to have finer particle sizes than the laboratory prepared samples, suggesting that the former may have been produced at a CDS level higher than what was employed in the study. This sample was obtained from an ethanol plant that employed

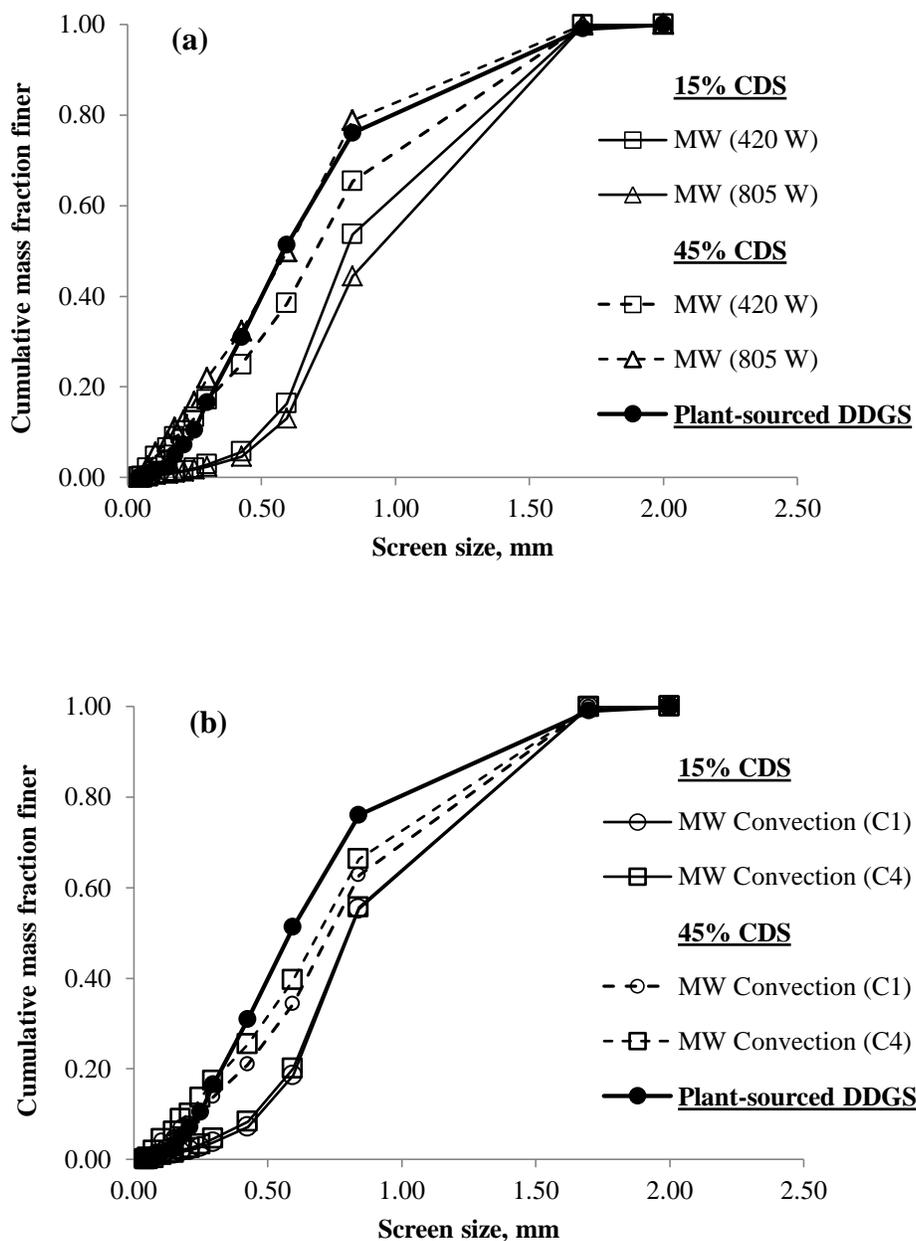


Fig. 5.3. Average particle size distribution of laboratory-prepared (8-9% moisture, wet basis) and ethanol plant-sourced (9% moisture, wet basis) wheat distillers dried grain with solubles (DDGS) samples (N = 2). The samples were prepared at 15%, 30%, and 45% condensed distillers solubles (CDS) and dried under microwave (MW) and microwave convection methods (Figure 5.3a and Figure 5.3b, respectively). Error bars were not presented because standard errors were very small.

a ring type dryer for DDGS production, wherein the WDGS is dispersed and conveyed through the dryer in a hot air stream at high speed. Incidence of particle agglomeration was not a concern compared to rotary drum drying. Plant-scale studies (Kingsly et al. 2010; Clementson and Ileleji 2012) on corn DDGS indicated that particle size increased as CDS level increased due to the agglomeration of particles during rotary drum drying, with CDS acting as a binding agent.

5.4.2.2 *Density and porosity*

Similar trends in particle size (Table 5.6) and particle size distribution (Fig. 5.1) were also observed in forced-air convection-dried wheat DDGS samples produced at the same CDS levels. (Mosqueda et al. 2013b). Bulk density values ranged from $312 \text{ kg}\cdot\text{m}^{-3}$ to $428 \text{ kg}\cdot\text{m}^{-3}$ (Table 5.6). CDS level, microwave power, and their interaction also had significant effect on the bulk density of microwave- and microwave convection-dried samples (Table 5.5). The ANOVA sums of squares associated with CDS level, however, accounted between 88% to 97% of the total sums of squares in both sample types, suggesting a stronger influence of CDS level compared to the other factors (microwave power, microwave convection setting). Samples with 45% CDS had significantly higher bulk density than those with 15% and 30% CDS. Similarly, those with 30% CDS had significantly higher bulk density than those with 15% CDS.

Particle density values ranged from 1314 kgm^{-3} to 1383 kgm^{-3} (Table 5.6). In microwave-dried samples, particle density was significantly affected by CDS level (Table 5.5). It increased as CDS level in the blend increased. Although CDS level – microwave convection setting interaction significantly affected the particle density of microwave convection-dried samples (Table 5.5), CDS level still have a dominant influence, with its associated ANOVA sums of squares accounted about 98% of the total sums of squares.

Increased CDS level in the blend translates to increased presence of finer but heavier solids and decreased amount of the larger, more fibrous particles. These finer particles would easily move through and fill the inter-particle spaces, consequently leading to a heavier bulk mass. Thus, porosity values also tended to decrease with increased CDS level in both sample types (Table 5.6). Bulk and particle density values of microwave- and microwave convection-dried samples also mirrored the trends reported by Mosqueda et al. (2013b) for forced-air convection-dried wheat DDGS samples. In corn DDGS, Ganesan et al. (2008a) reported that solubles level, along with moisture content, had significant effect on aerated and tapped bulk densities but did not observe a specific trend.

5.4.2.3 Color

Table 5.6 also shows the color parameters of microwave-, and microwave convection-dried samples. CDS level – microwave power and CDS level – microwave convection setting interactions did not have a significant effect on the color parameters of both sample types (Table 5.5).

In microwave-dried samples, both CDS and microwave power levels had significant effect on the lightness (L), redness (a), and yellowness (b) parameters. Those with 30% and 45% CDS had significantly higher L values (lighter) compared to those with 15% CDS. Samples dried under the higher microwave power level (P10 or 805 W) had also significantly higher L values than those dried under the P4 (420 W) setting. Samples with higher CDS were significantly redder and yellower, as well as lighter compared to those with lower CDS. Also, samples dried under the higher microwave power level (P10) were also significantly redder and yellower than those dried under lower microwave power.

In microwave convection-dried samples, the lightness parameter was significantly affected by microwave convection setting while the other two parameters were significantly by CDS level. Samples dried under the lower setting (C1, nominally at 130°C-30% power) had significantly higher L values (lighter) than those dried under the higher setting (C4, nominally at 190°C-30% power). Thus, at the same power level, higher temperature setting results in darker dried samples. Similar to the microwave-dried samples, those with higher CDS exhibited significantly higher intensity of redness and yellowness compared to those with lower CDS (Table 5.6).

Color changes during drying are attributed to Maillard reaction, which involves the interaction of reducing sugars with the amino groups of lysine (Fayle and Gerrard 2002). Factors such as temperature, time, water activity, and chemical composition, among others, affect the rate of Maillard reaction (Ames 1990; Owusu-Apenten 2004). Use of elevated temperatures during microwave convection drying and increased presence of sugars and protein (due to increased CDS level) caused increase in Maillard reaction rate and thus, increased darkness and the other Hunter color attributes.

5.4.2.4 *Flow properties*

Table 5.7 shows the various flow property measurements made from the wheat DDGS samples, from which the flowability and floodability indices were derived. The previously presented Table 5.5 also summarized the ANOVA results for these measurements.

Flowability index, which comprised the sum of the index values of compressibility, angle of repose, average angle of spatula, and uniformity coefficient, ranged from 69.5 to 75.5 in microwave-dried samples and from 70.3 to 74.8 in microwave convection-dried samples. Both sample types were classified as having a “fair” degree of flowability, which would sometimes

Table 5.7 Flow properties¹ of wheat distillers dried grain with solubles (DDGS) samples, produced at varying condensed distillers solubles (CDS, % by mass) level and dried under microwave (MW), and microwave convection (MWC) methods. Values in parentheses represent standard deviation (N = 2)

CDS, % mass	Drying parameters		Moisture, % wb	Compressibility, %	Angle of repose, °	Ave angle of spatula, °	Uniformity coefficient	Angle of fall, °	Angle of Difference, °	Dispersibility, %
	Method	Setting ²								
15	MW	P4	8.39 (0.34)	14.9 (0.7) ^{Aa}	41.9 (0.0) ^{Aa}	50.8 (1.3) ^{Aa,Ca}	2.3 (0.0) ^{Aa}	38.8 (0.6) ^{Aa}	3.1 (0.6) ^{Aa}	31.1 (2.1) ^{Aa}
	MW	P10	8.96 (0.10)	14.0 (1.1) ^{Aa,Ab}	43.4 (0.1) ^{Aa,Bb}	48.5 (0.0) ^{Aa,Bb}	2.6 (0.3) ^{Aa}	34.4 (0.3) ^{Ab}	9.0 (0.1) ^{Ab}	27.0 (0.1) ^{Ab}
30	MW	P4	9.00 (0.35)	16.9 (1.0) ^{Ba,Aa}	45.6 (1.3) ^{Ba,Aa}	51.1 (2.1) ^{Ba,Ca}	3.3 (0.0) ^{Ba}	36.7 (2.3) ^{Ba,Aa}	9.0 (1.1) ^{Ba,Aa}	21.9 (0.9) ^{Ba, Aa}
	MW	P10	9.67 (0.09)	14.5 (2.2) ^{Ba,Ab}	44.1 (1.8) ^{Bb}	44.9 (0.1) ^{Bb,Cb}	3.2 (0.0) ^{Ba}	35.4 (1.0) ^{Bb,Ab}	8.7 (2.8) ^{Bb,Ab}	20.0 (2.4) ^{Ba}
45	MW	P4	8.52 (0.01)	19.7 (0.8) ^{Ca}	47.2 (1.1) ^{Ca,Ba}	51.7 (0.8) ^{Ca,Aa}	4.0 (0.4) ^{Ca}	38.4 (1.4) ^{Ca,Aa}	8.8 (0.4) ^{Ca,Ba}	25.7 (0.6) ^{Ca}
	MW	P10	8.52 (0.07)	20.1 (0.6) ^{Ca}	46.9 (0.8) ^{Cb,Bb}	51.8 (1.8) ^{Ca,Cb}	3.7 (0.0) ^{Ca}	36.1 (0.8) ^{Cb,Ab}	10.9 (1.7) ^{Cb,Bb}	12.4 (0.0) ^{Cb}
15	MWC	C1	9.95 (0.16)	16.0 (2.0) ^{Ac}	41.8 (0.1) ^{Ac}	50.1 (2.2) ^{Ac}	2.5 (0.0) ^{Ac}	35.4 (0.9) ^{Ad,Ac}	6.5 (1.1) ^{Ac}	24.8 (0.8) ^{Ac}
	MWC	C4	9.50 (0.01)	13.6 (2.0) ^{Ac,Ad}	44.0 (2.6) ^{Ac,Ad}	48.9 (1.5) ^{Ad}	2.6 (0.0) ^{Ac}	33.6 (2.0) ^{Ad}	9.4 (2.1) ^{Ad}	19.9 (3.7) ^{Ad}
30	MWC	C1	8.39 (0.15)	15.5 (0.9) ^{Bc,Ac}	46.1 (0.8) ^{Bc,Ac,Cc}	46.6 (0.9) ^{Bc,Ac}	3.4 (0.0) ^{Bc}	36.7 (0.5) ^{Bd,Ac}	9.5 (0.4) ^{Bc,Ac}	22.7 (0.6) ^{Bc,Ac}
	MWC	C4	8.99 (0.09)	14.0 (2.1) ^{Bc}	46.1 (1.8) ^{Bc,Ad,Cd}	49.7 (2.0) ^{Bd,Ad}	3.4 (0.1) ^{Bc,Bd}	39.7 (3.9) ^{Bd,Ad}	6.4 (2.1) ^{Bd,Ad}	20.2 (1.0) ^{Bd,Ad}
45	MWC	C1	8.68 (0.47)	17.1 (1.3) ^{Cc}	44.6 (0.1) ^{Cc}	49.5 (0.2) ^{Cc,Bc}	3.6 (0.2) ^{Cc,Bc}	36.9 (0.5) ^{Cd,Ac}	7.7 (0.6) ^{Cc,Ac}	20.4 (0.9) ^{Cc}
	MWC	C4	9.09 (0.27)	19.2 (0.2) ^{Cc}	46.3 (1.1) ^{Cc,Cd}	54.4 (1.9) ^{Cd,Bd}	3.6 (0.2) ^{Cc,Bd}	32.7 (2.0) ^{Cd,Ad}	13.6 (0.8) ^{Cd}	10.9 (2.4) ^{Cd}

¹Tukey's test at 5% significance level for the same CDS level at two microwave power levels (a, b) or at two microwave convection settings (c,d) and for the same microwave power or microwave convection setting at varying CDS level (A,B,C). Values followed by same set of letters are not significantly different.

²P4 – 40% power (420 W); P10 – 100% power (805 W); C1 - 130°C, 30% power (303 W); C4 -190°C, 30% power (332 W).

require vibration to assure flow (Carr 1965). A previous laboratory-scale study on wheat DDGS samples produced at the same CDS levels but dried under forced-air convection method also yielded a similar range of flowability index values (Mosqueda et al. 2013b).

Compressibility, angle of repose, and uniformity coefficient of both microwave- and microwave convection-dried samples were significantly affected by CDS level (Table 5.5). Higher CDS blends tended to be more compressible, more differentiated in terms of particle size (higher uniformity coefficient), having steeper angles of repose, and therefore, less flowable, than samples with lower CDS level (Table 5.7). In terms of angle of spatula, its values in microwave-dried samples were significantly affected by the CDS level – microwave power interaction (Table 5.5). At 30% CDS, samples dried under the lower microwave power (P4 or 420 W) had significantly higher angle of spatula than those dried under P10 (805 W). For the P10-dried samples, those with 45% CDS showed higher angle of spatula than those with 30% CDS. The rest of the treatment combinations were statistically similar. In microwave convection-dried samples, the average angles of spatula were significantly affected by both CDS level and microwave convection setting (Table 5.5). Samples dried at the higher microwave convection setting and those with 45% CDS had significantly higher angles of spatula.

Increasing the CDS level in the blend increased the presence of finer solids and decreased the amount of fibrous particles, thus, leading to slightly more differentiated particle sizes. This also led to increased inter-particle friction because of the greater surface area of contact brought about by the higher percentage of fines, leading to steeper angle of repose and average angle of spatula.

Floodability index, on the other hand, ranged from 62.0 to 67.5 in microwave-dried samples and from 60.0 to 66.8 in microwave convection-dried samples. These values were derived from the

sum of the index values of flowability, angle of fall, angle of difference, and dispersibility (Carr 1965). Both sample types were classified as “floodable” and require the use of a rotary seal to prevent flushing (Carr, 1965). This means, for example, that in designing feed systems for these samples, adequate measures like rotary valves should be used to ensure that the bulk material is metered out under control. Forced air convection-dried wheat DDGS samples produced at the same CDS levels were also considered floodable (Mosqueda et al. 2013b).

Dispersibility values ranged from 10.0% to 31.1% (Table 5.7) and spanned the “inclined to flood” and “floodable” categories (Carr 1965). These were significantly affected by the CDS level- microwave power interaction in microwave-dried samples and by both CDS level and microwave convection setting in microwave convection-dried samples (Table 5.5). At the same CDS level (such as those at 15% and 45% CDS), samples dried under the lower microwave power level (P4, 420 W) were significantly more dispersible than those dried under the P10 (805 W) setting. Samples dried at the same microwave power level showed that those with 15% CDS were significantly more dispersible than those with 30% and 45% CDS. Lower CDS blends tended to be lighter in mass, less dense and are, therefore, more dispersible compared to higher CDS blends.

The angle of fall of microwave- and microwave convection-dried samples, which ranged from 32.7° to 38.8° (Table 5.7), fell under the “floodable” category (Carr 1965). Materials with lower angles of fall, for example between 10° to 25°, are considered to be very floodable. Table 5.5 shows that only microwave power affected angle of fall, with samples dried under the lower microwave power level showing significantly higher values.

The angle of difference, which was obtained by getting the difference between the angle of repose and the angle of fall, ranged from 3.1° to 13.6° (Table 5.7), straddling between the “won’t flood” to “floodable” categories (Carr 1965). In microwave-dried samples, it was significantly affected by both microwave power and CDS level. Samples with 45% CDS had significantly higher values compared to those with 15% CDS. Those dried under the higher microwave power level also had significantly higher angle of difference. In microwave convection-dried samples, the CDS level-microwave convection setting interaction significantly affected the angle of difference. The higher microwave convection setting-dried (C4) samples had significantly higher values when their CDS content was at 45% than when at the 15% or 30% level. In samples with 15% and 45% CDS, significantly higher angles of difference were observed when these were dried under the higher microwave convection setting than under the lower setting.

5.4.2.5 *Compression characteristics*

Table 5.8 shows the pellet density, the specific energy consumption during the compression and extrusion processes, and the parameters of the Kawakita-Ludde model. This model adequately described the compression characteristics of wheat DDGS samples. ANOVA results are presented in Table 5.5.

Pellet density values of microwave- and microwave convection-dried samples ranged from 1162 kg·m⁻³ to 1210 kg·m⁻³ (Table 5.8) and were about 85-88% of particle density values (Table 5.6). These values were significantly affected by CDS level (Table 5.5). In both sample types, blends with higher CDS produced significantly denser pellets than those with lower CDS level. These values were also close to those reported for wheat DDGS samples (1153-1189 kg·m⁻³) produced at the same CDS levels but dried under forced-air convection method (Mosqueda et al. 2013b).

Table 5.8 Compression characteristics¹ of wheat distillers dried grain with solubles (DDGS), produced at varying condensed distillers solubles (CDS) level and dried under microwave (MW) and microwave convection (MWC) methods. Values in parentheses represent standard deviation (N = 5)

CDS, % mass	Drying		Moisture, % wet basis	Pellet density, kgm ⁻³	Specific energy consumption, MJt ⁻¹		Kawakita-Ludde model parameters ³			
	Method	Setting ²			Compression	Extrusion	d	f ¹ , MPa	R ²	MSE
15	MW	P4	8.49 (0.27)	1154 (8) ^{Aa}	10.96 (0.29) ^{Aa}	0.40 (0.08) ^{Aa}	0.72 (0.00) ^{Aa}	0.59 (0.03) ^{Aa}	0.975	0.000
	MW	P10	9.04 (0.12)	1158 (13) ^{Aa}	18.18 (1.23) ^{Ab}	0.34 (0.15) ^{Aa,Ab}	0.77 (0.01) ^{Ab}	1.84 (0.29) ^{Ab}	0.991	0.000
30	MW	P4	9.50 (0.11)	1176 (9) ^{Ba}	12.01 (2.41) ^{Ba,Aa}	0.27 (0.10) ^{Ba,Aa}	0.72 (0.01) ^{Ba}	1.10 (0.43) ^{Ba}	0.990	0.000
	MW	P10	8.18 (0.09)	1187 (13) ^{Ba,Bb}	10.73 (1.12) ^{Ba}	0.42 (0.11) ^{Bb,Ab}	0.73 (0.01) ^{Ba}	0.71 (0.16) ^{Bb}	0.992	0.000
45	MW	P4	8.90 (0.04)	1180 (9) ^{Ca,Ba}	12.30 (1.44) ^{Ca,Aa}	0.35 (0.10) ^{Ca,Aa}	0.68 (0.01) ^{Ca,Aa}	1.58 (0.34) ^{Ca}	0.992	0.000
	MW	P10	8.76 (0.17)	1183 (10) ^{Ca,Bb}	12.72 (0.70) ^{Ca}	0.29 (0.03) ^{Ca,Ab}	0.67 (0.01) ^{Ca}	1.61 (0.27) ^{Ca,Ab}	0.994	0.000
15	MWC	C1	9.26 (0.03)	1153 (12) ^{Ac}	9.51 (0.26) ^{Ac}	0.41 (0.05) ^{Ac}	0.70 (0.01) ^{Ac}	0.42 (0.04) ^{Ac}	0.967	0.000
	MWC	C4	9.40 (0.29)	1153 (16) ^{Ac}	9.22 (0.26) ^{Ac}	0.47 (0.04) ^{Ad}	0.69 (0.00) ^{Ac,Ad}	0.38 (0.02) ^{Ac}	0.967	0.000
30	MWC	C1	8.44 (0.24)	1174 (3) ^{Bc}	17.18 (0.63) ^{Bc}	0.35 (0.01) ^{Bc}	0.71 (0.01) ^{Bc}	1.90 (0.17) ^{Bc}	0.995	0.000
	MWC	C4	9.13 (0.05)	1189 (4) ^{Bc,Bd}	11.46 (1.03) ^{Bd}	0.34 (0.04) ^{Bd}	0.71 (0.01) ^{Bc}	1.03 (0.21) ^{Bd}	0.994	0.000
45	MWC	C1	9.03 (0.14)	1181 (8) ^{Cc,Bc}	14.60 (2.55) ^{Cc}	0.28 (0.04) ^{Cc,Bc}	0.71 (0.01) ^{Cc,Ac}	1.81 (0.50) ^{Cc,Bc}	0.982	0.000
	MWC	C4	9.32 (0.03)	1186 (6) ^{Cc,Bd}	13.36 (0.79) ^{Cc,Bd}	0.34 (0.04) ^{Cd,Bd}	0.69 (0.00) ^{Cd,Ad}	1.82 (0.30) ^{Cc}	0.988	0.000

¹Tukey's test at 5% significance level for the same CDS level at two microwave power levels (a, b) or at two microwave convection settings (c,d) and for the same microwave power or microwave convection setting at varying CDS level (A,B,C). Values followed by same set of letters are not significantly different.

²P4 – 40% power (420 W); P10 – 100% power (805 W); C1 - 130°C, 30% power (303 W); C4 -190°C, 30% power (332 W).

³The model parameters d and f¹ relate to initial porosity and failure stress, respectively; R² and MSE stands for coefficient of determination and mean square error, respectively.

The effect of CDS level on the pellet density of these forced-air convection-dried samples was also similar. High CDS blends produced denser pellets.

Specific energy consumption during compression ranged from 8.8 to 19.8 MJt⁻¹ and accounted about 95-99% of the total specific energy consumption (Table 5.8). Specific energy consumption in extruding the pellet out of the die, on the other hand, ranged from 0.17 to 0.60 MJ·t⁻¹ (Table 5.8) and comprised the remaining 1-5% of the total specific energy consumption. Similar percentage breakdown of total specific energy consumption was reported for forced-air convection-dried wheat DDGS samples with the same CDS levels although the corresponding range of energy consumption values were slightly narrower: 9.9-17.2 MJt⁻¹ during compression and 0.20-0.45 MJ·t⁻¹ during extrusion (Mosqueda et al. 2013b).

Factorial interactions affected the specific energy consumption during compression of both microwave- and microwave convection-dried samples (Table 5.5). Samples with 15% CDS and dried under the higher microwave power level (P10, 805 W) consumed significantly higher energy during compression than those dried under P4 (420 W). Among those dried under the P10 microwave power level, samples with 15% CDS consumed significantly higher specific energy during compression than those with 30% and 45% CDS. In microwave convection-dried samples, those with 30% CDS and dried under the lower microwave convection setting showed significantly higher specific energy consumption than those dried under the higher setting (C4). At the same microwave convection setting, samples with higher CDS level consistently showed significantly higher specific energy consumption than those with lower CDS.

Specific energy consumption during pellet extrusion of microwave convection-dried samples was significantly affected by both CDS level and microwave convection setting (Table 5.5).

Samples with 15% CDS consumed significantly higher energy than those with 30% and 45% CDS, respectively. Samples dried under the higher microwave convection setting (C4) also consumed significantly higher energy per unit mass. The specific energy consumption in extruding microwave-dried pellets was statistically similar in all treatment combinations except for those with 30% CDS, wherein samples prepared under the higher microwave power (P10) consumed significantly higher energy than those prepared under the lower microwave power level (P4).

The Kawakita-Ludde model gave initial porosity (d) values ranging from 0.67 to 0.77 in microwave-dried samples and from 0.69 to 0.71 in microwave convection-dried samples (Table 5.8). These were close to the bulk porosity values presented in Table 5.2: 0.68-0.76 for microwave-dried samples and 0.69-0.74 for microwave convection-dried samples. Failure stress (f^1), on the other hand, ranged from 0.59 MPa to 1.84 MPa in microwave-dried samples and from 0.38 MPa to 1.82 MPa in microwave convection-dried samples (Table 5.8). The same model also adequately represented the compression characteristics of forced-air convection-dried wheat DDGS samples at the same CDS levels (Mosqueda et al. 2013b). The reported values for porosity and failure stress values were also comparative to the above results. Porosity values of forced-air convection-dried samples were from 0.68 to 0.75 while failure stress ranged from 0.49 MPa to 2.38 MPa.

Both model parameters were significantly affected by factorial interactions (Table 5.5). In terms of the initial porosity model parameter, microwave-dried samples with 15% CDS were significantly more porous than those with 30% and 45% CDS. This sample group also showed significantly higher porosity when dried under the P4 (420 W) than under P10 (805 W). In microwave convection-dried samples, those with 45% CDS and dried under the lower

microwave convection setting had significantly higher porosity than those dried under the higher setting. At the same microwave convection setting, samples with 30% CDS showed significantly higher porosity values than those with 15% CDS.

At each dryer setting, higher CDS blends in both microwave- and microwave convection-dried sample groups showed significantly higher failure stress (f^{-1}) values than those with lower CDS (Table 5.8). Pellets with higher CDS level would be harder to break than those with lower CDS. At each CDS level, however, no consistent trend could be observed on the failure stress differences due to dryer setting (Table 5.8). In microwave convection-dried samples, only those with 30% CDS showed significant difference, wherein those dried under the lower microwave convection setting showing significantly higher failure stress. In microwave-dried samples with 15% and 30% CDS, there were significant differences due to microwave power level but no consistent trend was observed.

Increased pellet density and failure stress of samples with higher CDS level would be attributed to the increased amount of finer particles in the blend as well as to the chemical composition. With increased CDS level, there is increased presence of finer particles that would easily fill the voids between larger particles during compression. These finer particles also have greater surface area for contact, thus, enhancing binding. Higher CDS level also translates to higher protein and ash but lower fat and fiber content. Heat applied during densification, as well as heat resulting from friction, may have also altered the state of the chemical constituents and could have promoted better binding characteristics. For example, protein could have been denatured, and combined with the sugars present, positively affected the strength of the pellets, as a result of Maillard reaction (Thomas and van der Poel 1996).

5.4.2.6 *Thermal properties*

Table 5.9 presents the thermal conductivity and thermal diffusivity values of the laboratory-prepared wheat DDGS samples while Table 5.5 gives the corresponding ANOVA results. Thermal conductivity of both microwave- and microwave convection-dried samples ranged from $0.04 \text{ W}\cdot\text{m}^{-1}\cdot\text{C}^{-1}$ to $0.06 \text{ W}\cdot\text{m}^{-1}\cdot\text{C}^{-1}$, while thermal diffusivity values ranged from $1.4 \times 10^{-7} \text{ m}^2\cdot\text{s}^{-1}$ to $1.7 \times 10^{-7} \text{ m}^2\cdot\text{s}^{-1}$. These were similar to those reported for the forced-air convection-dried wheat DDGS samples at the same CDS levels (Mosqueda et al. 2013b). Thermal conductivity of forced-air convection samples was $0.05 \text{ W}\cdot\text{m}^{-1}\cdot\text{C}^{-1}$ while the thermal diffusivity ranged from $1.35 \text{ m}^2\cdot\text{s}^{-1}$ to $1.65 \text{ m}^2\cdot\text{s}^{-1}$.

Both thermal properties of microwave-dried samples were significantly affected by CDS level (Table 5.5). Samples with 15% CDS had significantly higher thermal diffusivity than those with 30% and 45% CDS. Similarly, 30% CDS samples had significantly higher thermal diffusivity than those with 45% CDS. Thermal conductivity was significantly higher in samples with 45% CDS than those with 15% CDS (Table 5.9).

In microwave convection-dried samples, thermal conductivity was significantly affected by microwave convection setting while thermal diffusivity was significantly affected by the CDS level–microwave convection setting interaction (Table 5.5). Samples dried under the lower microwave convection setting had significantly higher thermal conductivity than those dried under the higher microwave convection setting (Table 5.9). For samples with 15% and 45% CDS, there was significant difference in the thermal diffusivity between the two microwave convection setting but no consistent trend was observed. For samples dried under the higher

Table 5.9 Thermal and frictional properties¹ of wheat distillers dried grain with solubles (DDGS), produced at varying condensed distillers solubles (CDS) level and dried under microwave (MW) and microwave convection (MWC) methods. Values in parentheses represent standard deviation (N = 2)

CDS, % by mass	Drying		Moisture, % wet basis	Thermal properties (at 23°C)			Frictional properties ³			Standard error of estimate
	Method	Setting ²		Bulk density kgm ⁻³	Conductivity Wm ⁻¹ °C ⁻¹	Diffusivity, (x10 ⁻⁷) m ² s ⁻¹	Coefficient of internal friction	Cohesion, kPa	R ²	
15	MW	P4	8.49 (0.27)	336.5 (2.1)	0.04 (0.00) ^{Aa}	1.70 (0.00) ^{Aa}	0.66 (0.03) ^{Aa}	2.07 (1.06) ^{Aa}	0.994	1.37
	MW	P10	9.04 (0.12)	323.4 (0.6)	0.04 (0.00) ^{Aa,Ab}	1.70 (0.00) ^{Aa}	0.63 (0.00) ^{Aa}	3.19 (0.11) ^{Aa}	0.998	0.67
30	MW	P4	9.50 (0.11)	353.7 (1.6)	0.05 (0.00) ^{Ba,Aa}	1.60 (0.00) ^{Ba}	0.75 (0.02) ^{Ba}	2.68 (0.26) ^{Aa}	0.997	1.05
	MW	P10	8.18 (0.09)	334.7 (1.6)	0.05 (0.01) ^{Ba,Ab,Bb}	1.65 (0.01) ^{Ba}	0.74 (0.01) ^{Ba}	4.14 (1.12) ^{Aa}	0.999	0.66
45	MW	P4	8.90 (0.04)	429.6 (2.3)	0.05 (0.00) ^{Ca,Ba}	1.40 (0.00) ^{Ca}	0.69 (0.01) ^{Ca}	4.18 (0.41) ^{Aa}	0.995	1.19
	MW	P10	8.76 (0.17)	434.8 (0.6)	0.06 (0.01) ^{Ca,Bb}	1.40 (0.00) ^{Ca}	0.69 (0.00) ^{Ca}	3.29 (0.90) ^{Aa}	0.995	1.22
15	MWC	C1	9.26 (0.03)	352.1 (0.7)	0.05 (0.00) ^{Ac}	1.55 (0.01) ^{Ac}	0.63 (0.01) ^{Ac}	3.38 (0.33) ^{Ac}	0.998	0.74
	MWC	C4	9.40 (0.29)	352.7 (2.4)	0.04 (0.00) ^{Ad}	1.70 (0.00) ^{Ad}	0.64 (0.03) ^{Ad}	3.20 (0.97) ^{Ac}	0.996	1.06
30	MWC	C1	8.44 (0.24)	381.1 (0.1)	0.05 (0.00) ^{Bc,Ac}	1.55 (0.01) ^{Bc,Ac}	0.65 (0.01) ^{Bc,Ac}	2.08 (1.07) ^{Ac}	0.995	1.14
	MWC	C4	9.13 (0.05)	374.0 (0.6)	0.05 (0.01) ^{Bd,Ad}	1.55 (0.01) ^{Bc,Ad,Cd}	0.72 (0.02) ^{Bd,Ad}	2.37 (1.28) ^{Ac}	0.997	1.02
45	MWC	C1	9.03 (0.14)	395.1 (0.7)	0.05 (0.00) ^{Cc,Ac}	1.50 (0.00) ^{Cc,Ac}	0.64 (0.05) ^{Cc,Ac}	4.92 (1.91) ^{Ac}	0.985	2.03
	MWC	C4	9.32 (0.03)	426.7 (1.0)	0.05 (0.00) ^{Cd,Ad}	1.40 (0.00) ^{Cd}	0.68 (0.02) ^{Cd,Ad}	4.48 (0.41) ^{Ac}	0.997	0.96

¹ Tukey's test at 5% significance level for the same CDS level at two microwave power levels (a, b) or at two microwave convection settings (c,d) and for the same microwave power or microwave convection setting at varying CDS level (A,B,C). Values followed by same set of letters are not significantly different.

² P4 – 40% power (420 W); P10 – 100% power (805 W); C1 - 130°C, 30% power (303 W); C4 -190°C, 30% power (332 W).

³ R² and MSE stand for coefficient of determination and mean square error, respectively.

microwave convection setting, those with 15% CDS had significantly higher thermal diffusivity than those with 45% CDS.

Differences in thermal properties are attributed to differences in bulk density and porosity of the samples. Samples with higher CDS were denser and relatively less porous than those with lower CDS. Since air has a very low thermal conductivity (Sreenarayanan and Chattopadhyay 1986), it is expected that materials with high porosity would have lower thermal conductivity than those with less porosity. Similarly, since air has higher thermal diffusivity compared to that of water (Kostaropoulos and Saravacos 1997), more porous materials, like the 15% CDS samples, would have higher thermal diffusivity than the less porous, 45% CDS samples at the same moisture content.

5.4.2.7 *Frictional properties*

Table 5.9 also presents the coefficient of internal friction and cohesion values derived for microwave and microwave convection-dried samples. The corresponding ANOVA results are also shown in Table 5.5. Coefficient of internal friction and cohesion values ranged from 0.63 to 0.75 and from 2.07 kPa to 4.92 kPa, respectively. These were close to the coefficient of internal friction (0.63-0.74) and cohesion (2.74 - 4.93 kPa) values reported by Mosqueda et al. (2013b) for forced-air convection-dried wheat DDGS samples at the same CDS levels.

The coefficient of internal friction of microwave-dried samples was significantly affected by CDS level (Table 5.5). Samples with 30% and 45% CDS, for example, had significantly higher coefficient of internal friction than those with 15% CDS (Table 5.9). Samples with higher CDS are denser and less porous compared to those with lower CDS content due to increased presence

of finer particles. This facilitates greater surface contact area between particles, resulting to greater frictional resistance to motion and thus, higher coefficient of internal friction. In microwave convection samples, dryer settings had significant effect on the coefficient of internal friction (Table 5.5). Those dried under the higher microwave convection setting had significantly higher coefficient of internal friction (Table 5.9). Cohesion values, on the other hand, were statistically similar across CDS level, microwave power level, and microwave convection settings (Tables 5.5 and 5.9).

5.4.2.8 Summary

Of the 22 physical properties of microwave-dried samples presented in Tables 5.6 – 5.9, seven were significantly affected by CDS level only and 13 were significantly affected by both microwave power and CDS level or by the interaction of these two factors. In microwave convection-dried samples, eight properties were significantly affected by CDS level while nine were affected by both CDS level and microwave convection setting or their interaction. The influence of microwave power or microwave convection settings in these latter groups of properties was secondary to, or more subtle than, the effect of CDS level. ANOVA sums of squares results seemed to suggest this stronger influence of CDS level. At the same CDS level, no consistent pattern could be observed on the property variations due to microwave power or microwave convection settings.

Variability in some of the physical properties due to microwave and microwave convection drying methods and settings could be attributed to the structural changes that occur during the drying process. Krokida and Moroulis (1999), for example, reported that microwave and microwave vacuum drying decreased the bulk density and increased the porosity of apple,

banana, carrot, and potato compared to convection drying. Witrowa-Rajchert and Rzaca (2009) also reported the microstructure of convectively dried apples had small cavities and very high density, while those of the microwave convection- and infrared convection-dried samples had cells with larger cavities. Nowak and Lewicki (2005) also shared similar observations in their analysis of apple tissue images, wherein the microstructure of convectively dried apples had smaller cells compared to the infrared-dried apples. They, however, indicated that drying rate could be the probable cause of the microstructure differences. Higher drying rates could result to larger shrinkage stresses and greater tissue damage than low drying rates (Nowak and Lewicki 2005). These differences could also be due to the nature of dielectric constant and loss factor values although this study did not focus on these determinants. Significant differences in the structural properties (such as bulk density and porosity) consequently, affect flow, compression, thermal, and frictional properties.

5.5 Conclusions

This study assessed the effects of condensed distillers solubles (CDS), microwave power, and microwave convection settings on the physico-chemical characteristics of wheat distillers dried grain with solubles (DDGS).

Although factorial interactions had a significant effect on most of the chemical constituents of microwave- and microwave convection-dried samples, CDS level was seen as having a stronger influence on chemical composition. As CDS level in the blend was increased, protein and ash increased while fat and fiber decreased. Fat content was markedly affected by the microwave oven settings.

CDS level was also seen as having a stronger influence on most of the physical properties compared to microwave power or microwave convection setting. Wheat DDGS samples from with higher CDS content were denser, smaller, less uniform in size, redder, less dispersible, lower thermal diffusivity, and produced pellets with higher density and failure stress. These were observed for both sample types. Additionally, higher CDS, microwave-dried samples also had steeper angles of repose and of internal friction, higher thermal conductivity, and were less flowable.

These results contribute toward better understanding of wheat DDGS variability, highlighting the importance of selecting the appropriate condensed distillers solubles: wet distillers grain (CDS: WDG) blending proportion and drying conditions to maximize its nutritive value as an animal feed ingredient and improve the efficiency of related handling and processing operations.

Chapter 6

Physico-chemical characteristics of wheat distillers dried grain with solubles sourced from a Saskatchewan ethanol plant

A similar version of this chapter has been submitted to the Canadian Biosystems Engineering Journal on 07 August 2013:

- Mosqueda, M. R., L.G. Tabil, and K. Muthukumarappan. Physico-chemical characteristics of wheat distillers dried grain with solubles sourced from a Saskatchewan ethanol plant.

Contribution of the PhD Candidate

Literature review, planning and execution of the experiments, data analysis, and manuscript preparation were performed by the PhD candidate. Dallas Nelson and Edward Robertson, undergraduate students of the Department of Chemical and Biological Engineering, assisted in the data collection. Guidance and editorial advice during the entire process were provided by the candidate's research supervisor, Lope G. Tabil. Professor Kasiviswanath Muthukumarappan of South Dakota State University facilitated the candidate's conduct of laboratory work on flow properties determination at the USDA North Central Laboratory. He also provided editorial advice during manuscript preparation. His graduate student, Yijing Wang, trained the candidate on the use of the associated laboratory equipment.

Contribution of the Paper to the Overall Study

This paper addressed one of the main objectives of this PhD research project on the physico-chemical characterization of commercially available wheat distillers dried grain with solubles (DDGS). While the physico-chemical characteristics of corn DDGS are well-studied, there is still very limited information on wheat DDGS characteristics. Since wheat DDGS is primarily utilized as an animal feed ingredient, most of the wheat DDGS investigations in Saskatchewan were, understandably, focused on its nutritional characteristics and their effect on animal growth performance and carcass quality. Baseline information on its physico-chemical characteristics could assist in product quality improvement efforts and in understanding and addressing related handling and storage challenges in wheat DDGS value chains. Aside from researchers, other expected users of this information would include ethanol plant personnel involved in quality assurance, materials handling, storage and transport of wheat DDGS, manufacturers of materials handling equipment, and animal feed processors.

6.1 Abstract

The proximate composition and the effect of moisture content on particle size and particle distribution, bulk and particle densities, color, flow properties, compression characteristics, moisture sorption behavior, and frictional properties of plant-sourced wheat distillers dried grain with solubles (DDGS) were assessed. Proximate composition significantly differed between samples obtained from two production batches. Protein content of wheat DDGS was higher while its fat content was lower compared to published corn DDGS values. Most of the physical properties were significantly affected by moisture content. Under the Carr classification system, plant-sourced wheat DDGS was considered as fairly flowable and floodable and may require

measures to assure flow and prevent flushing. Its compression characteristics and moisture sorption behavior were adequately described by the Kawakita-Ludde and Guggenheim, Anderson and de Boer (GAB) models, respectively.

6.2 Introduction

Wheat distillers dried grain with solubles (DDGS), a co-product of ethanol production, is primarily used as an animal feed ingredient in Canada (FOBI Network 2011). Thus, majority of the published information on the co-product is focused on nutritional aspects and their effect on animal growth performance and carcass quality. Like corn DDGS, wheat DDGS value chains are also confronted with a number of challenges, some of which were also highlighted in a number of animal feed-related studies. Widyaratne and Zijlstra (2007) and Nuez Ortin and Yu (2009), for example, indicated product inconsistency as among the challenges of wheat DDGS utilization. Reduced protein quality, because of adverse processing conditions employed during ethanol and DDGS production (Nyachoti et al. 2005; Widyaratne and Zijlstra 2007; Lan et al. 2008) is also a concern. The energy-intensive drying process during DDGS production is another challenge (Tang and Cenkowski 2001; Tang et al. 2005; Murphy and Power 2008). Handling and storing such a low-density product can also be problematic (Tumuluru et al. 2010). Unlike corn DDGS, however, where considerable work have been done to measure and understand its physical and chemical properties in relation to handling and storage issues (Rausch et al. 2005; Rosentrater 2006; Bhadra et al. 2009a, 2011a, 2012; Ganesan et al. 2008a; 2008b; 2008d; Liu 2008; Kingsly and Ileleji 2009; Clementson et al. 2009; Kingsly et al. 2010; Probst et al. 2013), there is still very limited baseline information on the physico-chemical characteristics of wheat DDGS that would be essential in addressing existing challenges.

The few studies relating to the physical and chemical characteristics of wheat DDGS are limited to two categories: those investigating the effect of drying conditions and condensed distillers solubles on these characteristics and those relating to its densification. The potential of alternative drying technologies, such as the use of superheated steam (Tang et al. 2005) and microwave energy (Mosqueda and Tabil 2011a), had been explored to reduce energy consumption and to minimize the adverse effect of high temperature drying the nutrient composition of DDGS. Laboratory-scale investigations were also conducted to assess the effect of forced-air convection, microwave, microwave convection drying conditions and condensed distillers solubles (CDS) level on protein quality (Mosqueda et al. 2013a), and to assess the effect of drying air temperatures on proximate composition, physical attributes, flow properties, compression characteristics, and frictional properties of wheat DDGS (Mosqueda et al. 2013b). There were also studies that evaluated the effect of superheated steam drying conditions and solubles level on the angle of internal friction and cohesion (Hargreaves et al. 2010) and on moisture diffusivity (Zielinska and Cenkowski 2012) of distillers spent grain. These studies, however, used a mixture of corn and wheat stillage as raw material.

Furthermore, there are also a few studies that assessed the quality characteristics of pelleted wheat DDGS. Densification was seen as a more efficient way of handling this low-density material (Opoku et al. 2009; Tumuluru et al. 2010). Opoku et al. (2009) investigated the possibility of pelleting wheat DDGS and assessed the effect of die diameter and steam conditioning on the durability of wheat DDGS pellets. Tumuluru et al. (2010) also studied the effect of process variables on the characteristics of wheat DDGS pellets produced from a single pelleting machine and a pilot-scale mill. Using wheat DDGS sourced from two Saskatchewan ethanol plants, Saha (2010) compared the effect of feed moisture content, particle size, and

temperature on the physical attributes and durability of steam-conditioned and non-steam conditioned pellets intended as biofuel. She also evaluated some characteristics of bulk DDGS.

Considering this lack of published information on wheat DDGS, this study aimed to provide more baseline information on its physico-chemical characteristics that may be helpful in addressing existing production, handling, storage, and utilization challenges. Specifically, this study assessed the proximate composition of commercial wheat DDGS and examined the effect of moisture content on its particle size, bulk and particle densities, color, flow properties, compression characteristics, moisture sorption behavior, and frictional properties.

6.3 Materials and Methods

Wheat DDGS samples were obtained from Terra Grain Fuels, Inc., an ethanol plant located in Belle Plaine, Saskatchewan, in two production batches, subsequently referred to in this paper as S1 and S2. The samples were placed in tightly sealed bins and stored in a 3°C environment until these were used.

6.3.1 Chemical composition

The proximate composition of wheat DDGS was determined using standard AOAC procedures. Moisture content was determined using the AOAC Official Method 920.36 (AOAC 2003a). Crude protein was estimated using the Kjeldahl method, AOAC Official Method 984.13 (AOAC 2003b). Crude fat was determined using the Goldfish fat extractor (Labconco Corporation, Kansas City, MO), following the AOAC Official Method 920.39 (AOAC 2003d), with anhydrous diethyl ether as extraction solvent. Crude ash was determined using AOAC Official Method 942.05 (AOAC 2003e), while acid detergent fibre and neutral detergent fiber were

estimated through AOAC Official Method 973.18 (AOAC 2003c) and through the method laid out by van Soest et al. (1991), respectively. A lamb starter feed sample, AAFCO 0728 (Association of American Feed Control Officials (AAFCO) Check Sample Program, AAFCO, Champaign, IL), was used as a check sample. The proximate analysis was conducted in duplicate runs.

6.3.2 Physical properties

For each production batch, samples at three moisture levels were generated. To achieve lower moisture levels, wheat DDGS samples were dried at 80°C to the desired moisture content using a thermogravimetric laboratory oven. To adjust moisture to a higher level, wheat DDGS was sprayed with an appropriate amount of water while being thoroughly mixed using a cement mixer, placed into sealed plastic bags, and stored for at least 24 h at room temperature (22-23°C) before use. Moisture levels of S1 samples were 5.3%, 8.4%, and 18.4% (wb), while those of S2 samples were 5.7%, 8.7%, and 13.2% (wb). These levels were used in the determination of basic physical attributes, flow properties, thermal properties, and compression characteristics. Moisture levels of S1 and S2 samples used in determining frictional properties were 6.9 – 14.7% and 5.8 – 14.0% (wb), respectively. Duplicate measurements were made for each property, unless otherwise stated.

6.3.2.1 Particle size and size distribution

Sieve sizes 12, 20, 30, 40, 60, 80, 100, 120, 140, 170, 200 and 230 were used for particle size analysis, following ANSI/ASAE S319.4 (ASABE 2008). The calculated geometric mean diameter was used to represent particle size in this study. Particle size is an important characteristic to evaluate because it can affect nutrient digestibility, feed mixing efficiency, feed

palatability, pellet quality, bulk density, and particle and ingredient segregation during transport and handling (Knott et al. 2004). Clementson et al. (2009) also showed that a DDGS bulk with a large particle size distribution led to particle segregation during handling, and consequently, nutrient segregation. Liu (2008) also reported compositional variation due to size differences of particles making up the DDGS bulk.

6.3.2.2 Bulk and particle density

Bulk density was determined by placing the sample on a funnel and was allowed to freely flow into a 0.5 L steel cup (SWA951, Superior Scale Co. Ltd., Winnipeg, MB). The cup contents were leveled using a steel rod and weighed. Bulk density was obtained by dividing sample mass contained in the cup with the cup volume. Particle density was determined using a gas multipycnometer (QuantaChrome, Boynton Beach, FL). Bulk porosity (ε), expressed as a percentage, was determined as a function of bulk (ρ_b) and particle densities (ρ_p) using Eq. 6.1 below.

$$\varepsilon = 1 - \frac{\rho_b}{\rho_p} \quad (6.1)$$

Assessing bulk density is important because it directly impacts on transport and storage costs. Transporting and storing low bulk density feed ingredients, like wheat DDGS, would be more costly because of the higher spatial requirements.

6.3.2.3 Color

Color of the samples was determined using the HunterLab spectrophotometer (Hunter Associates Laboratory Inc, Reston, VA) and was expressed in terms of the Hunter L, a, and b parameters,

which represent lightness, redness, and yellowness, respectively. Color was measured because it is used as a quick indicator of the protein quality of DDGS. Darker-colored DDGS is associated with heat-damaged proteins, and therefore, reduced protein quality (Fastinger et al. 2006; Batal and Dale 2006).

6.3.2.4 *Flow properties*

Flow characteristics are important for the efficient design of material handling equipment. To assess these characteristics, the Hosokawa Micron Powder Tester PT-R (Hokosawa Micron Corp., Osaka, Japan) was used. This equipment has been used in determining the flow properties of corn DDGS (Ganesan et al. 2008a; Bhadra et al. 2009; 2012) and other materials (Hokosawa Micron Corp. n.d.) with average particle sizes larger than wheat DDGS. Flowability and floodability indices were determined based on the methodology developed by Carr (1965). Flowability index was derived using four properties: compressibility, angle of repose, angle of spatula, and uniformity coefficient while the floodability index was determined using flowability index, angle of fall, angle of difference, and dispersibility. Each of these properties was assigned an index value, ranging from 0 to 25, based on the point score classification system developed by Carr (1965). The component index values are summed to comprise flowability and floodability indices, which ranged from 0 (very, very poor flowability; will not flood) to 100 (excellent flowability; very floodable).

Carr (1965) defined the angle of repose as the angle that a pile of material makes with the horizontal, while angle of spatula as the average of the angles formed by the material with horizontal when: (i) a flat blade (spatula) is inserted into, and lifted out of, a pile of the material, and (ii) when that blade containing the material is gently tapped. Compressibility and uniformity

coefficient were calculated from measured parameters. The former is the ratio of the difference between packed and aerated bulk density values to packed bulk density while the latter is ratio of the width of sieve opening that passed 60% of the sample to width of sieve opening that passed 10% of the sample (Carr 1965). Angle of fall was defined as the new angle of repose formed after a pile of material on a flat surface was jarred while the angle of difference is the numerical difference between the angle of repose and the angle of fall (Carr 1965). Dispersibility refers to the percentage of material that had been dispersed or lost during a fall and is determined by dropping a 10 gram sample through a plastic cylinder from a fixed height onto a watch glass and measuring the amount collected on the watch glass (Carr 1965).

6.3.2.5 *Moisture sorption behavior*

Wheat DDGS is subjected to varying environmental conditions during handling and storage and like any hygroscopic material, it can gain from or lose moisture to its surrounding environment. Determining the sorption characteristics of a material is essential to maintain its quality during handling and storage. Sorption characteristics of wheat DDGS at two temperature (3°C, 23°C) and five relative humidity (RH) levels (50, 60, 70, 80 and 90%) were determined using the static gravimetric method. Airtight cylindrical chambers (Fig. 6.1a), as described by Dadgar (2005), were used to hold the samples during the duration of the study. Each chamber contained 4 petri dishes, each holding about 0.75 g of thinly spread samples (Fig 6.1b and 6.1c). Saturated salt solutions (magnesium nitrate, potassium iodide, sodium chloride, potassium bromide, barium chloride, and potassium nitrate) were used to maintain the RH level inside these chambers. Thymol crystals (Ganesan et al. 2008b) were placed inside these chambers to prevent microbial growth during the experiment. Sample mass, RH, and temperature within these test chambers were regularly monitored until the sample mass reached equilibrium.

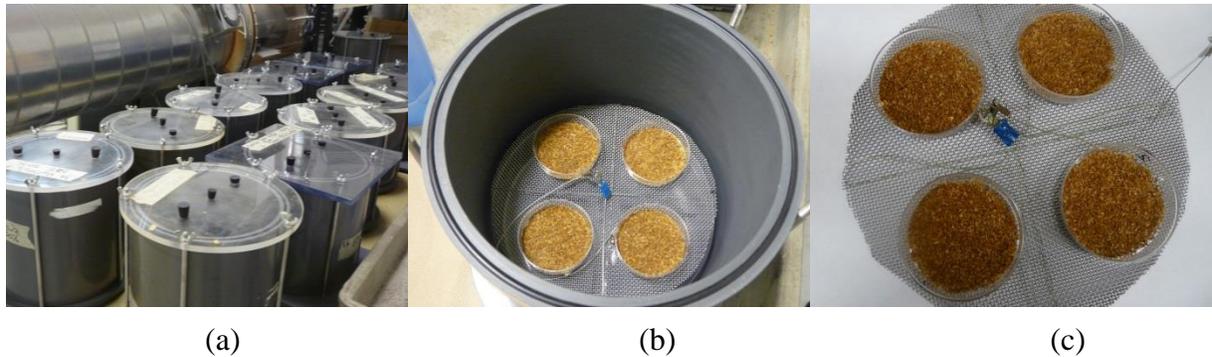


Fig. 6.1 Experimental set up for determining moisture sorption characteristics, showing the chambers (a), the sample inside the chamber (b), and the sample tray (c).

Experimental data were fitted to several moisture sorption models laid out in Table 6.1: Henderson, Chung-Pfost, Modified Halsey, Modified Oswin, Modified Smith, and Guggenheim-Anderson-deBoer (GAB) models. The first five models are empirical and predict the equilibrium moisture content (M_e) as a function of temperature and relative humidity. The GAB equation, on the other hand, is a three parameter, semi-theoretical model that does not incorporate the effect of temperature in predicting M_e (Jayas and Cenkowski 2006). The Henderson and Chung-Pfost models have been used to predict the M_e of various grains and seeds (Stroshine 1998) and selected starches (Boki and Ono 1991) while the modified Oswin model adequately described the moisture sorption behavior of wheat (Sun and Woods 1994) and chickpea flour (Durakova et al. 2005). The modified Halsey model described the moisture sorption isotherm of rapeseed and was used as basis in developing the Ganesan-Muthu-Rosentrater (GMR) model for corn DDGS (Ganesan et al. 2008b). The most suitable model was chosen using F-value, standard error of the estimate (SEE, Eq. 6.2) and mean relative percentage deviation (MRE, Eq. 6.3), F-value, and the coefficient of determination (R^2), as evaluation criteria (Ganesan et al. 2008b; Kingsly and Ileleji 2009). Residual plots were also assessed.

$$SEE = \sqrt{\frac{\sum(Y_o - Y_p)^2}{df}} \quad (6.2)$$

$$MRE = \frac{100}{N} \sum \frac{|Y_o - Y_p|}{Y_o} \quad (6.3)$$

In Eqs. 6.2 and 6.3, Y_o is observed value, Y_p is the predicted value, df is degrees of freedom, while N is the number of data points.

Table 6.1. Moisture sorption models* used in fitting data derived from wheat distillers dried grain with solubles (DDGS) samples.

Model name	Equation
Henderson	$M_e = \left[\frac{\ln(1-RH)}{-AT} \right]^{1/B}$
Chung-Pfost	$M_e = -\frac{1}{B} \ln \left[\frac{\ln(RH)T}{-A} \right]$
Modified Halsey	$M_e = \left[\frac{-\exp(A+BT)}{\ln(RH)} \right]^{1/C}$
Modified Oswin	$M_e = (A+BT) \left[\frac{RH}{1-RH} \right]^{1/C}$
Modified Smith	$M_e = (A+BT) - [(C+DT)\ln(1-RH)]$
Guggenheim, Anderson and de Boer (GAB)	$M_e = \frac{ABC(RH)}{(1-B(RH))(1-B(RH)+BC(RH))}$

Sources: ASABE (2009) and Ganesan et al. (2008c); M_e is equilibrium moisture content, dry basis; T is temperature ($^{\circ}C$); RH is relative humidity; A , B , C , and D are the model constants.

6.3.2.6 *Compression characteristics*

Densification of wheat DDGS lowers transport and storage costs because of better spatial utilization of storage and transportation capacities, eliminates particle/nutrient segregation since pellet composition remains fixed during materials handling, and provides better flow properties (Thomas and van der Poel 1996). Understanding wheat DDGS' compression characteristics would be essential in improving the efficiency of the densification process.

The compression characteristics of wheat DDGS samples were determined using a single pelleter (6.35 mm diameter and 135.34 mm length), heated at 90°C to simulate the pelleting conditions in commercial mills (Tumuluru et al. 2010). A plunger of the same diameter, attached to the Instron Model 1011 testing machine (Instron Corp., Canton, MA), was used to compress 1 g samples at 4000N. Crosshead speed was set at 50 mm·min⁻¹ (Tumuluru et al. 2010). Ten pellets were produced at each moisture level. Only S2, at varying moisture level, was used as feed material in pelleting.

Experimental data were fitted to Kawakita-Ludde model (Eq. 6.4), which was found to adequately describe the compression characteristics of laboratory-prepared wheat DDGS with varying CDS level (Mosqueda et al. 2013b).

$$\frac{P}{C} = \frac{1}{d \cdot f} + \frac{P}{d} \quad (6.4)$$

In Eq. 6.4, P is the applied compressive pressure; C is the degree of volume reduction, $C = \frac{V_0 - V}{V_0}$;

V_0 is the volume of compact at zero pressure; V is volume of compact at pressure P (MPa); d and f are the model parameters. Model parameter d is equal to the initial porosity of the sample

while the reciprocal of the model parameter f (f^{-1}) is related to failure stress (MPa) of the pelleted sample (Mani et al. 2004).

Pellet density was also determined by measuring the mass, length, and diameter of the resulting pellet immediately after it was extruded from the die. Specific energy consumption during compression and extrusion was estimated by calculating the area under the corresponding force-displacement curve using the trapezoidal rule and dividing it by the pellet mass.

6.3.2.7 Frictional properties

Handling and storage of wheat DDGS would frequently involve movement of the material across a solid surface. Knowing its frictional characteristics would be essential in the design of more efficient handling equipment and devices. A tilting table apparatus was used to determine the coefficient of static friction of wheat DDGS on stainless steel, concrete, and wood. The angle at which the material begins to slide was recorded. The coefficient of wall friction and adhesion was also determined using the Wykeham Farrance shear box apparatus (Wykeham Farrance International Ltd., Slough, U.K.), shown in Fig. 6.2 and described in detail by Mani et al. (2004) and Emami and Tabil (2008). Five normal loads (100, 200, 300, 400 and 500 N) and three surfaces (galvanized steel, Teflon, and polypropylene) were used during the test. The top box, which contained the sample, was held stationary, while the lower box, which contained the test surface, moved at a constant speed of $0.4 \text{ mm}\cdot\text{min}^{-1}$. Values of the friction coefficient and adhesion were obtained through regression analysis of experimental data using Eq. 6.5 (Mani et al. 2004; Emami and Tabil 2008), where τ is the shear stress (kPa), σ is the normal stress (kPa), θ is the angle of wall friction, $\tan \theta$ is the coefficient of wall friction, and A is adhesion (kPa).

$$\tau = \tan\theta\sigma + A \tag{6.5}$$



Fig. 6.2 Wykeham Farrance shear box apparatus.

6.3.3 Statistical Analysis

The one-way ANOVA procedure of SPSS 14.0 for Windows (SPSS Inc., Chicago, IL) was used to test the effect of moisture content on the physical properties of wheat DDGS as well as the effect of production batches on its physico-chemical characteristics at the same moisture level. Its general linear model univariate procedure was also used to test the effect of moisture content and type of surfaces on the static friction coefficient. Tukey's test was used to further evaluate statistically significant main effects and interactions. The regression procedure was used in fitting experimental data to selected models. All tests were conducted at the 0.05 significance level.

6.4 Results and Discussion

6.4.1 Chemical composition

Table 6.2 shows the proximate composition results (dry matter basis) for the wheat DDGS samples (column 2) and compares these with some of the published results of both wheat and corn DDGS and grains (columns 3-6). For both samples, mean crude protein content varied

Table 6.2 Chemical composition of wheat distillers dried grain with solubles obtained from two production batches (S1 and S2) of a Saskatchewan fuel ethanol plant, in comparison with published results on wheat and corn grains and DDGS. Numbers in parenthesis represent standard deviation.

Chemical Constituent		Wheat DDGS		Corn DDGS ³	Whole grain	
		This study ¹	Other studies		Wheat	Corn ³
Moisture % wet basis	S1	12.92	7.5 ²	8.56	10.6 ²	11.23
	S2	13.32	6.24 ³ 7.4-8.73 ⁴		10.48 ³	
Crude protein, % dry matter	S1	45.12 (0.52) ^a	44.5 ²	32.01	19.8 ²	10.13
	S2	38.81 (0.14) ^b	39.32 ³ 37.64-40.33 ⁴		14.28 ³	
Crude fat, % dry matter	S1	3.37 (0.05) ^a	2.9 ²	16.53	1.8 ²	4.59
	S2	4.89 (0.18) ^b	4.98 ³		1.91 ³	
Ash, % dry matter	S1	6.41 (0.02) ^a	5.3 ²	4.32	2.1 ²	1.73
	S2	7.10 (0.01) ^b	5.12 ³		2.12 ³	
Neutral detergent fiber, % dry matter	S1	42.74 (0.88) ^a	30.3 ²	49.46	9.4 ²	14.47
	S2	46.55 (0.80) ^b	48.07 ³ 51.04-51.79 ⁴		17.22 ³	
Acid detergent fiber, % dry matter	S1	21.10 (0.11) ^a	21.1 ²	14.68	2.7 ²	3.66
	S2	17.45 (0.27) ^b	10.99 ³ 15.78-22.93 ⁴		3.68 ³	
Acid detergent insoluble crude protein, % crude protein, dry basis	S1	11.80 (1.41) ^a	4.85 ³	6.44	0.00 ²	0.08
	S2	18.37 (1.71) ^b	9.07-23.98 ⁴			

¹Values followed by the same letter under each chemical constituent are not significantly different.

²Widyaratne and Zijlstra (2007). Moisture content was converted to wet basis.

³Nuez Ortin and Yu (2009). Moisture content (M, % wb) was obtained by the equation: M = 100 - % dry matter.

⁴Tumuluru et al (2010). Moisture content (M, % wb) was obtained by the equation: M = 100 - % dry matter.

from 38.8% to 45.1%, crude fat ranged from 3.4% to 4.9%, ash from 6.4% to 7.1%, neutral detergent fiber varied from 42.7% to 46.6%, and acid detergent fiber from 17.4 – 21.1%. These results were close to the values reported by Widyaratne and Zijlstra (2007), Nuez Ortin and Yu (2009), and Tumuluru et al. (2010) in Table 6.2 (columns 3-6). In comparison to the reported values of corn DDGS in Table 6.2, the wheat DDGS samples had higher protein but lower fat content. This was also reported by Nuez Ortin and Yu (2009). Compositional differences

between wheat and corn DDGS are attributed primarily to differences in the nutrient content between the two grains.

Proximate composition between the two wheat DDGS samples was significantly different. Sample 2 (S2) had significantly lower protein but higher fiber, ash, and fat contents compared to sample 1 (S1). These differences are primarily attributed to inherent nutrient variation of the starting wheat grains and to variation in the CDS and wet distillers grain (WDG) blending proportions used. Differences in the varieties (soft vs. hard wheat) used, for example, could result to differing DDGS protein contents. It has also been demonstrated that variations in the CDS:WDG blending proportions result to wheat DDGS compositional differences (Mosqueda et al. 2013b). The wheat-based CDS had higher crude protein and ash content but lower fat and fiber content than the WDG component. Thus, as CDS level in the blend increased, protein and ash content increased while fat and fiber content decreased. Processing conditions could also affect its chemical composition. Use of higher drying temperatures, for example, had affected the fat and ADF content of laboratory-prepared wheat DDGS (Mosqueda et al. 2013b).

Table 6.2 also presents the acid detergent insoluble crude protein (ADICP) content of the samples, which ranged from 11.8 to 18.4% of the total crude protein (dry matter basis). The values were lower than those reported by Nuez Ortin and Yu (2009) but within the range of values presented by Tumuluru et al. (2010). ADICP content was significantly affected by sample batch, with S2 showing significantly higher values than S1. High ADICP content, which indicates high incidence of heat-damaged proteins (Cromwell et al. 1993) would adversely affect the nutritive value of wheat DDGS as an animal feed ingredient. High ADICP content could be attributed to adverse processing conditions employed during ethanol and DDGS production. Use

of elevated drying air temperatures during these processes, for example, would lead to increased production of insoluble, heat-damaged protein aggregates via the Maillard reaction.

These plant-sourced samples had higher protein and ash content but lower fat and NDF content than the laboratory-prepared, forced air convection-dried wheat DDGS with 45% CDS. For the latter, Mosqueda et al. (2013b) reported 33.4 – 34.3% protein, 4.9% ash, 5.2-5.7% fat, and 49.9-54.2% NDF. These suggest that the plant-sourced wheat DDGS samples may have been produced at CDS levels higher than 45% of the total blended mass (wet basis).

6.4.2 Physical properties

6.4.2.1 Particle size and size distribution

Table 6.3 shows the average particle size, expressed in terms of the geometric mean diameter, while Fig. 6.3 presents the average particle size distribution of wheat DDGS samples. Particle size varied from 0.43 mm to 0.59 mm (Table 6.3). Moisture content significantly affected the particle size of S2 while it did not affect S1 particle sizes. Samples with statistically similar moisture contents (8.4% for S1 and 8.7% for S2, wet basis) did not show significant difference in their particle sizes.

Although there was no statistical difference in the average particle size between S1 and S2 at 8% moisture, their particle size distribution curves (Fig. 6.3) at various moisture levels show that S2, in general, had numerically smaller particle sizes compared to S1. This is illustrated in sieve sizes starting at 0.85 mm until 0.18 mm, where S2 had higher percentages of particles passing than S1. This could be attributed to differences in the CDS:WDG blending proportions used by the source ethanol plant. Mosqueda et al. (2013b) had reported decreases in particle size as CDS

Table 6.3 Physical attributes¹ of wheat distillers dried grain with solubles at varying moisture levels. Samples were obtained from a Saskatchewan ethanol plant in two production batches (S1, S2). Values in parentheses represent standard deviation.

Sample batch	Moisture content, % wet basis	Particle size, mm	Bulk density, kgm ⁻³	Particle density, kgm ⁻³	Porosity, %	Color parameters ²		
						L	a	b
S1	5.3 (0.1)	0.58 (0.03) ^a	339.7 (2.0) ^a	1294.0 (6.3) ^a	73.7	32.86 (0.60) ^a	8.86 (0.19) ^a	13.58 (0.06) ^a
	8.4 (0.3)	0.57 (0.04) ^{a,b}	359.0 (3.8) ^b	1296.3 (2.1) ^{a,b}	72.3	33.68 (0.22) ^a	8.64 (0.05) ^{a,b}	13.32 (0.06) ^a
	18.4 (0.1)	0.59 (0.04) ^a	380.4 (2.1) ^c	1274.9 (0.0) ^{b,c}	70.2	32.46 (0.93) ^a	9.03 (0.23) ^a	13.44 (0.11) ^a
S2	5.7 (0.1)	0.48 (0.03) ^{x,z}	424.1 (0.3) ^x	1331.3 (0.1) ^x	68.1	35.26 (0.59) ^x	10.17 (0.34) ^x	15.34 (0.53) ^x
	8.7 (0.3)	0.52 (0.00) ^{x,y,b}	429.6 (3.1) ^{x,y}	1326.6 (1.3) ^{x,y}	67.6	37.67 (0.59) ^y	8.89 (0.16) ^{y,b}	14.98 (0.30) ^{x,y}
	13.2 (0.0)	0.43 (0.02) ^z	437.0 (3.4) ^{y,z}	1320.5 (2.9) ^{y,z}	66.9	35.40 (0.02) ^{x,z}	7.68 (0.04) ^z	12.57 (0.01) ^z

¹Tukey's test at 5% significance level for S1 at varying moisture content (a, b, c) and for S2 at varying moisture content (x,y,z). Comparison between S1 and S2 properties was only made at the 8% moisture level. Values followed by same letters are not significantly different.

²The Hunter L,a, and b parameters represent lightness, redness, and yellowness, respectively.

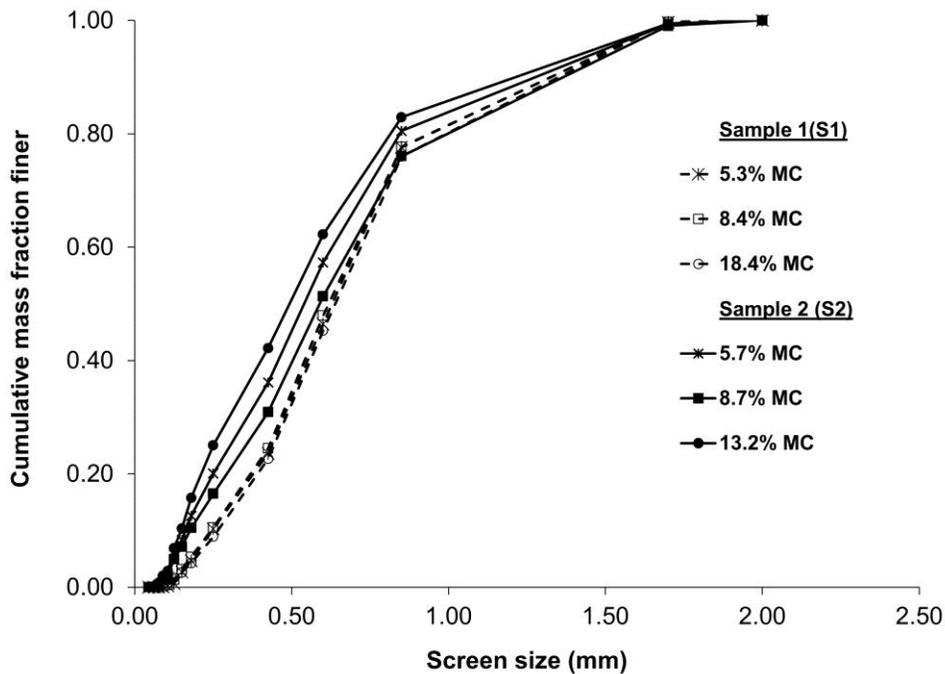


Fig. 6.3 Average particle size distribution of plant-sourced wheat distillers dried grain with solubles. Samples were obtained from two production batches (S1, S2) of an ethanol plant.

level was increased. Increased CDS in the blend resulted to higher percentage of finer, endosperm- and germ-derived solids and decreased amount of fibrous, bran-related particles.

Results of the particle analyses further supported the previously presented observation that the plant-sourced samples may have been produced at CDS levels higher than 45% of the total blended mass (wet basis). The particle sizes of the plant-sourced samples were smaller (thus, CDS level was higher) compared to those reported for laboratory-prepared samples with 15-45% CDS (Mosqueda et al. 2013b). The particle sizes obtained in this study were also smaller than the published values for plant-sourced corn DDGS. Bhadra et al. (2009a) reported corn-based DDGS particle sizes mostly ranging above 0.5 mm, although there was one batch in one plant that had a particle size of 0.21 mm. Liu (2008) also reported DDGS particle sizes ranging from 0.43 mm to 0.95 mm in 11 dry grind ethanol plants in the U.S. Plant-scale studies on corn DDGS (Kingsly et

al. 2010; Clementson and Ileleji 2012) indicated that particle size increased with CDS level due to agglomeration during rotary drum drying. In this study, the wheat DDGS samples were obtained from a fuel ethanol plant that employed a ring dryer for DDGS production. Under this drying system, the WDGS is dispersed and conveyed through the dryer in a hot air stream at high speed. Thus, incidence of particle agglomeration was observed in the wheat DDGS samples used in this study. Different screen sizes employed during the grinding of grains could also contribute to the size differences observed between corn and wheat DDGS.

6.4.2.2 *Bulk and particle density*

Bulk density of S1 varied from $340 \text{ kg}\cdot\text{m}^{-3}$ to $380 \text{ kg}\cdot\text{m}^{-3}$ while that of S2 were from $424 \text{ kg}\cdot\text{m}^{-3}$ to $437 \text{ kg}\cdot\text{m}^{-3}$ (Table 6.3). In both samples, bulk density was significantly affected by moisture content. In S1, those with higher moisture content had significantly higher bulk density than those with lower moisture content. In S2, the 13.2% moisture sample also had significantly higher bulk density than the one with 5.7% moisture. These positive linear relationships between moisture content and bulk density were statistically significant in both samples. For samples with similar moisture contents (S1 has 8.4% moisture while S2 has 8.7% moisture), S2 was significantly denser compared to S1.

Particle density values ranged from $1275 \text{ kg}\cdot\text{m}^{-3}$ to $1331 \text{ kg}\cdot\text{m}^{-3}$ (Table 6.3) and were significantly affected by moisture content. Samples with higher moisture had significantly lower particle density than those with lower moisture. This negative linear relationship was significant in both samples. For samples with statistically similar moisture contents (8.4% for S1 and 8.7% for S2, wet basis), sample 2 also had significantly higher particle density than sample 1.

Differences in bulk and particle densities between the samples of the same moisture level could be attributed to differences in chemical composition and in particle sizes. Increased CDS level in the DDGS blend increased the presence of finer but heavier solids and decreased the amount of the larger but lighter, fibrous particles. These finer particles could easily move through and fill inter-particle spaces within the bulk mass, resulting to a heavier and less porous bulk.

6.4.2.3 Color

Table 6.3 also shows the color parameters L, a, and b, which represent lightness, redness, and yellowness, respectively, of both samples. The lightness parameter varied from 32.4 to 37.7, redness from 7.7 to 10.2 and yellowness from 12.6 to 15.3. All three color parameters of S2 were significantly affected by moisture content. Samples with 5.7% moisture were redder and yellower than those with 13.2% moisture. The 8.7% moisture was significantly lighter than 13.2% moisture sample. In samples with statistically similar moisture content (S1 and S2 at 8% moisture level, wet basis), S2 was significantly lighter yet yellower than S1.

Observed differences in the color parameters of the two wheat DDGS samples could be attributed to variations in the CDS:WDG blending proportion and to the extent of development of Maillard reactions that may have occurred during various stages of ethanol and DDGS production. It was previously reported that as CDS level in the blend was increased, freeze-dried, laboratory-prepared wheat DDGS samples became significantly darker and redder (Mosqueda et al. 2013b). CDS obtained from the ethanol plant was darker and redder ($L = 45.7$, $a = 5.3$, $b = 16.9$) than the WDG fraction ($L = 52$, $a = 3.2$, $b = 15.6$).

Wheat DDGS samples were slightly darker ($L = 32.4-37.7$) but less yellow ($b = 12.6-15.3$), compared to commercial corn DDGS ($L = 41.8-48.8$; $b = 19.0-23.0$), based on the results

reported by Rosentrater (2006). Redness of the wheat DDGS samples ($a = 7.7-10.2$) was close to the range for commercial corn DDGS (8.3 – 9.7) (Rosentrater 2006).

Color darkening is one of the consequences of Maillard reaction, which involves the binding of amino groups with the carbonyl compounds of reducing sugars (Owusu-Apenten 2004, Nyachoti et al 2005; Widyaratne and Zijlstra 2007). During the initial stages of Maillard reaction, colorless products are formed while highly colored polymers are formed during the final stages (Hodge 1953; Yaylayan and Roberts 2001). The rate of Maillard reaction is affected by such factors as temperature, time, water activity, and chemical composition (Owusu-Apenten 2004; Ames 1990). Higher drying temperature and drying time, for example, would increase the rate of Maillard reaction. During convective drying, the surface of the material usually dries out first, producing a crust with a low water activity and favoring Maillard reaction. Increased protein and sugar content, as a result of increasing CDS level in the blend, coupled with use of elevated drying temperatures would increase the rate of Maillard reaction and intensify color darkening in wheat DDGS.

6.4.2.4 *Flow properties*

Tables 6.4 and 6.5 show the flowability and floodability indices of wheat DDGS, respectively, including the component properties that were used in the estimation of these indices.

The flowability index of plant-sourced wheat DDGS ranged from 70.5 to 73.8 (Table 6.4), classifying both samples as fairly flowable that may sometimes need vibration to assure flow (Carr 1965). The results were comparable to the flowability index of laboratory-prepared wheat DDGS samples (70.5-77.5) (Mosqueda et al. 2013b) but were slightly lower than those reported for corn DDGS (79.3-82.4) (Bhadra et al. 2009a).

Table 6.4 Flowability index¹ of wheat distillers dried grain with solubles at varying moisture levels (% , wet basis). Samples were obtained from a Saskatchewan ethanol plant in two production batches (S1 and S2). Values in parentheses represent standard deviation (N = 2).

Sample batch	Moisture content, % wet basis	Compressibility, %	Angle of repose, °	Ave. angle of spatula, °	Uniformity coefficient	Flowability ²	
						Index	Degree
S1	5.3 (0.1)	23.9 (0.1) ^a	44.7 (1.0) ^a	47.7 (0.2) ^a	2.9 (0.0) ^a	70.5 (0.7) ^a	Fair, aid not needed (vibrate if necessary)
	8.4 (0.3)	19.1 (0.8) ^{b,c}	45.7 (0.2) ^a	52.5 (4.1) ^{a,b}	3.0 (0.1) ^a	71.3 (0.4) ^{a,y}	
	18.4 (0.1)	16.7 (1.1) ^c	43.4 (0.8) ^a	49.7 (2.1) ^a	2.9 (0.1) ^a	73.8 (1.1) ^c	
S2	5.7 (0.1)	22.4 (0.4) ^x	44.5 (1.2) ^x	52.2 (0.1) ^{x,z}	4.0 (0.1) ^x	70.5 (0.7) ^x	Fair, aid not needed (vibrate if necessary)
	8.7 (0.3)	19.6 (0.5) ^{y,b}	44.0 (1.4) ^{x,a}	55.6 (0.1) ^{x,y,b}	3.8 (0.0) ^y	72.3 (1.1) ^{x,y}	
	13.2 (0.0)	19.7 (1.2) ^y	45.1 (0.6) ^x	49.2 (2.0) ^z	4.7 (0.4) ^z	71.3 (0.4) ^x	

¹Tukey's test at 5% significance level for S1 at varying moisture content (a, b, c) and for S2 at varying moisture content (x,y,z). Comparison between S1 and S2 properties was only made at the 8% moisture level. Values followed by same letters are not significantly different. ²Based on Carr classification system (Carr, 1965).

Table 6.5 Floodability index¹ of wheat distillers dried grain with solubles at varying moisture levels (% , wet basis). Samples were obtained from a Saskatchewan ethanol plant in two production batches (S1 and S2). Values in parentheses represent standard deviation (N = 2).

Sample batch	Moisture content, % wet basis	Flowability Index	Angle of fall, °	Angle of difference, °	Dispersibility, %	Floodability ²	
						Index	Degree
S1	5.3 (0.1)	70.5 (0.7) ^a	36.5 (0.1) ^a	8.2 (0.8) ^a	15.7 (0.9) ^a	61.8 (1.1) ^a	Floodable, rotary seal will be necessary
	8.4 (0.3)	71.3 (0.4) ^{a,b}	36.7 (0.8) ^{a,b}	9.0 (0.6) ^{a,b}	12.2 (1.1) ^{a,b,y}	62.5 (0.0) ^{a,b}	
	18.4 (0.1)	73.8 (1.1) ^c	34.6 (0.1) ^c	8.8 (0.8) ^a	11.5 (0.8) ^{c,b}	61.8 (1.1) ^a	
S2	5.7 (0.1)	70.5 (0.7) ^x	36.0 (0.8) ^x	8.5 (2.0) ^x	14.3 (1.2) ^x	61.1 (2.7) ^x	Floodable, rotary seal will be necessary
	8.7 (0.3)	72.3 (1.1) ^{x,b}	36.4 (1.0) ^{x,b}	7.6 (0.4) ^{x,b}	12.9 (1.0) ^{x,y}	60.1 (1.2) ^{x,b}	
	13.2 (0.0)	71.3 (0.4) ^x	34.4 (1.1) ^x	10.7 (0.6) ^x	12.9 (0.4) ^x	64.0 (1.4) ^x	

¹Tukey's test at 5% significance level for S1 at varying moisture content (a, b, c) and for S2 at varying moisture content (x,y,z). Comparison between S1 and S2 properties was only made at the 8% moisture level. Values followed by same letters are not significantly different. ²Based on Carr classification system (Carr, 1965).

The flowability index is the composite of the index values of compressibility, angle of repose, average angle of spatula, and uniformity coefficient. Compressibility values of both samples (16.7 - 23.9%) fell under the passable to fair flowability categories (Carr 1965). These were significantly affected by moisture content, with the lower moisture samples being more compressible. Angle of repose ranged from 43.4° to 45.7° while the average angle of spatula varied from 47.7° to 55.6° (Table 6.4). These values indicate that both samples have passable flowability with a tendency to hang up (Carr 1965). Only the average angle of spatula of S2 was significantly affected by moisture content. In terms of uniformity coefficient, the values ranged from 2.9 to 4.7 (Table 6.4) and both samples were classified as materials having excellent flowability (Carr 1965). Uniformity coefficients of both samples were not significantly affected by moisture content. Comparison between samples with statistically similar moisture contents (S1 and S2 at 8% moisture levels) showed that S2 was significantly more size-differentiated than S1. This could be attributed to differences in CDS:WDG blending proportions. Samples with higher CDS levels tended to be finer than those with lower CDS.

The floodability index, on the other hand, varied from 60.1 to 64.0 (Table 6.5), classifying both S1 and S2 as floodable and would require the use of appropriate measures, such as rotary seals, to prevent flushing (Carr 1965). These results were close to the values reported for the laboratory-produced samples in Chapters 4 (Table 4.7) and 5 (Table 5.7), particularly those with 45% CDS content. Floodability index values of both wheat DDGS samples were within the range reported for commercial corn DDGS. Bhadra et al. (2009b) reported a wider range of floodability index values for corn DDGS falling under the “inclined to flood” to “floodable” categories.

Contributing properties to the floodability index include flowability index, angle of fall, angle of difference, and dispersibility. The angle of fall (34.4 - 36.7°) values classified both samples as floodable. Moisture content significantly affected the angle of fall values of S1, with low moisture samples (those 5.3% and 8.4% moisture content) having significantly higher angles of fall than those with 18.4% moisture. In terms of the angle of difference, the values of both samples (7.6 - 10.7°) fell under the “could flood” to “inclined to flood” categories (Carr 1965). These were not significantly affected by moisture content. Lastly, both wheat DDGS samples had dispersibility values that ranged from 11.5% to 15.7% (Table 6.5), classifying them as materials that are inclined to flood (Carr 1965). In S1, dispersibility was significantly affected by moisture content, with the 5.7% moisture sample being more dispersible than the sample with 18.4% moisture. The effect of moisture content was not significant in S2. Samples with statistically similar moisture contents (8.4% sample 1, 8.7% sample 2) did not show significant difference in their angle of fall, angle of difference and dispersibility values.

6.4.2.5 *Thermal properties*

Table 6.6 shows that the wheat DDGS samples had low thermal conductivities (0.04 – 0.05 W·m⁻¹°C⁻¹) at 23°C. These values were not significantly affected by moisture content. Samples with statistical similar moisture contents (S1 and S2 at 8% moisture level) did not also differ in their thermal conductivities. These values were about the same compared to those reported for the laboratory-prepared wheat DDGS with 15-45% CDS (0.05 Wm⁻¹°C⁻¹) but were slightly lower than the values reported by Rosentrater (2006) for commercial corn DDGS (0.06 – 0.08 W·m⁻¹°C⁻¹).

Thermal diffusivity values varied from $1.40 \times 10^{-7} \text{ m}^2\text{s}^{-1}$ to $1.60 \times 10^{-7} \text{ m}^2\text{s}^{-1}$ (Table 6.6). The thermal diffusivity of S1 was significantly affected by moisture content while that of S2 was not. S1 sample with 8.4% moisture had significantly higher thermal diffusivity than that with 18.4% moisture. In samples with statistically similar moisture contents (S1 and S2 at 8% moisture level), S1 had significantly higher thermal diffusivity than S2. Differences in thermal diffusivity could be attributed to differences in density and porosity, which, in turn, are caused by differences in CDS:WDG blending proportions. Samples with lower CDS had significantly higher thermal diffusivity than those with higher CDS (Mosqueda et al. 2013b). Similar to lower CDS samples, those with lower moisture content are also more porous compared to those with higher moisture. Since air has a much higher thermal diffusivity compared to water (Kostaropoulos and Saravacos 1997), materials having the same chemical composition but are more porous would tend to have higher thermal diffusivity than the less porous ones.

Table 6.6 Thermal properties¹ of wheat distillers dried grain with solubles at 23°C and at the specified bulk density and moisture levels. Samples were obtained from a Saskatchewan ethanol plant in two production batches (S1 and S2). Values in parentheses represent standard deviation.

Sample batch	Moisture, % wet basis	Bulk density, kgm^{-3}	Thermal conductivity, $\text{Wm}^{-1}\text{°C}^{-1}$	Thermal diffusivity, $\times 10^{-7} \text{ m}^2\text{s}^{-1}$
S1	5.3 (0.1)	339.7 (2.0)	0.05 (0.00) ^a	1.57 (0.06) ^{a,c}
	8.4 (0.3)	359.0 (3.8)	0.04 (0.01) ^{a,b}	1.63 (0.06) ^a
	18.4 (0.1)	380.4 (2.1)	0.05 (0.00) ^a	1.50 (0.00) ^c
S2	5.7 (0.1)	424.1 (0.3)	0.05 (0.00) ^x	1.57 (0.06) ^x
	8.7 (0.3)	429.6 (3.1)	0.05 (0.00) ^{x,b}	1.43 (0.06) ^x
	13.2 (0.0)	437.0 (3.4)	0.05 (0.01) ^x	1.40 (0.10) ^x

¹Tukey's test at 5% significance level for S1 at varying moisture content (a, b, c) and for S2 at varying moisture content (x,y,z). Comparison between S1 and S2 properties was only made at the 8% moisture level. Values followed by same letters are not significantly different.

Thermal diffusivity of these plant-sourced samples were similar to those reported for the laboratory-prepared wheat DDGS samples ($1.35 \times 10^{-7} \text{ m}^2\text{s}^{-1}$ to $1.65 \times 10^{-7} \text{ m}^2\text{s}^{-1}$), with higher CDS samples showing lower thermal diffusivity values (Mosqueda et al. 2013b). Further, thermal diffusivity of wheat DDGS samples were also close to those reported by Rosentrater (2006) for commercial corn DDGS. Thermal diffusivity of corn DDGS ranged from $1.30 \times 10^{-7} \text{ m}^2\text{s}^{-1}$ to $1.50 \times 10^{-7} \text{ m}^2\text{s}^{-1}$.

6.4.2.6 *Moisture sorption behavior*

Figure 6.4 shows the adsorption isotherms of wheat DDGS samples at 3.6°C (0.2°C) and 22.8°C (0.5°C). The initial moisture contents (dry basis) of these samples were 5% (Fig. 6.2a), 9% (Fig. 6.2b), and 15% (Fig. 6.2c). In all three sample groups, water sorption increased with relative humidity and exhibited a behavior similar to the Type III isotherm (Brunauer et al. 1940). Studies on corn DDGS also reported a Type III moisture adsorption behavior (Ganesan et al. 2008b; Kingsly and Ileleji 2009).

The adsorption isotherms show that at relative humidity levels below 0.75 to 0.80, the sorption behavior of wheat DDGS was almost similar when stored under the 3°C and 22°C environments. Sorption capacity differences only became more noticeable at the higher relative humidity levels, where those stored at 3°C began to show higher equilibrium moisture contents than those stored at 22°C. Sorption capacity decreased, indicating lesser attraction between the water molecules in the surrounding environment and the wheat DDGS under the higher temperature environment.

Figure 6.4 also presents the predicted moisture sorption values using the GAB model. Table 6.6 shows the GAB and modified Halsey model parameters and the corresponding statistical

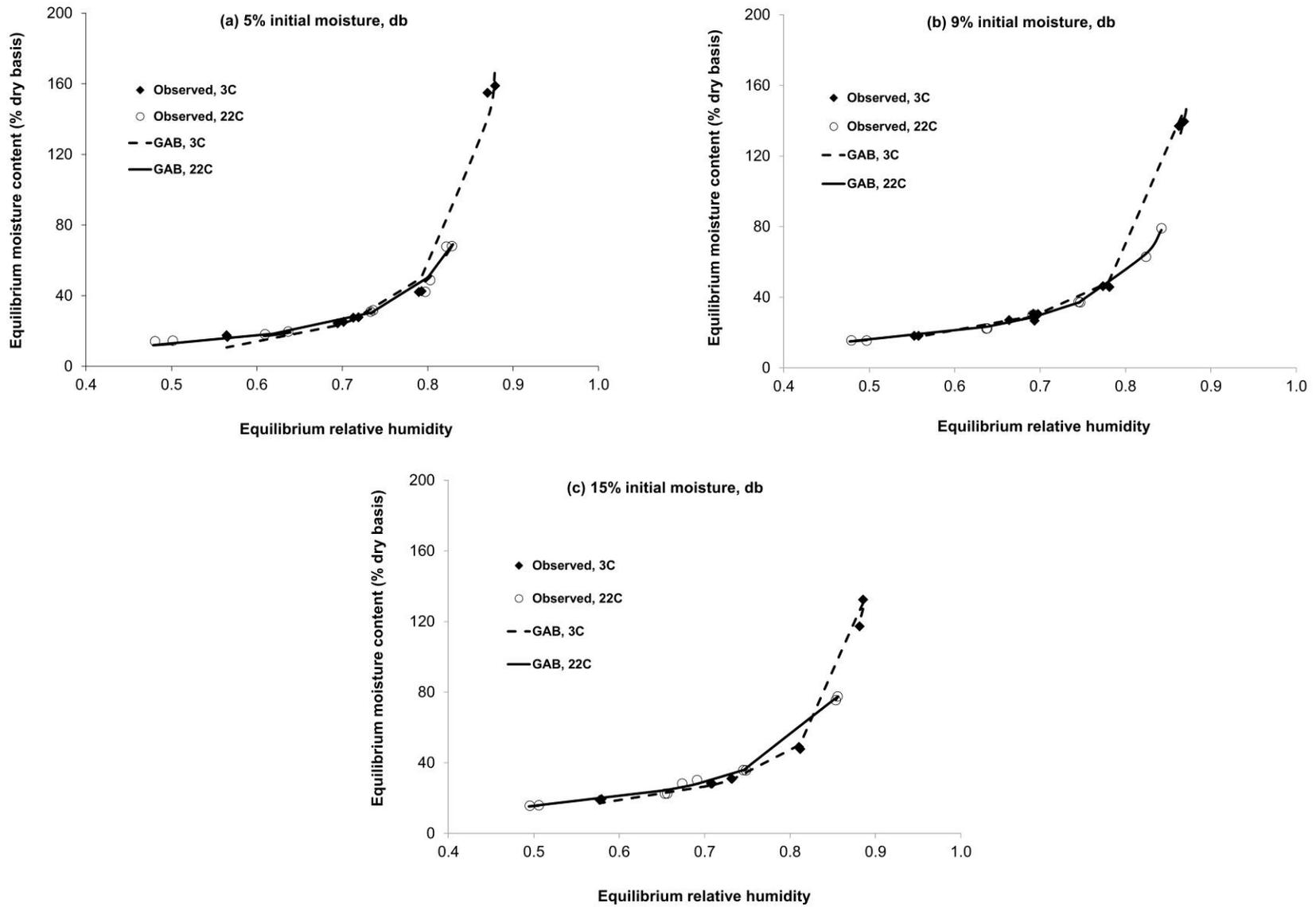


Fig. 6.4 Adsorption isotherms of plant-sourced wheat distillers grain with solubles at 3°C and 22°C. Initial moisture content (dry basis) of the samples were 5% (Fig. 6.4a), 9% (Fig. 6.4b), and 15% (Fig. 6.4c).

Table 6.7 Guggenheim, Anderson and de Boer (GAB) and modified Halsey model and statistical parameters for plant-sourced wheat distillers dried grain with solubles. Values in parentheses represent standard deviation (N=2).

Model	Temp. °C	Initial moisture, % dry basis	Model parameters			Standard error of the mean	Mean relative percentage deviation	F-value	R ²
			A	B	C				
GAB	3	5	10.455	1.075	0.435	8.46	5.52	258.96	0.982
		9	7.485	1.093	6.543	4.32	0.13	827.13	0.993
		15	6.524	1.073	5 x 10 ⁷	2.01	2.46	3316.36	0.998
	22	5	5.646	1.108	1 x 10 ⁷	3.30	2.78	501.54	0.980
		9	7.307	1.077	2 x 10 ⁷	1.17	0.04	3954.85	0.998
		15	7.974	1.049	11.41	1.55	-0.25	2414.03	0.996
Modified Halsey	3	5	-0.235	0.249	0.539	8.50	8.69	256.86	0.981
		9	-0.210	0.332	0.612	5.41	6.52	527.81	0.989
		15	4.396	-1.055	0.533	3.49	1.06	1096.92	0.994
	22	5	-3.286	0.230	0.908	1.55	0.07	2296.83	0.996
		9	-5.550	0.321	0.753	1.99	1.54	1373.68	0.993
		15	7.206	-0.225	0.899	1.71	0.47	1995.84	0.996

parameters. The GAB model yielded the smallest standard error of the mean and mean relative percentage error and the largest F and R² values in almost all cases except for the 22°C-5% moisture treatment combination where the modified Halsey model showed slightly better values. The other four models did not perform as well as the GAB and the modified Halsey model. The Henderson and Chung Pfoest models, for example, showed systematic patterns in their respective residual plots. Modified Oswin and Smith models also showed a few residual plots with systematic patterns. For the modified Oswin and Smith residual plots that showed random patterns, the resulting statistical parameters were less superior compared to those obtained for the GAB and modified Halsey models. The GAB model offered some advantages over the other empirical models. It was developed with a theoretical basis (Quirijns et al. 2005; Andrade et al. 2011), being a “refined extension of the Langmuir and Brunauer-Emmett-Teller (BET) theories” of physical absorption (Quirijns et al. 2005). It also described the sorption behavior of a wide

variety of biological materials with water activities ranging from 0 to 0.90 (Almuhtaseb et al. 2002; Andrade et al. 2011) and its model parameters have physical meaning (Quirijns et al. 2005).

6.4.2.7 Compression characteristics

Table 6.8 shows the pellet density, specific energy consumption during compression and extrusion processes, and the Kawakita Ludde model parameters for wheat DDGS (S2). Pellet density varied from 1185 kgm⁻³ to 1208 kgm⁻³ and was not significantly affected by moisture content. These values were about 89 – 91% of the particle density values presented in Table 6.3. These values were also close to the reported pellet density of laboratory-prepared wheat DDGS with 45% CDS (1187-1210 kgm⁻³) (Mosqueda et al. 2013b).

Table 6.8 Compression characteristics¹ of wheat distillers dried grain with solubles at varying moisture content. Values in parentheses represent standard deviation (N=10).

Property	Initial moisture, % wet basis		
	5.7	8.7	13.2
Pellet density, kgm ⁻³	1198.61 (17.41) ^a	1184.82 (16.04) ^a	1208.17 (22.93) ^a
Specific energy consumption, MJt ⁻¹			
Compression	18.61 (0.58) ^a	13.95 (0.16) ^b	8.64 (1.10) ^c
Extrusion	2.25 (0.18) ^a	0.99 (0.14) ^b	2.54 (0.54) ^a
Kawakita Ludde model parameters ²			
d	0.673 (0.004) ^a	0.666 (0.005) ^b	0.665 (0.006) ^{b,c}
f ⁻¹	2.671 (0.085) ^a	1.939 (0.024) ^b	0.957 (0.221) ^c
R ²	0.999 (0.001)	1.000 (0.000)	1.000 (0.000)
MSE	2.350 (0.153)	0.516 (0.051)	0.275 (0.038)

¹Values followed by the same letter/s within each row are not significantly different at the 0.05 level.

² Model parameters d and f⁻¹ were associated with initial porosity and failure stress, respectively.

Energy consumption during compression and extrusion processes ranged from 8.6 MJ·t⁻¹ to 18.6 MJ·t⁻¹ and from 0.99 MJ·t⁻¹ to 2.54 MJ·t⁻¹, respectively. Energy consumption during compression

accounted about 74.5 – 95.1% of the total energy consumption while pellet extrusion accounted the remaining fraction. Both compression and extrusion energy consumption were significantly affected by variations in the initial moisture content. Samples with lower moisture content (5.7%, 8.7%) consumed significantly higher energy during compression compared to those with higher moisture content (13.2%).

The Kawakita-Ludde model, which represented the compression characteristics of the laboratory-prepared, forced-air convection-dried wheat DDGS, also adequately described the compression behavior of plant-sourced wheat DDGS samples. The values of the model parameter d (Table 6.8), which represents initial porosity of the sample, were almost similar to the bulk porosity values presented in Table 6.3. Samples with 5.7% moisture content had significantly higher initial porosity than those with 8.7% and 13.20% moisture. The model parameter f^{-1} , on the other hand, is associated with failure stress. It varied from 0.96 MPa to 2.67 MPa and was significantly affected by moisture content. Samples with 5.7% and 8.7% moisture, for example, had significantly higher failure stress (higher f^{-1} values) than those with 13.2%. Similarly, those with 8.7% initial moisture also showed significantly higher f^{-1} values than samples with 13.2%. This observation was consistent with the results reported by Tumuluru et al. (2010), where higher pellet durability was achieved at lower feed moisture content and higher die temperature.

6.4.2.8 *Frictional properties*

Table 6.9 shows the coefficient of static friction of both samples on different surfaces using the tilting table. The static friction coefficient of wheat DDGS varied from 0.28 to 0.48 on a stainless steel surface, 0.49 to 0.54 on concrete, and 0.56 to 0.65 on wood. The friction coefficient was

lowest on stainless steel and highest when the sample was on a wooden surface with the wood grain perpendicular to the direction of sliding (0.62-0.65).

Table 6.9 Coefficient of static friction¹ of wheat distillers dried grain with solubles obtained from a south Saskatchewan fuel ethanol plant in two production batches (S1, S2). Values in parentheses represent standard deviation (N=3).

Sample batch	Moisture content, % wet basis	Surface			
		Stainless steel	Concrete	Wood, motion along the grain	Wood, motion across the grain
S1	6.90	0.328 (0.013) ^{Aa}	0.492 (0.011) ^{Ba}	0.589 (0.025) ^{Ca}	0.620 (0.014) ^{Da}
	11.00	0.436 (0.015) ^{Ab}	0.511 (0.014) ^{Ba}	0.593 (0.019) ^{Ca,Cb}	0.633 (0.022) ^{Da}
	14.70	0.491 (0.018) ^{Ac}	0.542 (0.020) ^{Bc}	0.609 (0.015) ^{Cc,Cb}	0.636 (0.023) ^{Da}
S2	5.85	0.285 (0.005) ^{Ax}	0.507 (0.006) ^{Bx}	0.565 (0.007) ^{Cx}	0.628 (0.016) ^{Dx,Dz}
	7.86	0.366 (0.008) ^{Ay}	0.517 (0.004) ^{Bx,By}	0.593 (0.001) ^{Cy}	0.616 (0.028) ^{Dx,Cy}
	13.97	0.480 (0.007) ^{Ay,Az}	0.542 (0.006) ^{By, Bz}	0.610 (0.015) ^{Cz}	0.649 (0.028) ^{Dz,Cz}

¹Tukey's test at 5% significance level for S1 at varying moisture content (a, b, c) and surfaces (A, B, C, D) and for S2 at varying moisture content (x,y,z) and surfaces (A,B,C,D) Values followed by same letters are not significantly different.

In both samples, static friction coefficient was significantly affected by the interaction between moisture content and the type of surface over which the samples moved. At all moisture levels, the static friction coefficient on stainless steel was the smallest as its relatively smooth surface offered lower resistance compared to the rougher surfaces of concrete and wood. In general, the static friction coefficient on wood, with the grain perpendicular to the direction of motion, was significantly higher compared when the wood grain was along the direction of motion. This was observed in almost all of the moisture levels in both samples, except for the two higher moisture levels in S2 where both wood surfaces gave statistically similar friction coefficients. For the same surface, samples with higher moisture content gave significantly higher static friction coefficients than those with lower moisture. This was observed in all surfaces, except in S1 where the static friction coefficient of wheat DDGS on wood, particularly those obtained when

wheat DDGS moved perpendicular to the wood grain, did not show significant difference across the three moisture levels.

The coefficient of wall friction and adhesion of wheat DDGS on various surfaces is presented in Table 6.10. These values were derived from the results of fitting Eq. 6.5 with the experimental data on normal stress (σ) and shear stress (τ). The linear model (Eq. 6.5) fitted well with the experimental data, as indicated by R^2 and standard error of the estimate values in Table 6.10.

Coefficient of wall friction of wheat DDGS samples at about 13-13.5% moisture varied from 0.252 to 0.282 on galvanized steel, 0.283 to 0.312 on Teflon, and from 0.205 to 0.357 on polypropylene surface. These wheat DDGS coefficients were lower compared to those found for chickpea flour with 10.6% moisture (Emami and Tabil 2008) on steel and Teflon surfaces. Adhesion of wheat DDGS was highest on the steel surface.

Table 6.10 Coefficient of wall friction and adhesion of wheat distillers dried grain with solubles on galvanized steel, Teflon, and polypropylene surfaces. Samples were obtained from two production batches (S1, S2) of the same ethanol plant (N=3).

Sample batch	Moisture, % wet basis	Surface type	Coefficient of wall friction ($\tan \theta$)	Adhesion (A), kPa	R^2	Standard error of the estimate
S1	13.47 (0.02)	Galvanized steel	0.252	4.912	0.943	0.947
		Teflon	0.283	2.195	0.991	0.414
		Polypropylene	0.357	0.396	0.981	0.760
S2	12.94 (0.46)	Galvanized steel	0.282	3.299	0.972	0.972
		Teflon	0.312	1.303	0.936	1.245
		Polypropylene	0.205	2.946	0.985	0.388

6.5 Conclusions

This study assessed the proximate composition and the effect of moisture content on the physical properties of commercially-sourced wheat distillers dried grain with solubles (DDGS). Proximate composition of samples obtained from two production batches of the same ethanol plant differed significantly.

When moisture content was increased, bulk density increased while particle density and compressibility decreased consistently in both samples. The effect of moisture content on the other physical properties was more apparent in the sample batch that has a wider moisture content range. Samples evaluated at statistically similar moisture content showed significant difference in their uniformity coefficient and thermal diffusivity.

Values of the physical properties measured for the plant-sourced samples were close to the ranges previously reported for laboratory-prepared, forced-air convection-dried wheat DDGS samples. In comparison with published values of corn DDGS, the wheat DDGS samples in this study had lower bulk density, were smaller in size, darker in color, slightly less flowable, and slightly more floodable. Moisture sorption and compression characteristics of wheat DDGS were adequately represented by the Guggenheim, Anderson and de Boer (GAB) and Kawakita-Ludde models, respectively.

Chapter 7

Techno-economic evaluation of drying wheat distillers grain with solubles using microwave energy in Saskatchewan fuel ethanol plants

A similar version of this chapter will be submitted to the Canadian Biosystems Engineering journal.

Contribution of the PhD Candidate

Development of the cost evaluation spreadsheet model, data analysis, and manuscript preparation were done by the PhD candidate. Cost evaluation was based on data and information obtained from literature, previous experiments, relevant industry webpages, and personal communication with a few industry and academic sources. Guidance and editorial advice during the entire process was provided by her research supervisor, Lope Tabil.

Contribution of the Paper to the Overall Study

One of the main objectives of this PhD research project was to evaluate the economic feasibility of incorporating microwave drying technology in Saskatchewan ethanol plants for wheat distillers dried grain with solubles (DDGS) production. This paper quantified the costs involved in, and assessed the economic feasibility of, incorporating microwave technology in Saskatchewan fuel ethanol plants for drying wheat wet distillers grain with solubles (WDGS). There is very limited published information on the cost of drying wheat WDGS using the

conventional hot air drying systems, much less on alternative drying technologies such as the use of microwave energy. Published papers on corn ethanol production economics focused only on overall production costs and did not have separate assessments on DDGS production. Thus, information derived from this evaluation could contribute toward addressing this gap and could be useful to ethanol plant operations, quality assurance, and finance managers in examining existing processing costs and quantifying costs of protein quality improvement. Information may also be of interest to microwave dryer manufacturers.

7.1 Abstract

The incorporation of microwave drying system in industry-scale wheat distillers dried grain with solubles (DDGS) production was evaluated for economic viability under three scenarios: (i) microwave drying, where only microwave energy was used in reducing product moisture from 70% to 10% wet basis (wb); (ii) booster drying, where microwave energy was applied after rotary drying when drying rates began to fall; and (iii) finish drying, where microwave drying was used near the end of the drying process. Complete replacement of the conventional hot air drying system with microwave energy was not economically feasible under the present set of assumptions. Although energy requirement during microwave drying was substantially lower than rotary drying, the cost of electricity in providing the microwave energy was seen as a major hindrance. Lower electricity rates, availability of cheaper power sources, and attractive market incentives, such as premium prices for high protein quality wheat DDGS, may be necessary to encourage ethanol producers to invest in the technology. Finish drying, which used lowest amount of electrical energy among the three scenarios, was seen as the more economically viable option. Costs associated with the other DDGS production processes also have to be assessed to

have a more comprehensive picture of the costs and the benefits of investing on microwave drying technology for protein quality improvement.

7.2 Introduction

Wheat distillers dried grain with solubles (DDGS), a protein-rich co-product of fuel ethanol production, is primarily utilized in western Canada as an animal feed ingredient (FOBI Network 2011). Its efficient production and maximum utilization enhances the revenue generation potential of ethanol plants. A spreadsheet model maintained by Hofstrand (2013), for example, estimated that corn DDGS contributed between 7 – 26% of the total revenues generated from every gallon of ethanol. A similar revenue contribution may be assumed for wheat DDGS.

Reduced protein quality and a highly energy intensive drying process are two of the current challenges in the production and utilization of wheat DDGS (FOBI Network 2011). High incidence of heat-damaged proteins in wheat DDGS, primarily due to high temperature drying, hinders its marketing prospects, particularly if it is utilized as a substitute for the more expensive, high-protein ingredients in non-ruminant feed formulations (Widyaratne and Zijlstra 2007). It has also been estimated that the drying process could account about one-third of the total thermal demand in wheat ethanol plants (Murphy and Power 2008). Rising costs of natural gas and the potential for increased revenue generation from improved wheat DDGS quality could provide impetus for fuel ethanol producers to evaluate and improve existing plant processes.

In this study, microwave-based methods were investigated for their potential in minimizing both energy consumption and protein damage. Laboratory-scale investigations using a domestic microwave oven had demonstrated that with proper selection of drying conditions, microwave-

based methods could provide DDGS protein quality associated with low temperature drying at much faster rates. Wheat wet distillers grain with solubles (WDGS) with 45% condensed distillers solubles (CDS) content and dried under 676 W microwave power or at a microwave convection nominal setting of 150°C-30% power (316 W), for example, had comparable lysine and acid detergent insoluble crude protein (ADICP) contents with those attained under 80°C forced-air convection drying (Mosqueda et al. 2013a). At these drying conditions and sample type (45% CDS), the drying rate constants (Page model parameter k) under microwave convection and microwave methods were 29% and 571% higher than the one obtained under 80°C forced-air convection, respectively. Microwave vacuum-dried wheat DDGS samples (Appendix A) had lower ADICP content (10.87 – 14.41% db) than an ethanol plant-dried (18.37% db) sample. Both sample types were assumed to be derived from the same wheat WDGS batch as these were sourced on the same date from the same ethanol plant. Laboratory drying of ethanol plant-sourced wheat WDGS using the same equipment also showed that the microwave method (676 – 805 W) produced drying rate constants that were 208 – 433% higher than those achieved under 120°C forced-air convection (Mosqueda and Tabil 2011a). These higher drying rates could translate to considerable energy savings.

The use of microwave energy, whether singly or in combination with forced-air convection or vacuum drying methods, had been investigated in drying a wide variety of plant-based materials. Some of these studies covered fruits (Mousa and Farid 2002; Meda et al. 2008; Sunjka et al. 2004; Dadali et al. 2007a; Wang et al. 2009b), grains and oilseeds (Lupinska et al. 2009; Walde et al. 2002), leafy plant parts (Cui et al. 2004; Ozkan et al. 2007), and bulbs, tubers, and root crops (Song et al. 2009; Lin et al., 1998; Sharma and Prasad, 2001). Areas commonly investigated include drying kinetics (Song et al. 2009; Drouzas et al. 1999), energy consumption

and drying efficiency (Mousa and Farid 2002; Meda et al. 2008; Wang et al., 2009; Du et al., 2005), and retention of selected product quality characteristics (Cui et al. 2004; Sunjka et al. 2004; Lin et al. 1998; Alibas et al. 2006; Ozkan et al. 2007; Lupinska et al. 2009). While these studies showed that the use of microwave energy could be an attractive alternative for drying plant materials, there is limited published literature on the economic implications of its incorporation in the commercial processing of these materials. None was found on wheat DDGS drying.

The objective of this paper, then, was to assess the economic viability of incorporating a microwave drying system for wheat DDGS production in Saskatchewan ethanol plants. Three scenarios were considered: (i) complete replacement of conventional hot air drying with a microwave drying system; (ii) use of microwave energy after drying rates of the conventional hot air drying process began to fall; and (iii) use of microwave energy toward the end of conventional hot air drying. Combining both microwave and hot air systems could lower the drying cost compared to using microwave energy alone.

7.3 Materials and Methods

A Microsoft Excel spreadsheet model was developed to evaluate the cost implications of incorporating a microwave drying system into Saskatchewan fuel ethanol plant operations. Evaluation was limited to the drying process only. Costs associated with plant design changes associated with microwave technology adoption as well as costs of operating the other DDGS production processes (centrifugation, evaporation, blending) were not considered.

7.3.1 Plant capacity and annual operating time

Saskatchewan has five grain-based ethanol plants, with nameplate capacities ranging from 15 to 150 million liters per year (MLPY) (Government of Saskatchewan 2013). Three of these plants utilized wheat as feedstock, while the other two utilized both wheat and corn. One of the wheat ethanol plants has integrated cattle feedlot operations that fully utilized its by-products (wet distillers grain and thin stillage) while the other two produced DDGS and sell this to third parties. These latter two plants, with nameplate capacities of 25 and 150 MLPY, were used as models in the assessment.

Annual operating time was assumed to be 8000 h, with about 4 weeks allowance for regular preventive maintenance programs and other unforeseen downtimes. This time is equivalent to operating about 92% of the plants' nameplate capacities, if the plants were to operate all year with 24 h-days. This plant utilization percentage also fell within the performance range of the plants in 2010-2011, wherein ethanol production ranged from 72% to 98% of their nameplate capacities (Government of Saskatchewan 2013).

7.3.2 Wheat DDGS production process

Figure 7.1 presents the wheat DDGS production process considered in the techno-economic evaluation. Whole stillage, derived from ethanol production, is separated into two streams through centrifugation: (i) the solid fraction or the wet distillers grain (WDG), and (ii) the liquid fraction or the thin stillage. The thin stillage is subjected to evaporation to produce condensed distillers solubles (CDS), often referred to as “syrup” in industry parlance. Then, CDS is blended with WDG and dried to produce DDGS with 10% moisture (wb). In this evaluation, it is

assumed that about 375 L of ethanol and 370 kg of DDGS are produced for every tonne of wheat grain processed (FOBI Network 2011).

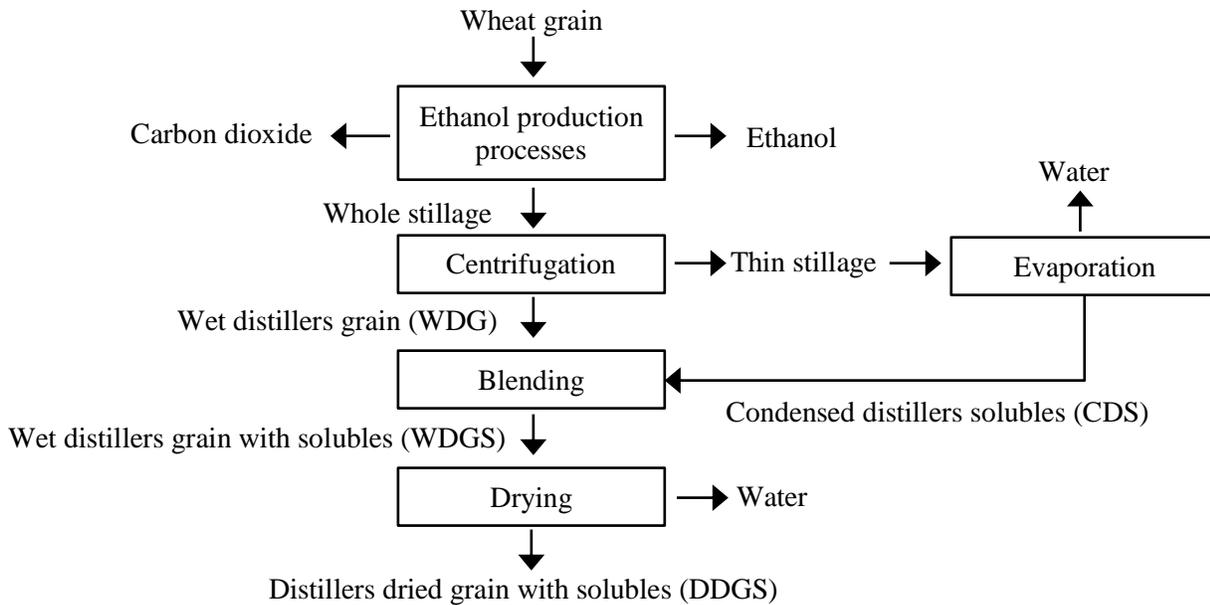
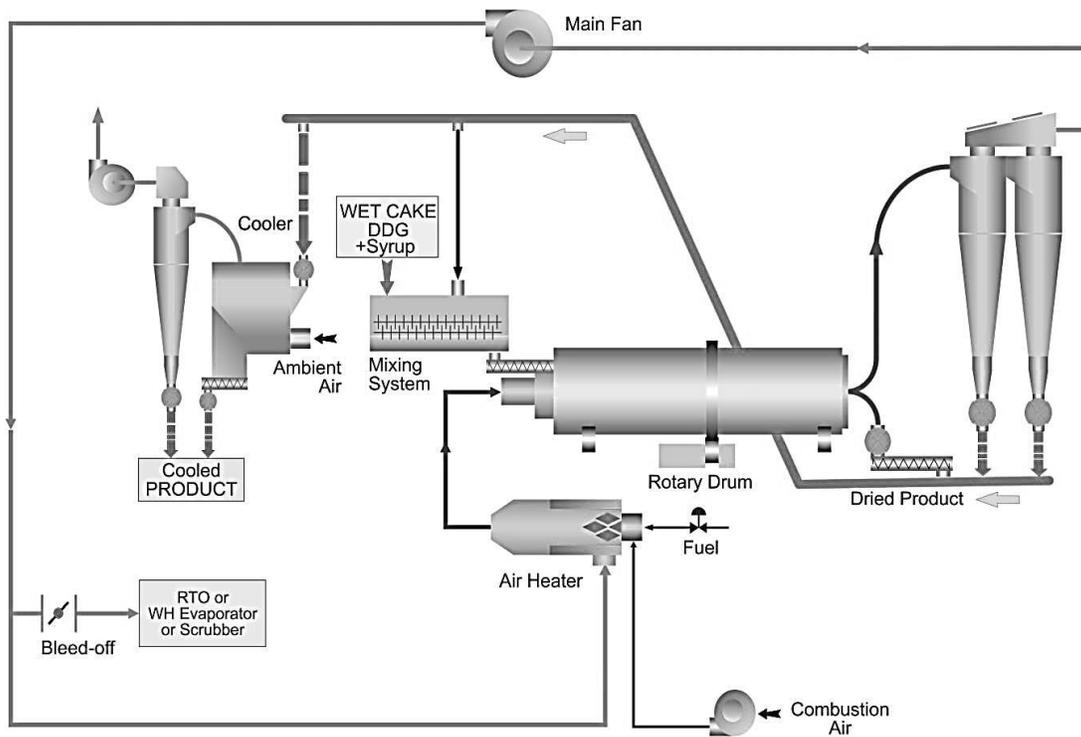


Fig. 7.1 Unit operations involved in the production of wheat distillers dried grain with solubles in selected Saskatchewan fuel ethanol plants.

7.3.2.1 Base case scenario

Reducing the wheat WDGS moisture from 70% (wb) to 10-12% moisture (wb) is usually accomplished using a rotary drum or a ring dryer. In this evaluation, the use of the direct-fired rotary dryers was considered as the conventional hot air drying method. Figure 7.2 shows a schematic diagram of blending and drying processes of DDGS production system involving a rotary drum dryer. It is further assumed that the air heater for the rotary drum dryer used natural gas as fuel. Rotary dryer thermal efficiency was assumed at two levels, 50% and 75% (Krokida et al. 2007), and combustion efficiency at 85% (Gilson Engineering Sales, Inc. n.d.)



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Fig. 7.2 A rotary drum drying system (With permission from GEA Barr-Rosin).

7.3.3 Microwave-based drying scenarios

The evaluation was made on three scenarios. Scenario 1 involved the use of microwave energy only in drying wheat WDGS from 70% to 10%, wet basis (wb). Scenarios 2 and 3 employed the sequential use of the conventional hot air and microwave drying systems.

In scenario 2, rotary drying initially reduced the moisture of the WDGS from 70% to 50% (wb). This was followed by microwave drying to further reduce the product moisture from 50% to 10% (wb). This scenario corresponds to what Schiffman (2007) referred to as booster drying. Microwave drying was employed when the drying rates under the conventional hot air system

began to fall. In a previous investigation on laboratory-scale forced-air convection drying of ethanol plant-sourced wheat WDGS (Mosqueda and Tabil 2011a), drying rates began to fall when moisture content was approximately between 55% (wb), under 120°C drying, and 62% (wb), under 40°C drying. Since typical drying temperatures employed in ethanol plants are higher than 120°C, it was assumed that drying rates would fall at a moisture content lower than 55% (wb). A 50% moisture (wb) was assumed in the evaluation.

In scenario 3, rotary drying reduced the moisture of wheat WDGS from 70% to 25% (wb) and microwave drying was employed, thereafter, to further reduce the moisture of the partially dried material to 10% (wb). This scenario corresponds to what Schiffman (2007) referred to as finish drying. The 25% moisture was chosen for the evaluation because it was half the initial product moisture in the booster drying scenario and near the target final product moisture of 10% (wb). These three drying scenarios were referred to in this report as “microwave” (scenario 1), “booster” (scenario 2), and “finish” (scenario 3).

7.3.3.1 *Drying system description*

Figure 7.3 shows the schematic diagram of a commercially available microwave drying system used in the cost assessment. The system’s main components include: (i) microwave generator, which converts alternating current line power to microwave energy; (ii) the waveguide assembly, which delivers the microwave energy from the generator to the oven assembly; (iii) the oven or the applicator, which is a sealed enclosure where the material to be dried is horizontally carried on a microwave-inert conveyor belt and exposed to microwave energy; and (iv) a control system, which is used to monitor and regulate the entire drying operation (Cellencor, Inc. 2013).

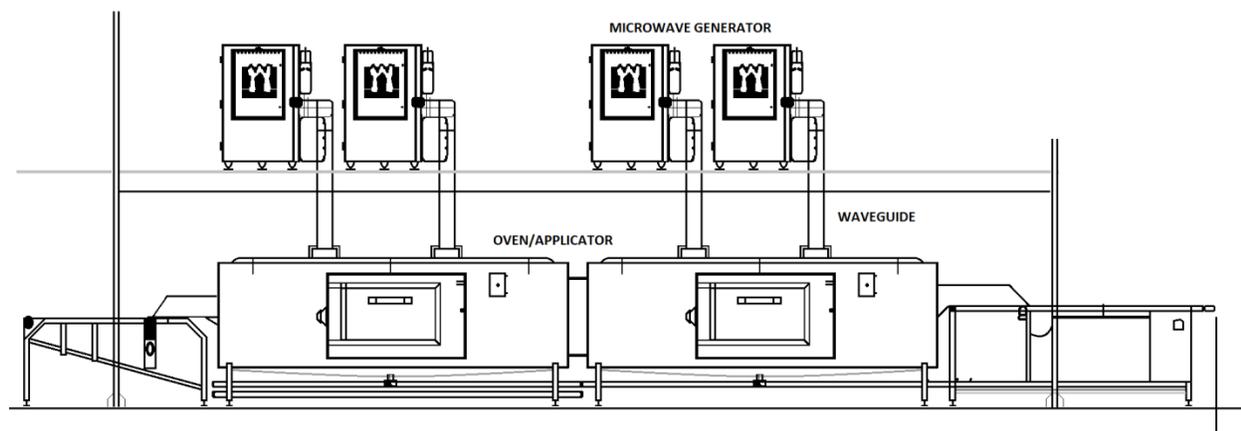


Fig. 7.3 Schematic diagram of a commercially available 915 MHz microwave drying system (Adapted with permission from Cellencor, Inc.)

It was assumed that the smallest and largest commercially available microwave drying systems were sized at 75 kW and 1 MW, respectively (Cellencor, Inc. 2013). The 75 kW microwave drying system, for example, comprised one - 75 kW 915 MHz microwave generator, one oven, a waveguide assembly, and a control panel while the 1 MW system would have ten - 100 kW 915 MHz microwave generators and five ovens. Each oven measures 3.66 m (length) x 1.32 m (width) x 1.22 m (height). Various size configurations between the smallest and largest units can be assembled using 75 kW or 100 kW microwave generators, depending on the microwave energy requirement of the process.

7.3.3.2 *Drying system efficiency*

Information on efficiency of microwave heating or drying systems from industry websites, brochures, journal articles, and reports showed a wide range of values. These are illustrated in Fig. 7.4. Schiffman (2007), for example, indicated a 45-50% conversion efficiency from electricity to microwave energy. This included about 4% loss in converting ac to dc, about 40%

loss from converting dc to microwave, and waveguide and applicator losses of about 10%. Industry websites, on the other hand, indicated conversion efficiencies of 85-88% (Industrial Microwave Systems, LLC 2012; Envirowave Corporation 2013). Others used or indicated efficiency values between 70 to 75% (Doering and Hennessy 2008; Hasna 2011). Efficiency values found at the oven/applicator level ranged from 75% to 97% (Disman 1966; Schiffman 2007; Industrial Microwave Systems, LLC 2012; Cellencor, Inc. 2013).

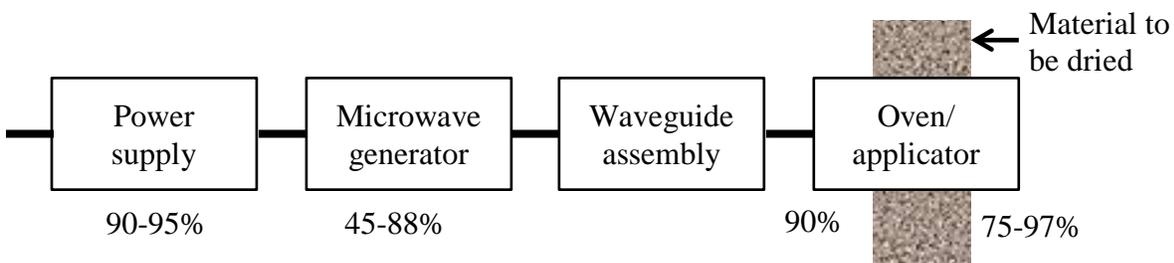


Fig. 7.4 Range of efficiency values of microwave drying/heating systems found in selected industry and technical literature sources.

Since drying system efficiency is a major factor in estimating energy requirement and costs (Disman 1996), the evaluation further used three energy efficiency levels under each of the three scenarios considered: *low* (50% microwave generator efficiency (e_G) and 75% applicator efficiency (e_A) or 38% overall system efficiency), *medium* (70% e_G and 85% e_A or 60% overall system efficiency) and *high* (85% e_G and 95% e_A or 81% overall system efficiency).

7.3.4 Mass and energy balance analyses

7.3.4.1 Mass flow rates

The quantity of wheat WDGS entering the drying process, water to be evaporated, and the DDGS produced per hour were obtained using mass balance analyses, involving the following

parameters: the plants' nameplate capacities, % plant utilization, annual operating time, and initial and desired final product moisture contents under each drying scenario. Equations used and corresponding sample calculations are presented in Appendix B.

7.3.4.2 Total heat load

The total heat load (E_T) for each drying scenario was estimated using Eq. 7.1, where m_w is the amount of water to be evaporated (kg) per hour, L_v is the latent heat of vaporization at 100°C (2260 kJ/kg), m_p is the mass of the material to be dried per hour, c_p is the specific heat of the feed material, T_i and T_f represent the initial and final product temperature.

$$E_T = m_w L_v + m_p c_p (T_f - T_i) \quad (7.1)$$

The values of m_w and m_p were derived from mass balance analyses. Initial temperature of WDGS (70% moisture) was 80°C, based from firsthand measurements made in the ethanol plant before the WDGS entered the drying process. Initial temperature of the other two feed materials for microwave drying under the booster and finish drying scenarios were assumed to be at 100°C, since these already underwent hot air drying. Process temperature under microwave drying was assumed at 100°C.

Specific heat (c_p , $\text{kJ}\cdot\text{kg}^{-1}\cdot\text{C}^{-1}$) of the wheat WDGS (70% moisture) was obtained using a differential scanning calorimeter (DSC Q2000, TA Instruments, New Castle, DE). Samples were obtained from two production batches of a Saskatchewan ethanol plant and were evaluated using three replicates. A heating rate of 10°C/min was used from an initial temperature of 20°C to 240°C. Experimental results at 80°C were obtained and compared to Eq. 7.2, Choi and Okos's generalized equation for specific heat (cited in Stroshine 1998), using the proximate composition

obtained from a previous study (Mosqueda and Tabil 2011c). The variable X in the equation represents the mass fraction of each chemical constituent. The subscripts w, p, f, c, and a, represent water, protein, fat, carbohydrates, and ash, respectively. Test results of commercial samples were found to be 7 to 13% lower than the result from Eq. 7.2. Specific heat of the other feed materials (50% and 25% moisture DDGS) was estimated using Eq. 7.2.

$$c_p = 4.180X_w + 1.711X_p + 1.928X_f + 1.547X_c + 0.908X_a \quad (7.2)$$

7.3.5 Estimation of equipment size and number

The results of mass and energy balance analyses were used in estimating the required size and number of the drying units under each scenario. Other assumptions are presented in the next pages with the corresponding sample calculations presented in Appendix B.

7.3.5.1 Microwave drying system

The microwave energy requirement (E_G) for drying was determined using Eq. 7.3, where e_A is the efficiency of the applicator/oven and E_T is the total heat load, previously estimated in Eq. 7.2.

$$E_G = \frac{E_T}{e_A} \quad (7.3)$$

Using the results of Eq. 7.3, the equivalent number of 75 or 100 kW microwave generators, waveguide assemblies, and ovens/applicators required by each drying scenario was estimated. For simplicity in calculations, two microwave generators were assumed to be connected to each oven/applicator. A maximum of 1 MW was assumed to comprise one drying line.

7.3.5.2 Rotary dryer

An evaporation rate of $14.64 \text{ kg}\cdot\text{m}^{-2}\cdot\text{h}^{-1}$ (Gibson 1994) was used in estimating required size of rotary direct-fired dryers. Equivalent dryer diameter, length, and number of units required under each drying scenario were estimated based on commercially available sizes (Zhengzhou Kehua Industrial Equipment Co., Ltd. 2013).

7.3.6 Equipment and operating costs

Cost estimation was limited to the drying equipment, fuel and utilities consumption, magnetron replacement, personnel and general maintenance. Except for personnel, the rest were among the basic cost components outlined by Disman (1966), Jolly (1976), and Schiffman (2007) in evaluating the economic viability of microwave drying systems. Depreciation and operating costs were summed to obtain the total unit cost of drying. Costs were calculated in Canadian dollars (CAN\$) per hour of operation but were expressed later on in CAN\$ per tonne DDGS (10% moisture, wb) to facilitate comparison with historical market prices. Sample calculations for these cost items are presented in Appendix B.

7.3.6.1 Microwave drying system

It was assumed that smallest microwave drying system (75 kW) costs about US\$ 150,000.00 and the 1 MW system about US\$ 1,200,000.00 (personal communication, K.Kaplan, President, Cellencor, Inc., 30 June 2013).

Equipment price was calculated based on the cost of each 1 MW system while fractions of 1 MW was estimated using the six-tenths rule (Chilton 1950), shown as Eq. 7.4. In this equation, I_2 represents investment cost for the desired capacity (Q_2), I_1 is the investment cost for the known

capacity (Q_1) while x is the cost capacity factor, assumed at 0.60 (Chilton 1950). In this calculation, Q_1 and I_1 values were 1 MW and US\$ 1,200,000.00, respectively.

$$I_2 = I_1 * (Q_2 / Q_1)^x \quad (7.4)$$

In estimating the purchase price of a 5.6 MW system, for example, the 5 MW would be valued at US\$ 1,200,000.00 per MW while the cost of the remaining fraction (0.6 MW) would be estimated using Eq. 7.4. Thus, the 5.6 MW system would be valued at US\$ 6,883,226.00.

Installed cost of the drying equipment was assumed to be 1.4 times the purchase price (Couper et al. 2010). The 40% increase was assumed to cover for the cost of the allocated building space, instrumentation, ancillary equipment such as blowers to remove water vapor from ovens/applicators, specialized materials handling equipment, and other associated installation costs.

Since the equipment would be sourced from the US, other charges such as freight cost, US sales tax (4%), Canadian custom duties (8%), and goods and services tax (5%) were added to comprise the total equipment cost. Freight cost was estimated using number of required container vans to transport the equipment, distance between the supplier and the Saskatchewan plant, and the prevailing freight rate per mile (Freight Rate Index 2013). Foreign exchange rate of US\$ 1.00:CAN\$ 1.03 (Bank of Canada 2013a) was assumed.

Total equipment cost (C_E) was amortized over a 10 year economic life (L_E) using straight-line depreciation method. Depreciation cost per hour (C_{DEP}) was obtained using Eq. 7.5, where i is the interest rate, S_V is the salvage value and t_A is the annual operating time (8000 h). Interest rate used was 15% (Bank of Canada 2013b) while salvage value was assumed to be zero.

$$C_{DEP} = \frac{(C_E - S_V) * (1+i)}{L_E * t_A} \quad (7.5)$$

7.3.6.2 Rotary drying unit

The cost of the rotary direct-fired dryer (C_{RD} , in US\$ 1000) was estimated using Eq. 7.6 (Couper et al. 2010), where A refers to the lateral surface area in sq. ft., while f_g and f_m are the drying gas and material factors, where values of 0.12 (direct contact) and 1.4 (stainless steel 304 type) were assumed, respectively.

$$C_{RD} = 1.218(1 + f_g + f_m) * \exp(4.9504 - 0.5827 \ln(A_S) + 0.0925 (\ln(A_S))^2) \quad (7.6)$$

The same previous assumptions for the installation cost, freight cost, US sales tax, Canadian custom duties, and goods and services tax in the microwave drying equipment were applied.

7.3.6.3 Electricity

It was assumed that the ethanol plants did not generate their own electricity and relied on an external service provider for their power supply. Cost of electricity per hour (C_p) was estimated using Eq. 7.7, where E_p is the electrical energy consumption, in kW, and U_p is the unit energy cost, assumed at CAN\$ 0.082 per kilowatt-hour. This unit cost was derived from calculations using the 2013 SaskPower standard rates (SaskPower 2013) covering basic monthly charge, demand charge, energy charge, and surcharges and taxes.

$$C_p = E_p * U_p \quad (7.7)$$

In microwave drying, E_P is equal to the amount of electrical energy drawn from the main supply lines (E_S), estimated using Eq. 7.8. In this equation, E_G was derived from Eq. 7.3 while e_G represents the microwave generator efficiency.

$$E_S = \frac{E_G}{e_G} \quad (7.8)$$

In rotary drum drying, power requirement of electric motors used to drive dryer rotation was based on the accompanying technical details of each rotary dryer size chosen (Zhengzhou Kehua Industrial Equipment Co., Ltd. 2013). This power requirement was also verified using Eq. 7.9, proposed by the CE Raymond Division, Combustion Engineering (as cited in Krokida et al. 2007) to determine power required to drive a dryer with flights. In this equation, bhp is the brake horsepower, S_R is rotational speed in rpm, D is the shell diameter (ft), w is the load of the material to be dried (lb), W is the total rotating load (equipment and material to be dried, lb), and D' is the riding-ring diameter and is equal to $D + 2$. Result of Eq. 7.9 was converted to kW and compared with the supplier-prescribed power requirement. The larger value of the two was used in the computation.

$$\text{bhp} = \frac{S_R(4.75Dw + 0.1925D'W + 0.33W)}{100,000} \quad (7.9)$$

7.3.6.4 Water

About 23 – 38 L·min⁻¹ of water were needed to cool each microwave generator (Cellencor, Inc. n.d (a)). Cost of water consumption per hour (C_W) was estimated using Eq. 7.10, where M_W , N_G , and U_W stand for the cooling water requirement per microwave generator (30 L·min⁻¹), required number of microwave generators, and the cost of water, respectively. Cost of water was assumed

at CAN\$ 0.6201 per cubic meter, based on updated SaskWater non-potable water rates (personal communication, SaskWater customer service agent, 04 Oct 2013), plus a 7% tax rate.

$$C_W = M_W * N_G * U_W * 1.07 \quad (7.10)$$

7.3.6.5 *Natural gas*

The cost of natural gas per hour of operation (C_{NG} , Eq. 7.11) was obtained by multiplying the total energy requirement to be supplied by natural gas and the unit cost of natural gas (U_{NG} , CAN\$·kJ⁻¹). Total energy requirement to be supplied by natural gas was estimated by dividing the result of Eq. 7.1 (E_T) with rotary dryer thermal efficiency (e_D) and combustion efficiency (e_C). The assumed values for e_D were 0.50 and 0.75 (Krokida et al. 2007) while that for e_C was 0.85 (Gilson Engineering Sales, Inc. n.d.), respectively. Unit cost of natural gas was derived from the average Jan – Aug 2013 price of US\$ 3.70 per million BTU at the Henry Hub (Canadian Gas Association 2013). Since E_T was expressed in kJ·s⁻¹, a conversion factor of 3600 was introduced in Eq. 7.11 to express C_{NG} on a per hour basis.

$$C_{NG} = \frac{E_T * 3600}{e_D * e_C} * U_{NG} \quad (7.11)$$

7.3.6.6 *Magnetron replacement*

It was recommended to replace the magnetrons annually or after 8000 h of operation (Industrial Microwave Systems, LLC 2012). Magnetron replacement cost per hour (C_M) was estimated using Eq. 7.12, where N , U , and t_A stand for number of magnetrons, unit replacement cost, and the annual operating hours. The subscripts 75 and 100 refer to the magnetron size in kW. Unit costs of new 75 kW and 100 kW magnetrons were assumed at CAN\$ 8485.00 and CAN\$

9806.00, respectively, inclusive of US sales tax (4%), custom duties (8%), goods and services tax (5%), and freight cost. Magnetron price quotes were obtained from a US supplier.

$$C_M = \frac{N_{75} * U_{75} + N_{100} * U_{100}}{t_A} \quad (7.12)$$

7.3.6.7 General maintenance cost

General maintenance cost per hour (C_{GM} , Eq. 7.13) of the microwave and rotary drying system was assumed at 2% (Schiffman 2007) and 10% (Krokida et al. 2006) of the total installation cost, respectively. In Eq. 7.13, C_i is the total installation cost, p_i is the percentage of the total installation cost, and t_A is the annual operating hours of the plant.

$$C_{GM} = \frac{p_i * C_i}{t_A} \quad (7.13)$$

7.3.6.8 Personnel

Average annual salary of plant personnel was assumed at CAN\$ 90000.00, based on the median salary of Saskatchewan engineers and geoscientists for 2013 (APEGS 2013). Number of personnel (N_M) for the 25 and 150 MLPY plants was assumed to be 10 and 40, respectively. Labor cost directly associated with the drying process was determined by dividing the total labor cost with the total number of major ethanol and DDGS unit operations. Ten unit operations were used in the calculation.

Incorporating microwave drying technology into existing plant operations requires the workforce to have a new set of specific skills and knowledge and to be more adaptable with the associated changes in plant operations. To meet this need, it was assumed that an appropriate training program will be crafted and implemented to benefit existing plant personnel in various aspects of

production, equipment maintenance, materials handling, laboratory, and management of DDGS processes. Training cost (C_T) was pegged at CAN\$ 1800.00 per person per year (U_M) and estimated using Eq. 7.14.

$$C_T = \frac{U_M * N_M}{t_A} \quad (7.14)$$

7.4 Results and Discussion

Tables 7.1 and 7.2 summarize the evaluation results for the 25 and 150 MLPY ethanol plants, respectively.

7.4.1 Energy requirement

Rows b and d of Tables 7.1 and 7.2 show the energy input requirement per unit time at the dryer and at the main power supply line. To dry the same amount of wheat WDGS to the same target moisture level, these tables and figure showed that microwave drying (scenario 1) required the least amount of energy per unit time among the scenarios evaluated (row b of Tables 7.1 and 7.2, first stacked column of Fig. 7). The energy requirement for microwave drying was 49-60% and 23-40% lower than rotary drying when the latter was assumed to operate at 50% and 75% thermal efficiency, respectively. This was followed by booster drying (scenario 2), with an energy requirement that is 10-14% and 0-6% lower than rotary drying at the same thermal efficiency levels. Microwave drying under this scenario accounted 18-29% of the total energy requirement and removed about 40% of the water targeted for evaporation. The energy requirement of finish drying (scenario 3) was closest to that of rotary drying. The microwave drying phase under this scenario removed 10% of the total moisture to be evaporated and accounted 4-7% of the total energy requirement. Values for energy requirement at the source

Table 7.1 Techno-economic evaluation results of drying wheat distillers grain with solubles using rotary, microwave, booster, and finish drying systems in a 25 MLPY¹ Saskatchewan ethanol plant operating at 92% of their nameplate capacity. Drying systems were evaluated at two (low, high) efficiency levels².

Particulars	Rotary drying (Base case)	Scenario 1- Microwave		Scenario 2 - Booster Drying		Scenario 3 - Finish drying	
		Low	High	Low	High	Low	High
a. Material capacity (moisture, wb)							
WDGS input rate, kg·h ⁻¹ (70%)	8510.0	8510.0	8510.0	8510.0	8510.0	8510.0	8510.0
DDGS output rate, kg·h ⁻¹ (10%)	2836.7	2836.7	2836.7	2836.7	2836.7	2836.7	2836.7
b. Energy input during drying, kJ·s⁻¹							
Rotary dryer efficiency: Low	9703.1	4951.1	3908.8	8753.4	8353.5	9465.7	9365.7
High	6468.8	4951.1	3908.8	6468.8	6068.9	6468.7	6368.8
c. Equipment size and number							
Rotary drying system							
Size (<i>diameter x length</i> , m)	2.4 x 20			1.8 x 14	1.8 x 14	2.2 x 18	2.2 x 18
Number of units	3			3	3	3	3
MW drying system							
Total size, kW		5000	3950	1900	1500	500	400
No. of ovens		25	20	10	8	3	2
d. Energy requirement from source, kW							
Rotary dryer efficiency: Low	11584.2	10000.0	4647.1	11975.9	9940.6	11746.2	11216.8
High	7779.1	10000.0	4647.1	9288.1	7252.8	8220.4	7691.0
e. Drying cost, CAN\$·t⁻¹ DDGS							
Rotary dryer efficiency: Low	101.16	394.80	2p19.30	219.31	153.78	134.13	116.33
High	83.72	394.80	219.30	206.99	141.46	117.96	100.16

¹MLPY – million liters of ethanol per year;

²Microwave drying system efficiency: *low* (50% generator efficiency, 75% applicator efficiency), and *high* (85% generator efficiency, 95% applicator efficiency); Rotary dryer efficiency: *low* (50% dryer efficiency, 85% combustion efficiency) and *high* (75% dryer efficiency and 85% combustion efficiency)

Table 7.2 Techno-economic evaluation results of drying wheat distillers grain with solubles using rotary, microwave, booster, and finish drying systems in a 150 MLPY¹ Saskatchewan ethanol plant operating at 92% of their nameplate capacity. Drying systems were evaluated at two (low, high) efficiency levels².

Particulars	Rotary drying (Base case)	Scenario 1- Microwave		Scenario 2 - Booster Drying		Scenario 3 - Finish drying	
		Low	High	Low	High	Low	High
a. Material capacity (moisture, wb)							
WDGS input rate, kg·h ⁻¹ (70%)	51060.0	51060.0	51060.0	51060.0	51060.0	51060.0	51060.0
DDGS output rate, kg·h ⁻¹ (10%)	17020.0	17020.0	17020.0	17020.0	17020.0	17020.0	17020.0
b. Energy input during drying, kJ·s⁻¹							
Rotary dryer efficiency: Low	58218.8	29706.8	23452.8	52520.9	50120.9	56794.2	56194.3
High	38812.5	29706.8	23452.8	38812.0	36413.1	38812.5	38212.7
c. Equipment size and number							
Rotary drying system							
Size (<i>diameter x length</i> , m)	2.8 x 24			2.8 x 24	2.8 x 24	2.8 x 24	2.8 x 24
Number of units	12			7	7	10	10
MW drying system							
Total size, kW		29750	23500	11400	9000	2850	2250
No. of ovens		149	118	58	45	15	12
d. Energy requirement from source, kW							
Rotary dryer efficiency: Low	69617.0	59500.0	27647.1	71836.4	59624.7	70102.1	67049.1
High	46786.8	59500.0	27647.1	55709.7	43497.9	48947.2	45894.3
e. Drying cost, CAN\$·t⁻¹ DDGS							
Rotary dryer efficiency: Low	95.43	390.14	215.75	212.45	145.00	122.69	106.21
High	77.99	390.14	215.75	200.13	132.68	106.53	90.10

¹MLPY – million liters of ethanol per year;

²Microwave drying system efficiency: *low* (50% generator efficiency, 75% applicator efficiency), and *high* (85% generator efficiency, 95% applicator efficiency); Rotary dryer efficiency: *low* (50% dryer efficiency, 85% combustion efficiency) and *high* (75% dryer efficiency and 85% combustion efficiency)

(row d of Tables 7.1 and 7.2) per unit time took into account the combustion efficiency of natural gas in the case of rotary drying and microwave generator efficiency in microwave drying.

Lower energy requirement of microwave drying (row b of Tables 7.1 and Table 7.2) was due to the use of higher efficiency levels compared to rotary drying. Energy efficiency at the microwave applicator/oven was assumed to range from 75% to 95% while efficiency at the rotary dryer was at 50-75%. Unlike in rotary drying, microwave energy directly couples with the material to be heated and is not used to heat the air surrounding the material and the dryer walls and other parts of the system (Schiffman 2001; 2007). The internal heat generated by the microwave field results to an internal pressure gradient within the material that enables higher drying rates compared to that observed in conventional hot air drying (Schiffman 2001; 2007).

Figure 7.5 shows the breakdown of these energy requirements for the microwave, boost, finish, and rotary drum drying systems at high efficiency levels (75% for rotary drum and 81% for microwave drying). Energy requirement for microwave drying would be derived from electricity, which was assumed to cost about CAN\$ 0.086 per kWh. About 98% of the energy requirement for rotary drum drying would be sourced from natural gas, which would cost about CAN\$ 0.013 per kWh, and the remaining 2% would be from electricity to run the motors needed for drum dryer rotation. In boost drying, about 26% of the total energy requirement would be supplied by electricity while the bulk (74%) would be sourced from natural gas. In finish drying, electricity and natural gas accounted about 8% and 92% of the total energy requirement, respectively. Since the cost of natural gas would be about 5-6x lower than the cost of electricity, on a per kilowatt-hour basis, rotary drum drying would be cheapest method and microwave drying the most

expensive. Because of the lower amount of electricity required in finish drying, this drying method would have a cost closest to rotary drum drying.

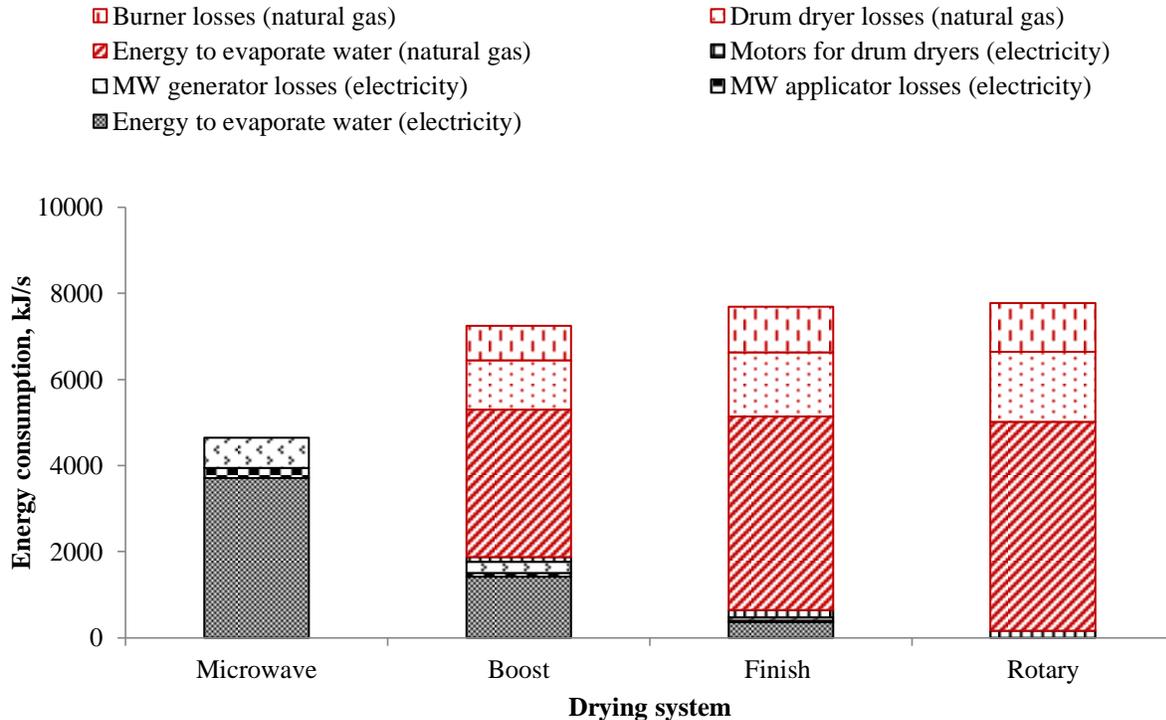


Fig. 7.5 Energy consumption of drying wheat distillers grain with solubles under microwave, boost, finish, and rotary drum drying methods. Assumed thermal efficiencies of rotary drum and microwave drying were at 75% and 81%, respectively.

7.4.2 Equipment size, number, and cost

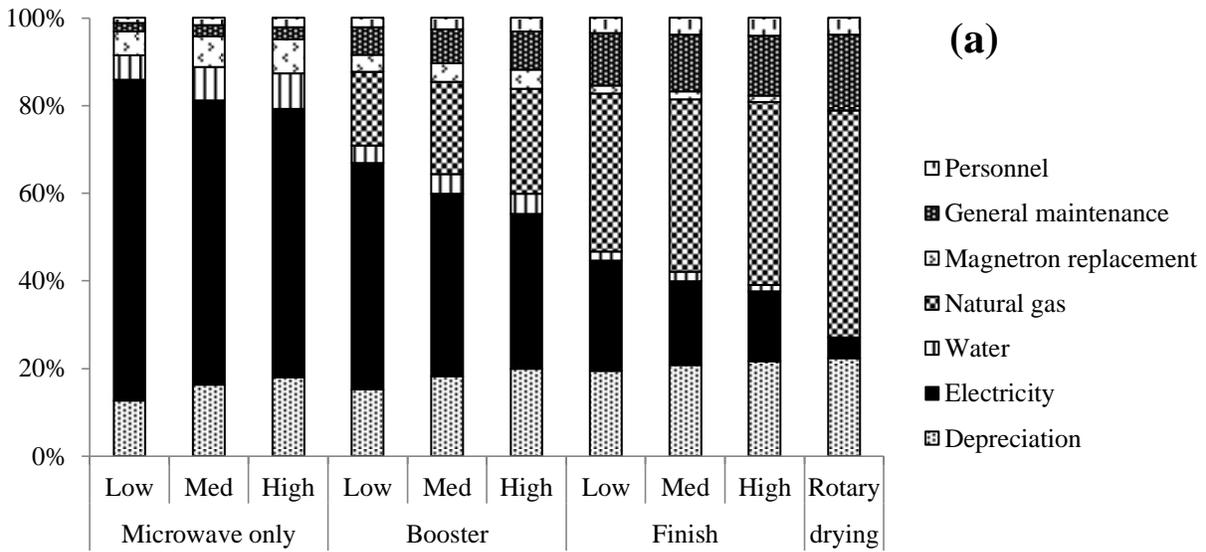
Tables 7.1 and 7.2 (row c) also show the estimated equipment size and number required under the various drying scenarios. Under rotary drying, the 25 MLPY and the 150 MLPY plants required three units of 2.4 m x 20 m and 12 units of 2.8 m x 24 m rotary drum dryers, respectively. These correspond to estimated initial equipment costs of about CAN\$ 4.45 million and CAN\$ 14.88 million, respectively. Use of 100% microwave energy (scenario 1), on the other hand, required initial equipment costs that were about 1.7 to 2.5 times higher than that of

rotary drying. Microwave drying equipment under scenario 1 was estimated to cost about CAN\$ 4.91 – 6.18 million and CAN\$ 29.24 – 36.8 million for the 25 and 150 MLPY plants, respectively. Equipment size and number required under booster and finish drying scenarios are also reflected in Tables 7.1 and 7.2. Equivalent initial equipment costs for the 25 MLPY plant, in CAN\$, were 3.82 - 4.17 million and 3.15 -3.25 million under the booster and finish drying scenarios, respectively. For the 150 MLPY plant, estimated equipment costs were CAN\$ 19.80 - 22.99 million for booster drying and CAN\$ 15.41 – 15.99 million for finish drying.

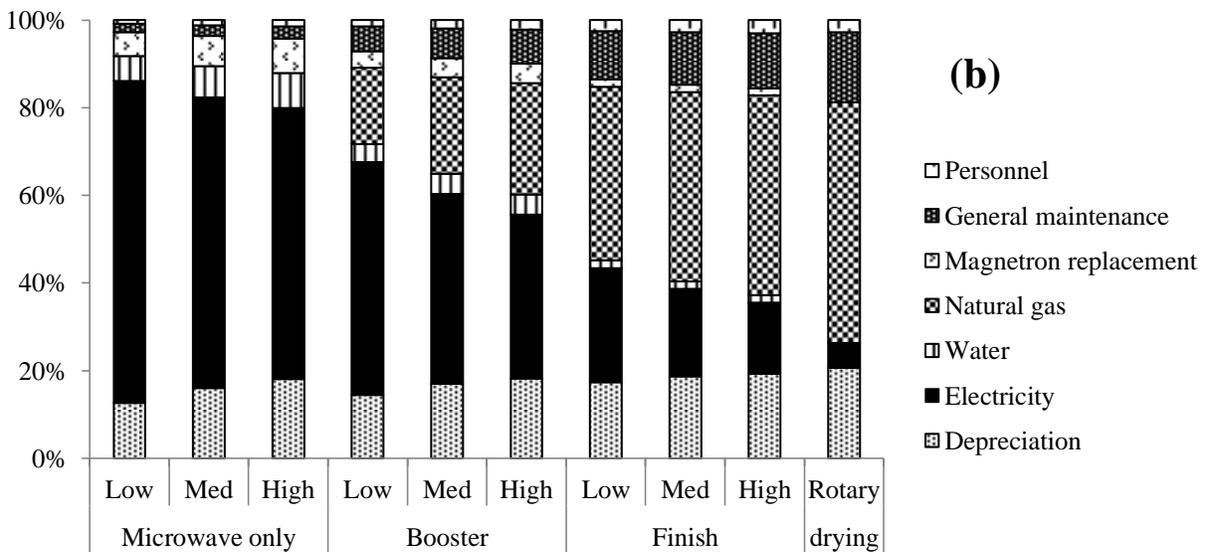
Efficiency levels assumed for the microwave drying system substantially affected the size and number of drying equipment and consequently, the associated acquisition and operating costs. Drying using the least efficient microwave system (50% microwave generator efficiency and 75% applicator efficiency) under scenario 2 in the 25 MLPY plant, for example, would require five more microwave ovens than the most efficient system assumed (85% generator efficiency, 95% applicator efficiency). In the 150 MLPY, this translates to needing 31, 13, and three ovens more under microwave, boost, and finish drying, respectively.

Row e of Tables 7.1 and 7.2 show the estimated unit costs of drying under each scenario for the 25 and 150 MLPY plants, respectively. Depending on the plant size and drying system efficiency level, drying costs in CAN\$ per tonne of DDGS were 77.99 - 101.16, 90.10 - 134.13, 132.68 – 219.31, and 215.75 – 394.80 under rotary, finish, booster, and microwave drying scenarios, respectively.

Figure 7.6 presents the contribution of each cost component to the total drying cost under each scenario. Although all scenarios required substantial equipment investment, the equivalent



Drying system and drying system efficiency



Drying system and drying system efficiency

Fig. 7.6 Cost distribution of drying wheat distillers grain with solubles (DDGS) from 70% to 10% moisture (wb) using rotary, microwave, booster, and finish drying systems in (a) **25 MLPY** and (b) **150 MLPY** ethanol plants. Microwave-based systems were evaluated at three efficiency (low, medium, high) levels. Rotary dryer at 50% thermal efficiency and 85% combustion efficiency.

depreciation costs only accounted about 13-27% of the drying costs. In rotary drying, depreciation is about 21-27% of the total cost, 13-18% in microwave drying, 14-22% in booster drying, and 15-25% in finish drying.

The bulk of the estimated drying costs came from electricity and natural gas consumption. On a per kilowatt-hour basis, assumed electricity cost was about CAN\$ 0.082 while that of natural gas was about CAN\$ 0.013. Cost of electricity per kWh was about 5 times higher than that of natural gas, thus, its increased use during drying would lead to higher total drying cost. In microwave drying, electricity costs ranged from CAN\$ 134.33 to 289.07 per tonne of DDGS and comprised about 61% to 73% of the total drying cost. Lowering electricity cost, through lower power rates, use of cheaper alternative energy sources and availability of even more efficient microwave drying systems, could substantially improve the cost of producing microwave-dried DDGS. In contrast, the costs of natural gas consumption during rotary drying were much lower compared to the cost of electricity under microwave drying. The cost of natural gas consumption ranged from CAN\$ 34.89 to CAN\$ 52.33 per tonne of DDGS and accounted about 42 – 55% of the total drying costs. In boost drying, the unit costs of electricity and natural gas consumption were CAN\$ 54.17 - 113.10 and CAN\$ 24.64 – 36.97 per tonne DDGS, respectively. Both collectively accounted 56-71% of the total drying costs. In finish drying, electricity costs (CAN\$ 17.27 – 33.79 per tonne DDGS) were lower and natural gas consumption costs were slightly higher (CAN\$ 32.33 – 48.49 per tonne DDGS) than those obtained in boost drying.

7.4.3 Comparison between drying costs and market prices

Figure 7.7 compares the estimated drying costs under the various drying scenarios and drying system efficiencies of the 25 MLPY plant with the 2011-2013 average weekly market prices per tonne of DDGS.

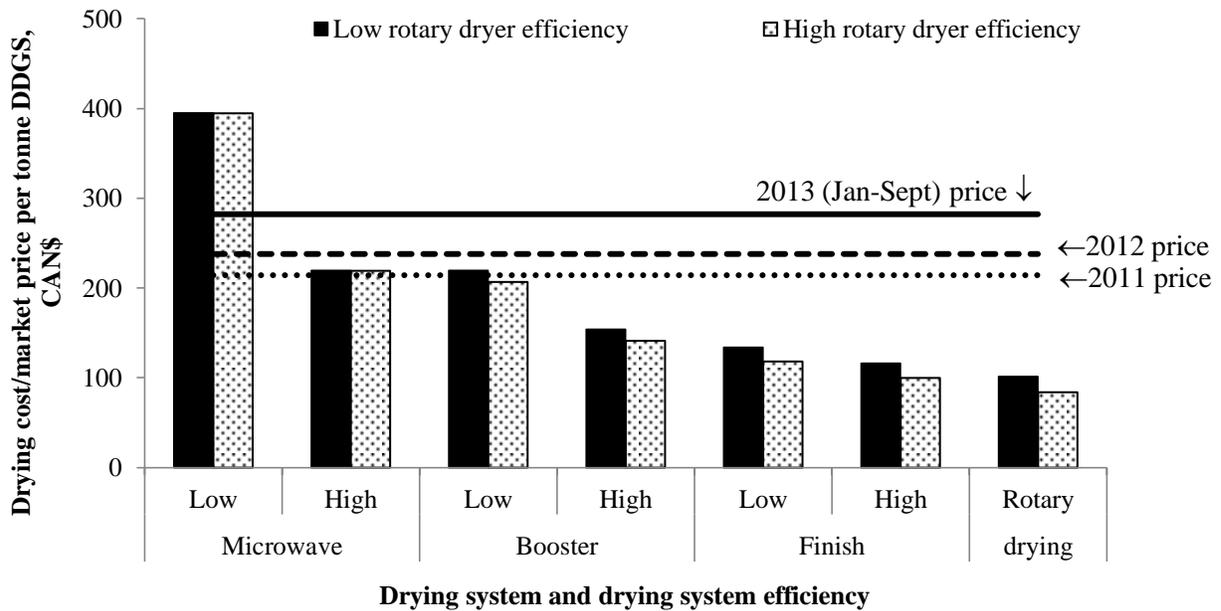


Fig. 7.7 Comparison between estimated drying costs under various scenarios and historical weekly market prices of wheat distillers dried grain with solubles. (Source of market prices: Agriculture and Agri-Food Canada (2013))

Rotary drying, the base case scenario, had the lowest estimated drying cost per tonne of wheat DDGS. The estimated cost of microwave drying under a low drying system efficiency was 40-82% higher than the historical average weekly prices. Under a high drying system efficiency, the cost of microwave drying was about the same as the 2011 average weekly price. The same can be said for booster drying, particularly under a low efficiency microwave drying system. These results indicate that, under the given set of assumptions used in the evaluation, full use of

microwave drying at the assumed microwave generator (85%) and applicator (95%) efficiency levels is not yet an economically viable option for wheat WDGS. Adding material cost and the costs associated with the other wheat DDGS processes, such as centrifugation, evaporation, and blending, may result to an overall wheat DDGS production cost that is higher than the average selling price. This would leave no or very little room for profit.

Microwave finish drying was seen as the most economically viable option among the three alternative scenarios. Its costs were 37-59% lower than the historical market prices and were the lowest among the three scenarios. It also showed the lowest increase in cost relative to rotary drying. Lower costs were attributed to lower electricity costs per tonne of wheat DDGS since microwave energy was applied only towards the end of the drying process, thus resulting to lower electrical energy requirement compared to the levels observed in microwave and booster drying. Estimated electricity ranged between CAN\$ 18.48 and 33.79 and accounted about 18-29% of the total finish drying cost in a 25 MLPY plant. This alternative drying scenario, however, need to be further assessed since the evaluation did not consider material cost and the costs of other wheat DDGS production processes. Incorporating these additional costs would enable a more comprehensive financial analysis of the costs and benefits of using microwave energy in drying wheat WDGS.

7.5 Conclusions

A techno-economic evaluation was conducted to assess the viability of incorporating microwave technology for drying wheat-based wet distillers grain with solubles (WDGS) in Saskatchewan ethanol plants. Three alternative scenarios (microwave, booster, and finish drying) were compared with the conventional hot air drying process (rotary drying). Two (low, high) and three

efficiency (low, medium, high) levels were assumed for the rotary and microwave drying systems, respectively. Two ethanol plants, with 25 and 150 million liters of ethanol per year nameplate capacities, were used as models in the evaluation.

Use of microwave energy substantially reduced the energy requirement of the drying process. Under the given set of assumptions, it cannot completely replace the conventional hot air drying process yet because of high electricity cost. Estimated microwave drying costs were close to or higher than the historical market prices per tonne of wheat distillers dried grain with solubles (DDGS). Finish drying, which uses the least amount of electricity among the three scenarios, was seen as the more economically viable option of incorporating the use of microwave energy in an ethanol plant. A more comprehensive assessment of wheat DDGS production cost, however, is needed to provide a better estimate of the potential earnings under this scenario.

Chapter 8

A case study application: Drying and physico-chemical characterization of brewers spent grain for productivity improvement of Philippine smallholder animal production

A similar version of this chapter will be submitted to a Philippine peer-reviewed journal focusing on agricultural extension and outreach.

Contribution of the PhD Candidate

Reconnaissance and mobilization activities, rapid assessment, physico-chemical characterization, and testing of the drying system, data analysis, and manuscript preparation were performed by the PhD candidate. She sought the cooperation of the agriculture and local government agriculture and veterinary officers, a village official and his staff, and officers of a federation of dairy cooperatives in conducting a rapid baseline assessment in El Salvador City, Misamis Oriental, Philippines. She partnered with the Agricultural Engineering faculty members (Mark Alexis Sabines and Fred Ynion) of Xavier University College of Agriculture (XUCA), her host institution in Philippines, in the design and development of a prototype batch dryer. She was assisted by several individuals (undergraduate student assistants, XUCA staff, and her relatives) during the dryer evaluation and physico-chemical characterization phases of the study.

Contribution of the Paper to the Overall Study

This paper addressed the secondary objective of the PhD research project, which sought to extend the knowledge and skills acquired in the PhD program in addressing a problem situation

in a developing country context. This auxiliary project provided the candidate a first-hand glimpse of the complex dynamics involved in doing research work involving local government units, small farmers, and industry in the Philippines, and of the challenges of implementing projects in an environment with very limited research facilities.

There is paucity of information about the supply and utilization of brewers spent grain in the Philippines. Results of this study can be used by local government units and by the Department of Agriculture – Northern Mindanao in providing more information about this bio-resource to smallholder animal producers, particularly those located near the brewery, and in developing programs that could assist resource-poor farmers in accessing and utilizing cheaper animal feed alternatives. These can also be used by animal feed processors, farmers’ cooperatives, and other parties interested in drying this brewery by-product and other similar processing wastes. These can also provide helpful information to the local brewery in crafting social responsibility programs that can benefit smallholder animal farms located near their facility. The results of the study may be helpful to local researchers interested in further exploring the feed and food potential of this bio-resource.

8.1 Abstract

Rapid assessment of supply and utilization of brewers spent grain, physico-chemical characterization of brewers spent grain (BSG), and development and evaluation of a prototype batch dryer were conducted in Misamis Oriental, Philippines. These were aimed toward providing more information about local BSG production and utilization, enhancing the efficiency and safety of its utilization as an animal feed ingredient, and broadening the animal feed resource

base of smallholder animal farms, thereby improving their productivity and revenue generation potential.

Brewers spent grain produced in Misamis Oriental, Philippines was disposed, transported, stored, and utilized in its wet form. Drying was rarely practiced. A solar energy- or biomass-powered batch dryer was designed and fabricated. Initial tests showed that it can reduce the moisture content from 71% to 10%, wet basis, in about 2 h, using a biomass furnace at an initial sample weight of 10 kg. Solar drying, however, took about 5.5 - 6 h and may not be practical to use under the present design. Basic physical attributes, proximate composition, and moisture sorption characteristics of the dried BSG were also assessed. Proposed measures to improve the performance of the prototype dryer and to encourage the use of drying and other spoilage prevention methods were also presented.

8.2 Introduction

Low productivity continues to characterize Philippine smallholder animal production (FAO 199; Villar et al. 2002; Chang 2007), with inadequate animal nutrition identified as one of the common constraints (FAO 1999). Because of resource constraints, animals are typically fed with sub-maintenance or maintenance diets (FAO 1999). Many smallholder swine producers, for example, incorporate kitchen refuse in combination with commercial feeds and other available feed ingredients (Villar et al. 2002). Backyard native chickens are often raised under the scavenging system, relying mainly on “naturally occurring feeds (grasses, insects, worms, and other edible plants and animals), fallen grains, and household refuse” (Lambio 2005 in Chang 2007). Livestock typically graze and scavenge on common grassy land and/or fed with crop residues from the farm (FAO 1999). While these feeding systems do promote low resource input,

these, however, translate to low animal productivity, lower market prices (Villar et al. 2002; Chang 2007), and lower income for the smallholder producers, ultimately affecting their capacity to provide for the needs of their families.

Aside from enhancing their means of livelihood, improving the animal productivity in smallholder farms is also essential to the overall competitiveness of the Philippine animal industry. Figure 8.1 shows that the Philippine animal industry is largely comprised of smallholder producers, which accounted about 94%, 99%, 70%, and 49% of the total cattle, goat, swine, and chicken production volume in 2008-2011 (Bureau of Agricultural Statistics 2012a; 2012b). A smallholder or backyard farm, in the Philippines, is defined as any farm which does not meet the definition of a commercial farm (Table 8.1). The livestock and poultry sub-sectors accounted an average of 23.6% of the gross value-added in agriculture, hunting, forestry and fishing in 2008 – 2011, based on 2000 constant prices (Bureau of Agricultural Statistics 2012c).

Table 8.1 Definition of commercial livestock and poultry farms in the Philippines

A commercial farm is any farm which satisfies at least one of the following conditions:

Livestock:

at least 21 heads of adults and zero young

at least 41 heads of young animals

at least 10 heads of adults and 22 young

Poultry:

at least 500 layers or 1,000 broilers

at least 100 layers and 100 broilers if raised in combination

at least 100 head duck regardless of age

Source: Bureau of Agricultural Statistics (2012d)

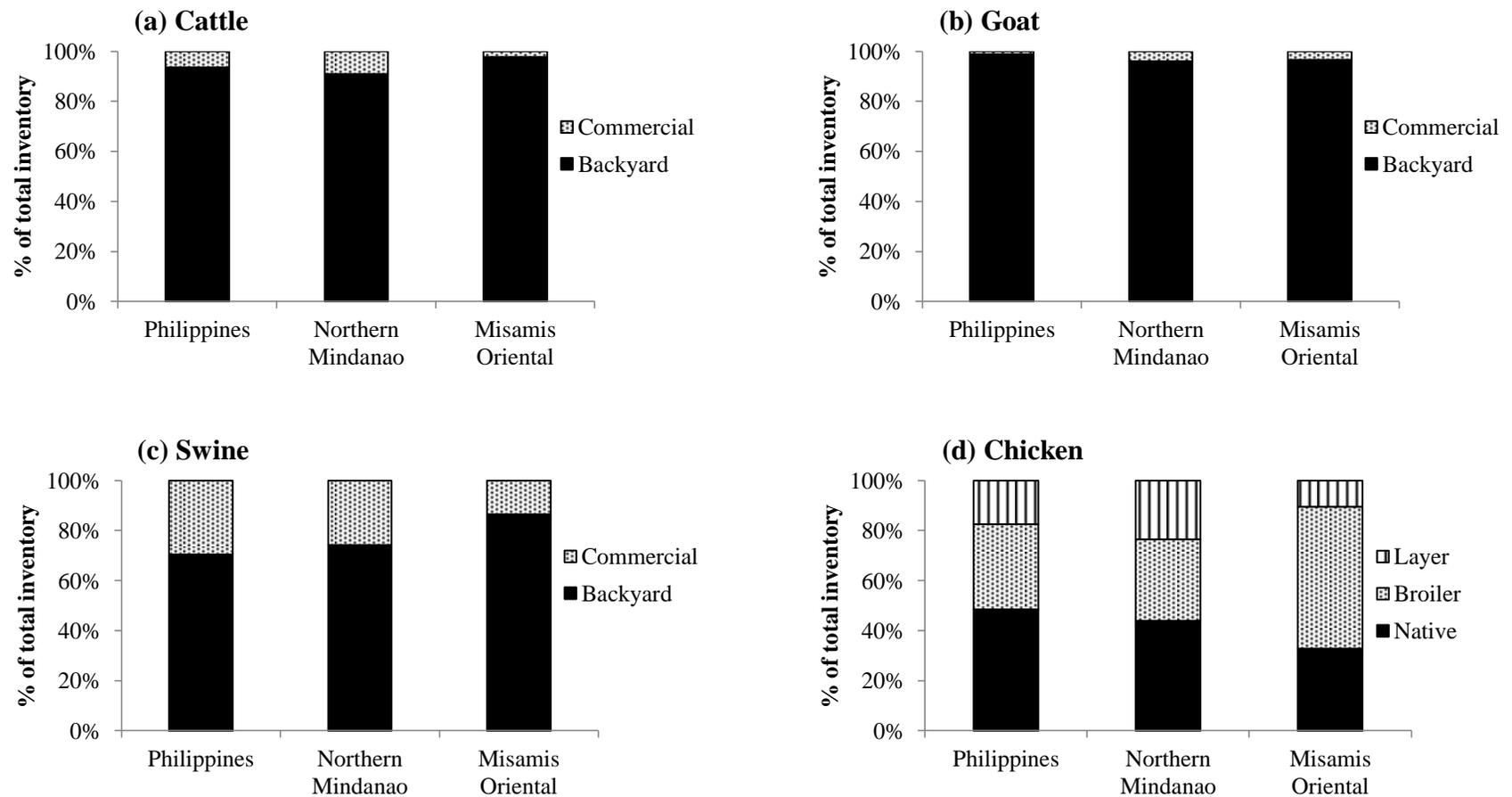


Fig. 8.1 Livestock and poultry production breakdown, by farm type, in the Philippines, Northern Mindanao, and in Misamis Oriental, 2008 – 2011. (Source: Bureau of Agricultural Statistics 2012a; 2012b)

Moreover, the Philippine feed milling sector is a net importer of prepared feeds and feed ingredients (Agriculture and Agri-Food Canada 2009). Figure 8.2 shows that over the past 17 years, there was an increasing trend in the importation of feeding stuff for animals. Price volatility of local raw materials and fluctuations in supply of local raw materials, in the world prices of feed ingredients and in foreign exchange rates are some of the challenges that confront the feed milling industry (Livestock Development Council in Cardenas et al. 2005; Villar et al. 2002). These challenges contribute to high feed production costs and highlight the need for alternative feed sources that can partially or wholly substitute the more expensive, imported feed ingredients. While results from numerous investigations focusing on the potential of locally available feed sources had been promising, most of these alternatives were not commercialized because of quality and supply challenges (Villar et al. 2002).

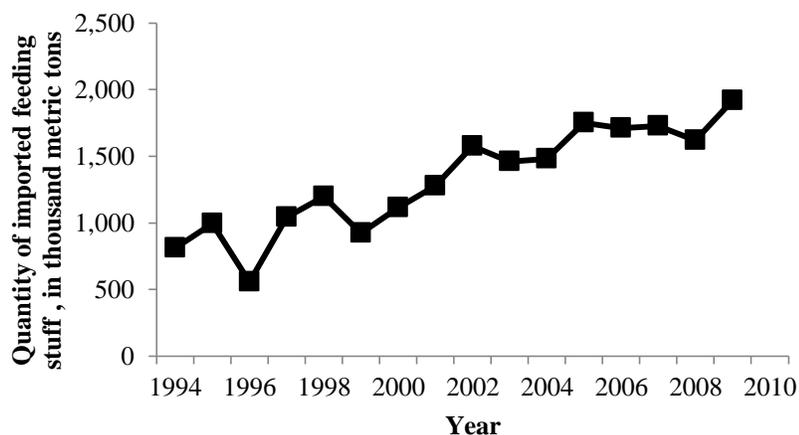


Fig. 8.2 Quantity of imported feeding stuff for animals (excluding unmilled cereals) in the Philippines, 1994-2009 (Source: Bureau of Agricultural Statistics 2011)

Brewers spent grain (BSG), a by-product of the brewing industry, for example, had been extensively studied for its use as an alternative animal feed ingredient. It is cheap, available all

year round, and has high nutritional value (Mussatto et al. 2006). Supply, storage, and transport challenges may have impeded its full utilization. Because of its high moisture and high nutrient content, it can deteriorate rapidly (Mussatto et al. 2006). To extend its shelf life, some cattle producers in northern Philippines incorporated salt to the spent grain but they indicated that the material has to be used within 4 days after delivery (Mitra 2001). Transporting wet BSG would also be expensive because of its low bulk density (Mussatto et al. 2006).

Drying can be a potential means to improve the storage and handling of BSG. Aside from improving its shelf life and lowering the storage volume, drying the BSG could enable users to further explore its incorporation in the rations of other animals. Drying BSG typically involved a two-step process: (a) pressing, where the moisture is reduced to less than 60% (wb), and (b) drying, to further reduce the moisture to 10% (w.b.) or less (El-Shafey et al. 2004; Santos et al. 2003). Although hot-air rotary drum dryers are typically used, use of freeze-drying (Bartolome et al. 2002) and superheated steam (Tang et al. 2005; Stroem et al. 2009) had also been investigated.

The Philippines has seven breweries with a total beer production of 2.1 million cubic meters in 2010 (Euromonitor International 2011). This is equivalent to about 422,000 t of BSG, assuming that about 200 kg of BSG would be generated for every cubic meter of beer produced (Mussatto et al. 2006). This volume, however, may be too small for independent feed mills (not affiliated with a brewery) to further exploit its utilization. It may be uneconomical and too cumbersome for feed mills to procure, transport, dry, store, and process wet spent grain generated from several breweries located in the archipelago. Further, BSG availability is dependent on beer production, thus, its supply may not also be as consistent as the other feed ingredients used by commercial

feed mills. Thus, smallholder animal production farms situated near breweries, would be the most ideal users of BSG.

There is, however, very limited published information about the characteristics of locally generated BSG, its supply, and the extent of its utilization by smallholder farmers in the country. Since BSG could be one of the cheaper alternative feed sources that smallholder producers can utilize, this study, then, aimed to address this gap by (i) conducting a rapid assessment to understand and obtain an overview of the supply and utilization of brewers spent grain in cattle value chains; (ii) determining the physico-chemical characteristics of locally dried brewers spent grain; and (iii) developing and evaluating a solar- and biomass-powered drying system for brewers spent grain that smallholder producers can collectively use.

8.3 Materials and Methods

The study has three parts: (i) rapid baseline assessment on the supply and utilization of brewers spent grain and smallholder animal production and marketing; (ii) physico-chemical characterization of dried brewers spent grain; and (iii) development and evaluation of a brewers spent grain dryer. This report focused only in assessing brewers spent grain utilization within the cattle farms. Preliminary reconnaissance indicated these farms are among the regular users of brewers spent grain.

The study location was Misamis Oriental (Fig. 8.3), a province which hosts one of the two breweries in Mindanao. It is part of Northern Mindanao, a region that ranked first, third and fifth in the cattle, chicken, and swine production volume in 2012, respectively (Bureau of Agricultural Statistics 2013a; 2013b).

8.3.1 Assessment of brewers spent grain supply and utilization in smallholder animal farms

To gain a quick preliminary understanding of the local situation, interviews with selected key informants, and visits to livestock farms, livestock auction market, and dairy processing plant were conducted in El Salvador City and surrounding areas, Misamis Oriental. Key informants included: (i) a brewery personnel, in charge of coordinating with brewers spent grain buyers; (ii) seven smallholder dairy cattle farmers who are members of a federation of dairy cooperatives; (iii) a veterinarian-commercial livestock farm owner; (iv) a dairy processing plant manager; and (v) an official and his staff of an El Salvador City *barangay* (village) that operates the weekly livestock auction market. Interview guide questions used are presented in Appendix C.1.

8.3.2 Physico-chemical characterization

Brewers spent grain samples obtained from the brewery were packed in plastic bags and stored in a freezer. Bags of samples were thawed overnight at room temperature (28-29°C) before the drying runs.

8.3.2.1 *Thin layer drying characteristics*

One hundred gram samples were spread thinly on cloth-lined wire trays (0.19 m x 0.25 m) and dried using a cabinet solar dryer (Fig. 8.4a and 8.4b) and a biomass-fueled, flatbed dryer (Fig. 8.4c and 8.4d), with drying temperature ranging between 50°C to 60°C.

Sample mass during drying was regularly monitored until 10% moisture (wb) was reached. Two runs were conducted under each drying method. Drying data were converted into moisture ratio values and were fitted into four thin layer drying models (Table 8.2) using IBM SPSS Statistics

20.0 (IBM Corp., Armonk, NY). Best fit model was selected using coefficient of determination (R^2) and mean square error (MSE) as criteria. Analyses were conducted at the 0.05 significance level.



Fig. 8.4 Thin layer drying of brewers spent grain using a cabinet solar dryer (a,b) and a biomass-fired flat-bed drying system (c,d)

Table 8.2 Thin layer drying models used in fitting brewers spent grain drying data

Model	Equations*
Henderson and Pabis	$MR = g \cdot \exp(-k \cdot t)$
Newton	$MR = \exp(-k \cdot t)$
Logarithmic	$MR = g \cdot \exp(-k \cdot t) + h$
Page	$MR = \exp(-k \cdot t^n)$

* MR means moisture ratio, k means drying rate in min^{-1} , t is drying time in min, and the variables g, h, and n represent the model parameters

8.3.2.2 *Proximate composition*

Samples from two production batches were obtained from the brewery and were sun-dried for proximate composition analysis. Determination of crude protein (AOAC Official Method 984.13), crude ash (AOAC Official Method 942.05), crude fat (AOAC Official Method 920.39), and crude fiber (AOAC Official Method 962.09) content was outsourced due to equipment limitation in the host institution. Acid detergent insoluble crude protein (AOAC Official Methods 973.18 and 984.13) content of samples from two drying runs using the second batch of sample material was compared with the sun-dried sample. Moisture content was determined using AOAC Official Method 920.36 (AOAC 2003a).

8.3.2.3 *Physical attributes*

Bulk density, particle density, particle size and size distribution, and color of dried BSG were determined. Dried samples were produced using the prototype batch dryer presented in section 8.3.3 of this report.

Bulk density. Bulk density was estimated by pouring 20 g of dried brewers spent grain through a funnel into a 100 mL graduated cylinder. Values obtained were later verified using the bulk density apparatus at the University of Saskatchewan.

Particle density. Particle density was determined using a gas multipycnometer (QuantaChrome, Boynton Beach, FL). Bulk porosity (ε), expressed as a percentage, was determined as a function of bulk (ρ_b) and particle densities (ρ_p) using Eq. 8.1 below.

$$\varepsilon = 1 - \frac{\rho_b}{\rho_p} \quad (8.1)$$

Particle size and size distribution. Sieve sizes of 4, 6, 8, 10, 16, 25, 30, 40, 50, 60, 80, 100, 140, and 200 and a sieve shaker were used for particle size analysis, following ANSI/ASAE S319.4 (ASABE 2008). The calculated geometric mean diameter was used to represent particle size.

Color. Samples for color determination were also sent to an external laboratory. Color was determined using a Konica Minolta Color Reader 10 (Konica Minolta, Inc., Osaka Japan) and reported in terms of the CIE L*a*b* color space. These color parameters, L*, a*, and b*, indicate the level of lightness, redness, and yellowness, respectively.

8.3.2.4 *Moisture sorption characteristics*

Moisture sorption behavior of brewers spent grain was determined using the static gravimetric method. Airtight storage chambers were prepared by using a 152 mm polyvinyl chloride (PVC) pipe and a 4.76 mm thick acrylic glass (Fig. 8.5). Varying concentrations of sulfuric acid solutions (Mok and Dick 1991) were used to achieve and maintain four relative humidity (RH) levels at ambient temperature (28°C).



Fig. 8.5 Storage chambers used in determining moisture sorption characteristics of brewers spent grain.

Two grams of solar-dried brewers spent grain were placed in each chamber, immediately above the beaker containing the sulfuric acid solution. Mass changes were monitored regularly until

equilibrium was reached. Temperature and relative humidity inside the chambers were monitored using a TPI 597 digital psychrometer (Test Products International, Inc., Ontario, Canada). Initial and final moisture content of the samples was determined using AOAC Official Method 920.36 (AOAC 2003). Two replicates for each RH level were set up. Experimental data were fitted to the moisture sorption models presented in Table 8.3 and the best fit model was selected using the same criteria and methods outlined for drying characteristics.

Table 8.3 Moisture sorption models used in fitting brewers spent grain data at 28°C

Model name	Equation*
Henderson	$M = \left[\frac{\ln(1-RH)}{-F} \right]^{1/G}$
Oswin	$M = (F) \left[\frac{RH}{1-RH} \right]^{1/G}$
Modified Henderson	$M = \left[\frac{\ln(1-RH)}{-F(T_C+G)} \right]^{1/H}$
Modified Chung-Pfost	$M = \left(\frac{-1}{H} \right) \ln \left(\frac{\ln(RH)(T_C+G)}{-F} \right)$
Modified Oswin	$M = (F+GT_C) \left[\frac{RH}{1-RH} \right]^{1/H}$
Modified Halsey	$M = \left[\frac{-\exp(F+GT_C)}{\ln(RH)} \right]^{1/H}$

Source: Al-muhtaseb et al. (2002); Kingsly and Ileleji (2009)

* M, T_C, and RH represent equilibrium moisture content (% db), temperature (°C), and equilibrium relative humidity (decimal), respectively. F, G, H are the model parameters.

8.3.3 Development and evaluation of a brewers spent grain prototype drying system

A prototype batch drying system for brewers spent grain was designed and developed in collaboration with the Agricultural Engineering faculty members of Xavier University College of Agriculture. Design was based on baseline assessment results and preliminary drying runs. Construction of the drying system was contracted to external service providers. Appendix C.2 shows the details of the drying chamber and the heating system.

8.3.3.1 System description

The drying system developed in this study comprised of a drying chamber, which has a rotating wheel and a side hopper, and a heating system (Fig. 8.6). The feed material is loaded into the drying chamber through the side hopper. Inside the chamber, the material is moved using a rotating wheel (estimated speed of 28 rpm), facilitating greater particle contact with the heated air. The bottom of the drying chamber can be opened to unload the dried material. Details of the drying chamber and the heating system are presented Appendix C.2 (Figs. C.2.1 and C.2.2).



(a)



(b)

Fig. 8.6 A prototype batch drying system for brewers spent grain, operated using (a) solar or (b) biomass energy.

A 200 mm portable propeller blower (Torq SHT-20 model, China) pushes air from heating system to the drying chamber. A 0.20 m PVC-coated ventilation duct connects the blower to the heating system and the heating system to the drying chamber. Air is heated using either the solar heat collector (Fig. 8.7a) or biomass furnace (Fig. 8.7b). The solar heat collector, 0.41 m x 0.90 m x 2.50 m long, is comprised of a glass top cover, a metal frame, and 3-layer walls and floor made of plain GI sheet, insulating foam, and wood. Six layers of corrugated galvanized iron roofing sheets, painted black, were placed over its depth. The walls and floor of the biomass-fueled furnace, on the other hand, were comprised of bricks (interior) and mild steel plate (exterior).

Exhaust air exits through the vents located on top of the drying chamber, just above the section where heated air enters (Fig. C.2.1c). Location of the air vents was primarily chosen to maximize contact of heated air with the bulk material. When heated air reaches the end of the chamber, it is forced to turn back and is made to go through the length of the chamber again before exiting through the air vents, facilitating greater contact with the material to be dried.

8.3.3.2 *Dryer performance evaluation*

Preliminary drying runs using the first batch of brewers spent grain obtained from the brewery to identify necessary design modifications. Dryer performance was evaluated in Manresa Farm, Xavier University College of Agriculture after the needed adjustments were made. Two drying runs were conducted under each method during the evaluation.

Ten kilograms of wet brewers spent grain were used for each drying run. Samples were collected every 15 min from two specific locations in the dryer to monitor moisture content reduction. Target final moisture content was 10%, w.b. (wet basis). Moisture content was determined using

AOAC Official Method 920.36 (AOAC 2003a). Amount of biomass fuel used, ambient, drying, and exhaust air conditions were monitored. Biomass used for fuel was mainly sourced from Manresa Farm and included tree trimming wastes, storm-felled tree branches, coconut husk, and some wood chips from tomato box construction.

8.4 Results and Discussion

8.4.1 Dairy cattle value chain overview

Figure 8.7 shows an overview of the interaction between selected dairy cattle farms in Misamis Oriental, brewery, and other actors in the value chain.

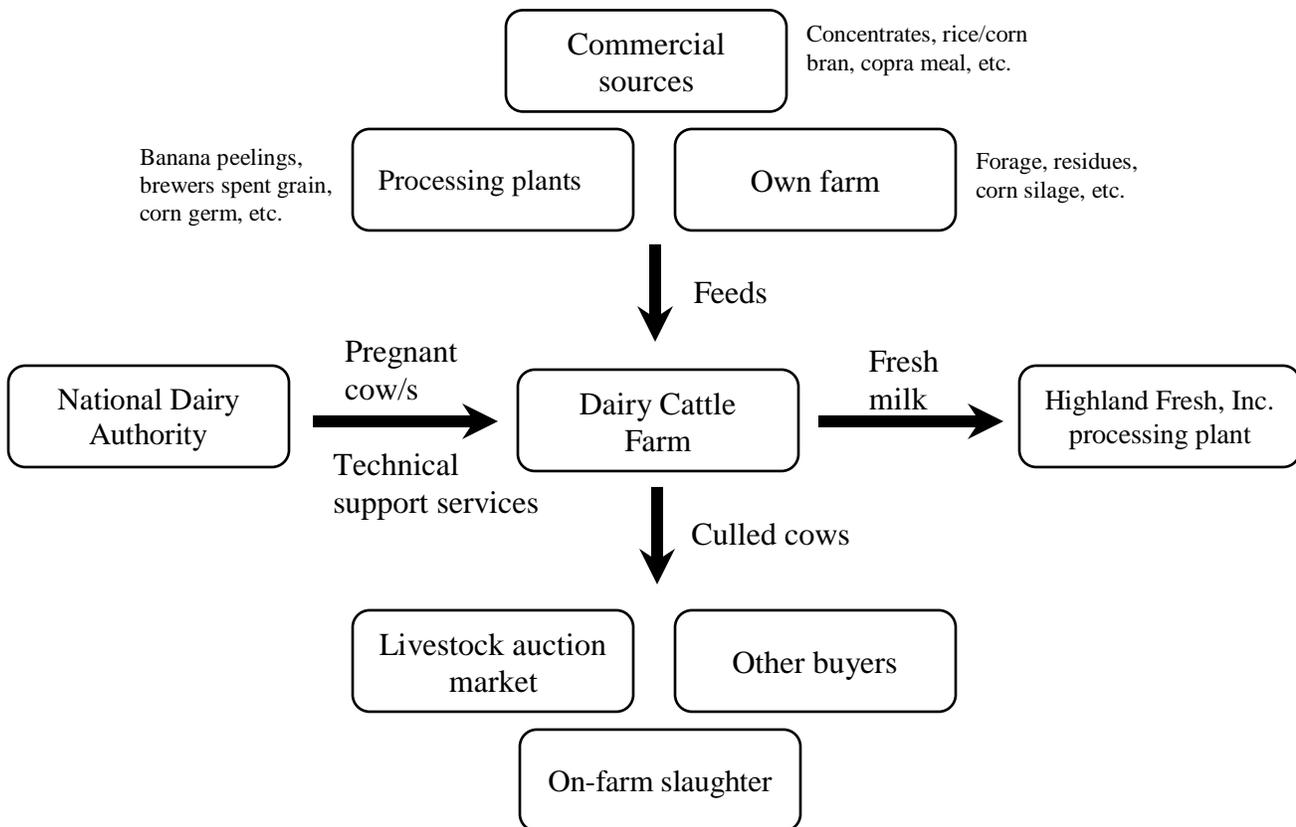


Fig. 8.7 An overview of the interactions between selected dairy cattle farms, the brewery, and other value chain actors in Misamis Oriental, Philippines, Aug – Sept 2012.

Sources of dairy cattle and technical support services. The farmers obtained their cattle through the dispersal program of the National Dairy Authority (NDA), an agency attached to the Department of Agriculture and “mandated to ensure the development of the Philippine dairy industry” (NDA 2012). They need to satisfy certain requirements before they can avail animals under this program. For their stock, the farmers pay NDA in kind. For every imported pregnant cow obtained, the farmers pay NDA two pregnant cows within seven years from the day the stock was acquired. If the cows are of local breed, the farmers pay three cows for every two cows that they would acquire from NDA. The agency also provides technical support services to the farmer-beneficiaries.

Feed and feed sources. Compared to the other backyard farmers (Mosqueda 2014), the dairy cattle backyard farmers have a more diverse feed resource base: (i) processing plant wastes (brewers spent grain, banana peelings, corn husks), (ii) their own and/or neighboring farms (grasses, corn silage, forage crops like *Gliricidia sepium* and *Leucaena leucocephala*, rice straw, corn stover, banana trunks, etc.), and (iii) animal feed suppliers (dairy concentrates, copra meal, rice and corn bran, molasses, salt).

Sale of raw milk and culled cows. Milk produced by the member-farms is delivered to a dairy processing plant in El Salvador City or to its designated collection centers in various areas of Misamis Oriental, based on an agreed schedule. The dairy processing plant is owned and managed by a federation of cooperatives. The plant buys milk from both members and non-members at PHP 24.00 (CAN\$ 0.60) per L and at PHP 22.00 (CAN\$ 0.55) per L, respectively, depending on the quality. Farmer-members can also engage in milk product retail distribution.

Culled cows are either sold or slaughtered in the farm for sale as meat. Most of the dairy farmers interviewed sell their culled cows to the livestock auction market in El Salvador City, while a few sell to selected business enterprises in neighboring Cagayan de Oro City (steakhouses, butcheries) and other regular buyers. Overview of the livestock auction market is presented in Appendix C.3.

8.4.2 Brewers spent grain supply and utilization

8.4.2.1 *Brewers spent grain supply*

The only source of brewers spent grain in Northern Mindanao is the Asia Brewery, Inc. plant located in *barangay* (village) Sinaloc, El Salvador City, about 20 km from Cagayan de Oro City, Misamis Oriental. Established in 1992 and considered as Mindanao's first brewery, it is rated to produce about two million hectoliters of beer per year (Asia Brewery, Inc. 2013), with about 40,000 t of brewers spent grain co-produced annually. Interviews with a brewery personnel, however, indicated that average production is about 25 – 35 brews every two weeks. One brew produces about 4 – 6 t of brewers spent grain. Peak production months are October, December, January, March, and May, wherein as much as 32 to 34 brews could be done per week. During lean months, about 15 – 20 brews could be done every two weeks.

Wet spent grain is sold by the brewery to registered buyers only, based on a schedule prepared by its materials division. Buyer registration requirements include a security deposit of 5,000.00 Philippine pesos (PHP), a vehicle (whether owned or rented) to receive and transport the spent grain from brewery, and an accomplished application form. Depending on their classification, buyers could purchase wet brewers spent grain at a minimum of one brew (4 – 6 t) per month to as much as four brews (16 – 24 t) every other week. Brewers spent grain is currently sold at PHP

0.50 per kilogram. Spent grain buyers come from various parts in Mindanao, including Misamis Oriental, Bukidnon, Agusan del Norte, Lanao del Sur, Lanao del Norte, and Zamboanga del Norte.

8.4.2.2 *Brewers spent grain utilization*

Hauling and storage. Provision of a vehicle, whether rented or owned, to haul the spent grain is one of the requirements when applying to become a registered spent grain buyer. The cattle farmers or their caretakers indicated that they spent between PHP 1000.00 – 1500.00 (CAN\$ 25.00 – 37.50) for fuel in transporting the spent grain to their farms. Only one of the eight farms rented a vehicle to haul the spent grain from the brewery as the rest have their own transportation.

Figure 8.8 shows the two ways of storing wet brewers spent grain employed in the farms visited. All of the dairy cattle farms utilized an open top concrete bin (locally referred to as a “silo”) to store spent grain (Fig. 8.8a and 8.8b). Most of these structures are either housed inside a shed or have makeshift roofs made of nipa palm leaves or plain/corrugated galvanized iron sheets. Only one farm stored its spent grain in a concrete bin without any form of covering to shield the material from the weather elements. One farmer indicated that they compress the spent grain



Fig. 8.8 Brewers spent grain storage methods used by dairy cattle farmers (a,b) and by a farm with beef cattle and small ruminants (c), Misamis Oriental, Philippines, Aug – Sept 2012.

layer by layer when storing to minimize mold development. The other farms did not observe this method of storage and withdrawal. Only the non-dairy livestock farm used plastic drums to store its spent grain (Fig. 8.8c).

Drying. Spent grain is commonly fed wet. Only one farm employed sun drying. This farm also used to mechanically dry its spent grain using a flat-bed dryer commonly used for grains but had stopped because of the lower quantities obtained from the brewery. One farmer indicated that they have not considered sun drying their spent grain because it is too labor intensive and they do not have the resources to pay for additional workers. Presence of molds and maggots was observed in the stored spent grain in one of the farms visited in this study.

Feeding practices. Spent grain is fed to the animals along with other kinds of feeds. One farm indicated feeding 90% spent grain and 10% legumes (such as the leaves of *Leucaena leucocephala*). Another farm estimated that it used 25% spent grain, 25% corn silage, 25% grass, and a farm-prepared mixture of copra meal, rice or corn bran, soybean meal, salt and molasses comprised the remaining 25%. In this farm, cows were also given ad libitum access to banana peelings, sweet corn husks, and rice straw. In another farm, spent grain comprised 60% of the feed while the remaining 40% comprised of copra meal, rice or corn bran, banana peelings, corn silage, and mineral supplement. In a farm with that had both dairy and beef cattle, spent grain was given only to lactating cows along with copra meal, corn germ, corn silage, and dairy concentrate. Three other farms fed their cows with spent grain, dairy concentrate, and grass while another farm used spent grain along with a variety of other biomass feed sources, such as napier grass, leaves of *Gliricidia sepium* and *Leucaena leucocephala*, the tender core of banana trunks, corn stalks, and banana peelings.

Perceived benefits and disadvantages. Lower feed cost and increased milk production were the commonly cited benefits derived from incorporating brewers spent grain into the dairy cattle ration. Odor and limited supply of brewers spent grain were among the disadvantages identified. One farm owner-veterinarian interviewed also indicated that use of wet spent grain stored for a long time can also cause listeriosis in goats. Sudden change of the feeding pattern, particularly when there is limited supply of spent grain, can also adversely affect body weight of the ruminants.

8.4.2.3 *BSG utilization and spoilage rates*

Storing the high moisture, high nutrient content BSG in open piles, such as those shown in Fig. 8.8, under warm and humid weather conditions would make it an excellent medium for microbial growth. Although there was no study found in Misamis Oriental focusing on local BSG storage and utilization, a study in Argentina reported the presence of a range of fungal species (*Aspergillus*, *Cladosporium*, *Penicillium*, *Fusarium*, *Rhizopus*, and *Alternaria* spp.) in the wet BSG samples studied that could pose a risk to animal health (Asurmendi et al. 2012). Westendorf and Wohlt (2002) also indicated that an uncovered pile of BSG could be stored less than five to seven days while Amaral-Phillips and Hemken (2006) mentioned that in the summer, wet BSG should be used within two to five days. The need to curb BSG spoilage would be particularly important in smallholder farms, where their BSG usage rate could be lower than the rate at which the material could spoil. In Misamis Oriental, the minimum quantity a BSG buyer could obtain from the brewery ranged from four to six tons, equivalent to the amount of residue generated from one brew. In a farm with six heads of mature dairy cows, for example, it would take at least a month to consume this amount, assuming that the wet BSG feeding rate would be limited to about 13.6 – 22.7 kg (30 – 50 lb) per cow per day (Thomas et al. 2010). This is longer

than the recommended storage life of wet BSG (Westendorf and Wohlt 2002; Amaral-Philips and Hemken 2006). Further studies need to be conducted to assess and minimize the risks associated with prolonged storage of wet BSG under local conditions.

8.4.3 Physico-chemical characteristics

8.4.3.1 Thin layer drying characteristics

Figure 8.9 shows the thin-layer drying behavior of brewers spent grain as moisture was reduced from 71.7% about 10%, wet basis (wb), under solar and mechanical methods. Mechanical drying took about 54 – 60 min while solar drying time was between 142 – 164 min. Drying behavior under both methods was best described by the logarithmic model (Eqs. 8.2 and 8.3), with the drying rate constant (model parameter k , Table 8.2) under mechanical drying being 4.5 times higher compared to the value obtained under solar drying. In Eqs. 8.2 and 8.3, MR is moisture ratio while t is drying time in min.

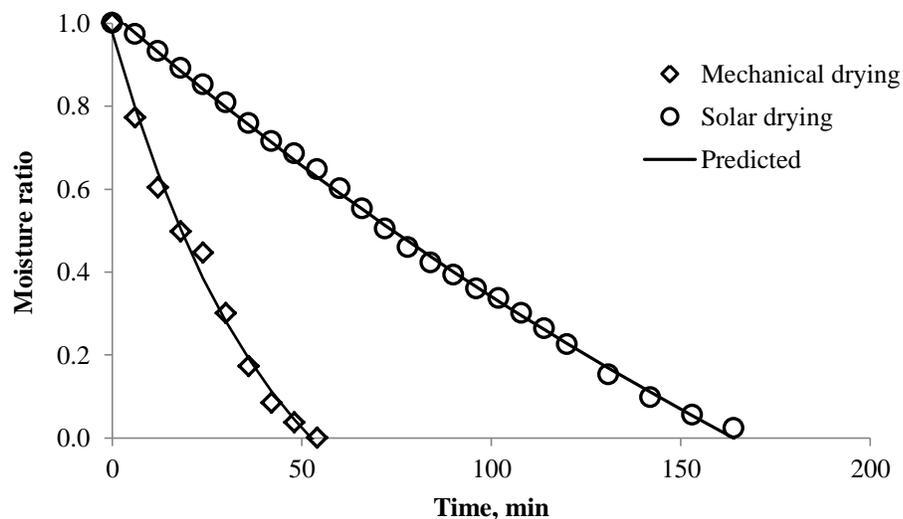


Fig. 8.9 Thin layer drying characteristics of brewers spent grain under solar and mechanical drying methods at a 50 – 60°C drying temperature range, August 2012.

$$\text{Solar drying (R}^2 = 0.998\text{):} \quad \text{MR} = 2.39 \exp^{-0.004t} - 1.36 \quad 8.2$$

$$\text{Mechanical drying (R}^2 = 0.992\text{):} \quad \text{MR} = 1.35 \exp^{-0.022t} - 0.36 \quad 8.3$$

8.4.3.2 Chemical composition

Table 8.4 shows the crude protein, crude fiber, fat, and ash content of the two batches of brewers spent grain samples obtained from the brewery. Differences were observed in the protein, fiber, fat, and ash content between the two batches. Although the causes of these differences were not investigated, differences in the raw materials used (malted barley, type of adjuncts) and processing conditions (such as the ratio of malted barley and the adjuncts used) could be two possible sources of variation. Distribution patterns of the chemical constituents between the two batches, however, were similar.

Table 8.4. Chemical composition (% dry matter) of brewers spent grain samples obtained from a Misamis Oriental brewery in comparison with results from other studies.

Component	This study		Santos et al. (2003)	Huige (2006)	Makowska et al. (2013)
	Batch 1	Batch 2			
Starch	37.1	-	-	-	4.56
Crude Protein	18.0	21.8	24.2	28.0	25.49
Crude Fiber	19.4	22.3	-	15.0	-
Crude Fat	9.9	7.6	3.9	7.2	8.53
Crude Ash	4.6	3.9	3.4	4.0	3.76
ADICP*					
Open sun-dried		9.8			
Drying run 1		10.8			
Drying run 2		14.2			

*Note: ADICP – acid detergent insoluble crude protein. Drying runs 1 and 2 were made using the prototype dryer. Open sun-drying

The compositional results from three studies were also presented for comparison. The crude protein results in this study were lower compared to those obtained by Huige (2006), Santos et

al. (2003), and Makowska et al. (2013) while ash content was close to those reported in the four studies. Huige (2006) and Santos et al. (2003) reported lower crude fiber and crude fat content, respectively, compared to the values obtained in this study. The fat content values in the study were close to those reported by Huige (2006) and Makowska et al. (2013). Differences in the compositional results between the studies are expected due also to differences in raw materials (variety of barley, type of adjuncts), processing conditions, and methods of proximate analysis employed.

Increase in acid detergent insoluble crude protein (ADICP) content of the samples dried using the prototype drying system (presented in section 8.3.3.1) was observed compared with the open sun-dried sample. ADICP content is used as a measure of protein damage. Increased ADICP could be attributed to Maillard reaction. Use of higher temperatures in the prototype dryer may have increased the rate of Maillard reaction, and thus increased protein damage incidence compared to open sun-drying.

8.4.3.3 *Physical attributes*

Table 8.5 shows the density, porosity, size, and color parameters of dried brewers spent grain. The samples were larger in size, less dense, and more porous compared to the commercial wheat DDGS samples presented in Table 6.1. Bulk density values were close to the values presented in the bulk density tables of Brabender Technologie (n.d.) and Tapco, Inc. (n.d.) for dry brewers spent grain (loose bulk density of $14 \text{ lb}\cdot\text{ft}^{-3}$ or $224 \text{ kg}\cdot\text{m}^{-3}$). Figure 8.10 shows that about 71% of the total amount sieved had particle sizes that were greater than 0.71 mm but less than the 2.00 mm sieve opening. Both dried samples showed similar particle size distribution curves.

The dried brewers spent grain samples were brown in color. In terms of their color parameters, the dried brewers spent grain samples were lighter, less red, but more yellow (Table 8.5) compared to the wheat DDGS samples (Table 6.1). The mean CIE Lab values presented in Table 8.5 were converted to the HunterLab scale before comparison was made with the Table 6.1 color parameter values.

Table 8.5 Some physical attributes of dried brewers spent grain, generated from two drying runs of the prototype batch dryer.

Property	Batch 2 sample	
	Drying run 1	Drying run 2
Moisture, % wet basis	9.31 (0.76)	8.60 (0.13)
Bulk density, kg·m ⁻³	221.14 (3.83)	215.43 (10.73)
Particle density, kg·m ⁻³	1281.27 (2.79)	1257.02 (4.67)
Porosity, %	82.7	82.9
Particle size, mm	1.11 (0.00)	1.12 (0.01)
L*	47.7 (1.4)	50.6 (1.9)
a*	8.9 (0.4)	8.3 (0.8)
b*	25.1 (0.5)	23.8 (0.3)

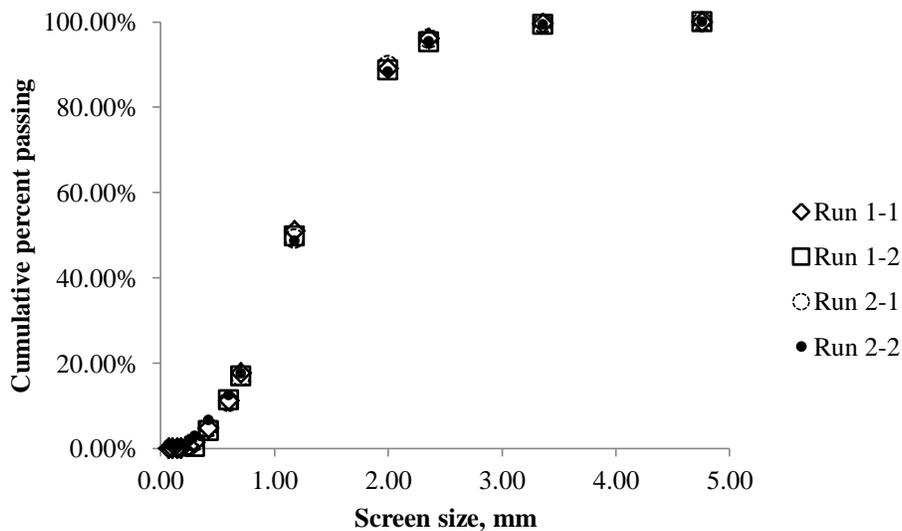


Fig. 8.10 Particle size distribution of dried brewers spent grain samples

8.4.3.4 Moisture sorption characteristics

Among the models presented in Table 8.3, the Oswin model (Eq. 8.4) best described the moisture sorption characteristics of dried brewer's spent grain at ambient temperature (28.7°C). Fig. 8.11 shows the observed and the predicted values of equilibrium moisture content (M) at varying equilibrium relative humidity (RH) levels. This information would be useful in determining the appropriate moisture content at which the dried BSG can be stored in a 28.7°C environment at a given relative humidity. This sorption isotherm closely resembled the Type III behavior (Brunauer et al. 1940), similar to what was observed in wheat DDGS (Chapter 6, p. 196).

$$M = 0.089 \left(\frac{RH}{1-RH} \right)^{\frac{1}{1.709}} \quad R^2 = 0.934 \quad 8.4$$

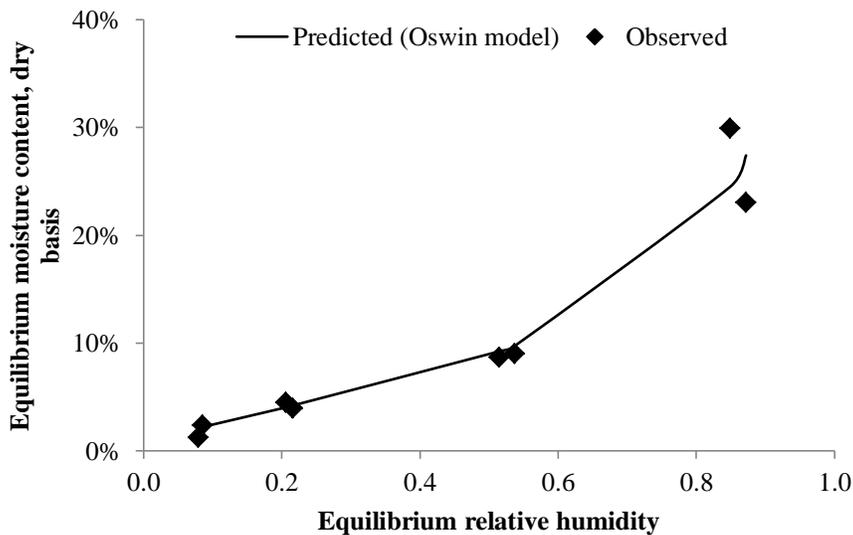


Fig. 8.11 Moisture sorption characteristics of brewers spent grain at 28.7°C.

8.4.4 Dryer performance evaluation

Initial moisture content of the wet brewers spent grain used during the drying evaluation runs averaged at 70.8% (wb). Table 8.6 shows the ambient conditions at the start and end of these runs.

Table 8.6 Ambient air temperature and relative humidity at the start and end of the each drying run.

Drying method	Run	Start of drying		End of drying	
		Relative humidity, %	Temperature, °C	Relative humidity, %	Temperature, °C
Mechanical drying (Jan 2013)	1	91.1	25.5	94.5	24.2
	2	79.6	28.7	90.1	26.2
Solar drying (Apr 2013)	1	49.5	35.0	53.6	35.2
	2	53.2	32.2	62.4	33.3

8.4.4.1 Mechanical drying

Figure 8.12 shows the moisture content reduction of brewers spent grain samples obtained from the two specific sections at the bottom of the prototype dryer. Reducing the moisture content from 70.8% (wb) to about 10% (wb) took between 1.75 and 2.00 h under the temperature ranges (85 – 95°C) indicated.

Figure 8.13 shows that it took between 5.5 to 6.0 hours for brewers spent grain to reach about 10% moisture. There is also a pronounced difference in the moisture content of samples obtained from the front and back sections of the prototype dryer. Samples taken from the back section had higher moisture compared to those obtained from the front section. While this difference was also observed under mechanical drying (Fig. 8.12), it was more pronounced under solar drying since the latter operated under much lower air temperature and higher relative humidity compared to the former. Under the present set up, solar drying may not be an attractive

option because of the longer drying time involved even during the hot and dry season (March – May).

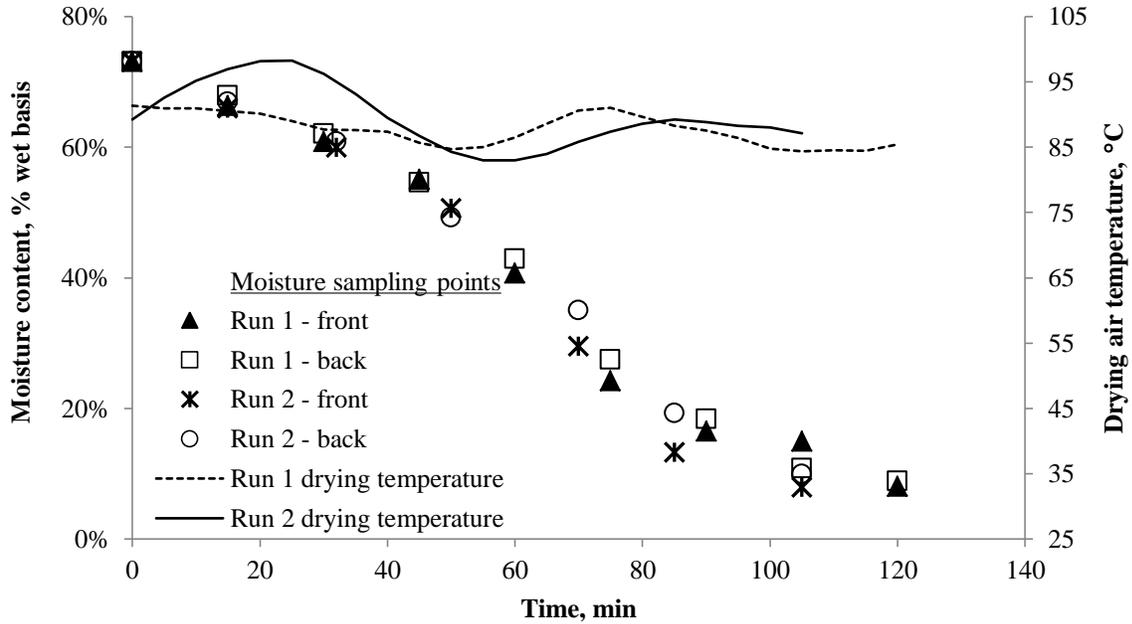


Fig. 8.12 Moisture content reduction and drying air temperature during mechanical drying of brewers spent grain. Samples were obtained from the bottom of the batch dryer’s front and back sections.

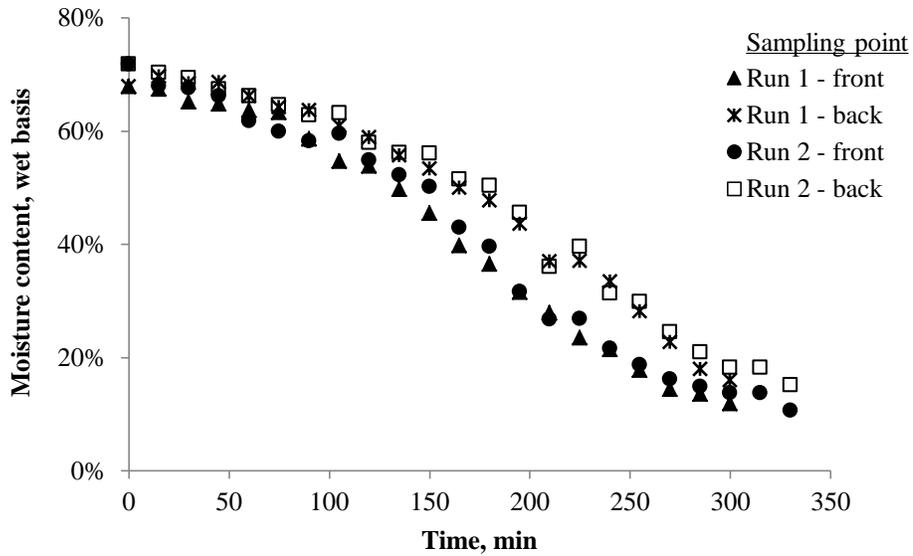


Fig. 8.13 Moisture content reduction during solar drying of brewers spent grain. Samples were obtained from the bottom of the prototype dryer’s front and back sections.

Figure 8.14 shows the temperature and relative humidity profile of ambient and heated air during the two solar drying runs. During these runs, the solar heat collector raised the air temperature by 42-46% and lowered the relative humidity by 48-50% from ambient levels. Heat loss occurred in the duct connecting the solar heat collector and the drying chamber. These improvements in the air temperature and relative humidity levels, however, were not enough to shorten the drying time.

8.4.4.2 Areas for improvement/Recommendations

While it has been shown that the batch dryer was able to reduce the moisture of brewers spent grain from 70.8% to about 10% (wb), there were a number of areas that need to be improved before this dryer (using the biomass furnace) can be field-tested in the villages. These include product yield, thermal efficiency, and ease of unloading. Addressing these would enable better estimation of the costs involved in drying BSG.

Low yield. A significant amount of the wet material accumulated on the blades and central shaft of the rotating wheel at the end of the drying process, which lowered the product yield. Average accumulation on the blades and shaft was estimated to range from 965 to 1700 g·m⁻², with moisture content varying from 63 to 65% (wb). Higher amount of accumulation (3480 g·m⁻²) was observed when the input quantity to be dried was tripled. While the accumulation on the wheel blades and shaft can be scraped with relative ease by hand, this would translate to longer downtimes and additional labor resource which smallholder producers may not find attractive. Investigations need to be conducted to better understand the sticking behavior of BSG vis-à-vis its material properties and drying system variables. In superheated steam drying of BSG, Stroem

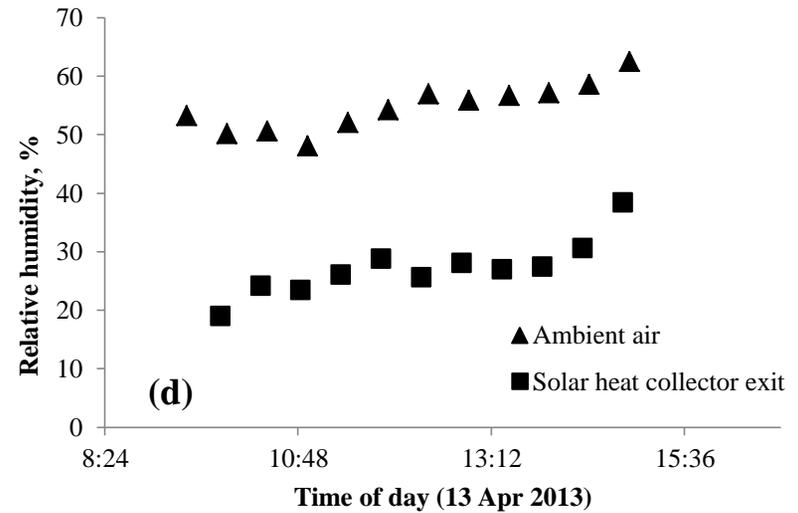
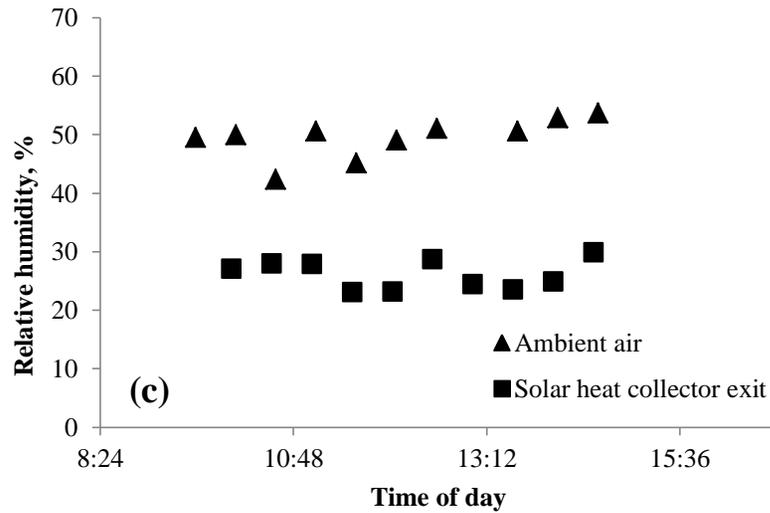
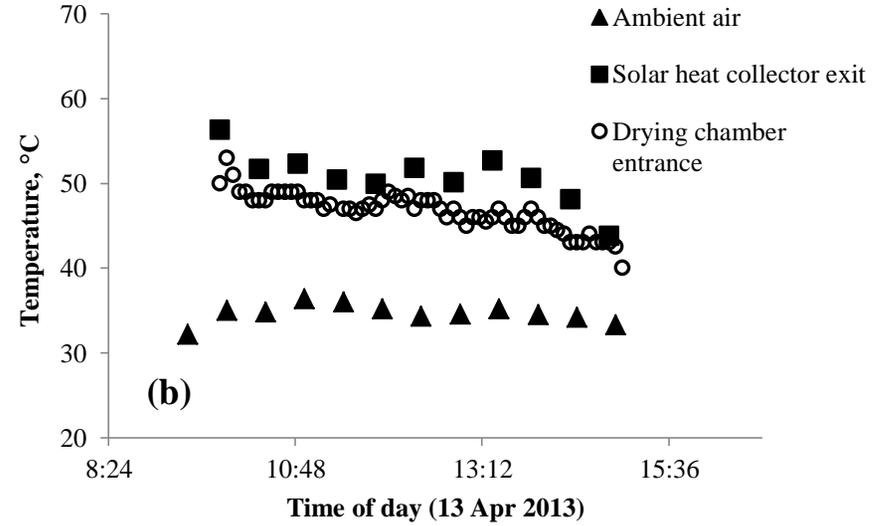
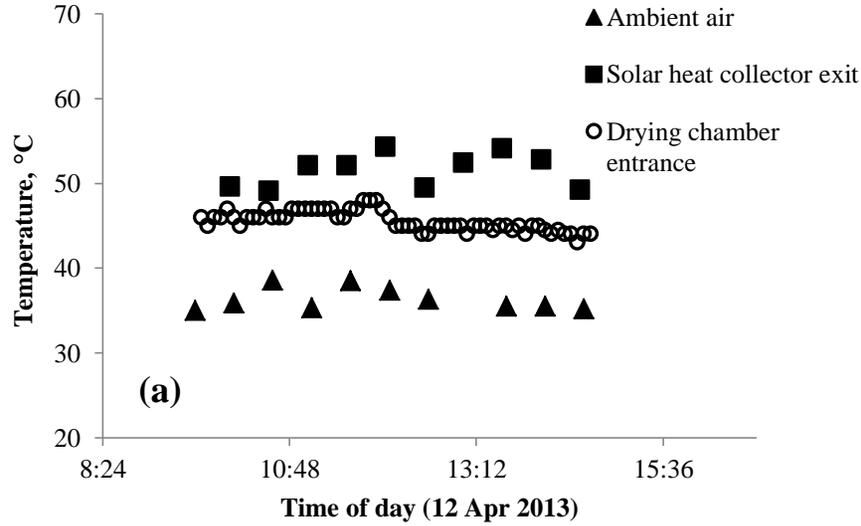


Fig. 8.14 Air temperature and relative humidity at ambient condition, at the exit of the solar heat collector, and at the entrance of the drying chamber during two solar drying runs.

et al. (2009) illustrated the sticky deposit profile across the length of the rotary drum dryer, with the highest accumulation per unit surface area found on the first 0.5 m of the dryer length.

Low thermal efficiency. Heat losses were expected in the present system setup because of a number of considerations that were prioritized in the study. These losses, however, need to be addressed to minimize drying time and energy consumption. The drying chamber and the duct connecting the heating system, for example, were not insulated during the study because of logistical considerations. The duct type and material was also chosen to facilitate assembly and dismantling when switching from solar to biomass heating system and transport from one drying location to another. Heat losses through the duct and drying chamber surfaces would, consequently, contribute to low efficiency values. Heating values and moisture content of the fuel wood also need to be determined to provide more accurate thermal efficiency estimates.

Unloading of dried material. Unloading dried samples under the present design can be time consuming as some of the dried material could not be easily discharged. The bottom section needs to be slightly modified to minimize unloading time as this could disrupt other farm or household activities. Farmers had previously articulated that sun drying BSG was not an attractive option, even if it can increase its shelf life, because of the amount of work involved.

Solar heat collector design improvement. Since solar drying is a cheaper alternative, studies on improving the solar heat collector can also be undertaken.

Combined hot air and sun drying. Since the last stage of drying is typically the most inefficient because it takes considerable energy to remove the remaining moisture, it is also recommended to dry the spent grain from 70% moisture to 20-25% moisture using the batch dryer, instead of

drying it directly to 10% moisture. Sun drying can be used to dry from 25% to 10% moisture. This alternative will translate to reduced residence time in the batch dryer, lesser amount of biomass fuel consumed, increased amount of BSG that can be dried, and consequently, lower the drying costs. The labor requirement associated with sun drying also needs to be assessed.

Wet storage. The wet storage method currently employed in cattle farms needs to be improved to minimize mold development and BSG spoilage. Ensiling BSG could be investigated to produce a more stable product with minimal losses in nutritional quality. The effect of the various phases comprising the ensiling process on silage quality, for example, could be studied and optimized. An initial study on the effect of incorporating ensiled BSG as chicken feed on animal growth performance and carcass quality had been conducted in collaboration with two Animal Science undergraduate students. The study, however, needs to be improved to ensure optimum condition is achieved during each phase of the ensiling process.

8.5 Conclusions

Brewers spent grain (BSG) in Misamis Oriental is disposed, transported, stored, and used as an animal feed ingredient in its wet form. Drying was rarely practiced in the cattle farms visited during the study. A solar- or biomass energy-powered batch dryer, equipped with a rotary wheel for agitating the spent grain, was fabricated and evaluated to improve the shelf life and safe use of brewers spent grain. Solar drying, using the present design, was not an attractive option because of longer drying times. Drying using biomass energy was the more feasible option.

Locally produced BSG is a high protein high fiber material. Dried BSG, with a particle size of about 1.10 mm, has low bulk density ($215 - 221 \text{ kg}\cdot\text{m}^{-3}$) and high porosity (83%). Its drying and

moisture sorption characteristics were best described by the logarithmic and Oswin models, respectively.

Further studies need to be conducted to assess and minimize potential risks of prolonged storage of wet BSG under local conditions and to improve prototype dryer performance in terms of product yield, thermal efficiency, and unloading time. Use of wet storage methods, such as ensilage, also needs to be investigated.

Chapter 9

General Discussion

Reduced protein quality, a highly energy-intensive drying process, and product variability are some of the challenges that currently confront the production of wheat distillers dried grain with solubles (DDGS) in western Canada. This PhD research project mainly focused on the laboratory – scale investigations of the blending and drying processes of DDGS production to understand some of the contributory factors to these challenges. In particular, this project examined the effect of condensed distillers solubles (CDS) : wet distillers grain (WDG) blending proportion and drying methods/conditions on protein quality, proximate composition, and various physical properties. Microwave-based drying methods were specifically studied because of their potential to minimize protein damage and energy consumption.

An auxiliary study on drying and physico-chemical characterization of brewers spent grain (BSG) was also pursued in the Philippines. It enabled the candidate to apply the knowledge and skills learned in her PhD research project to a related problem situation within a developing country context. This chapter, then, summarizes the key findings of these laboratory-scale investigations and the implications of these results in their respective contexts.

9.1 The by-products of fuel ethanol and beer production

Wheat DDGS and BSG are high protein, high fiber by-products of the fuel ethanol and brewing industries, primarily utilized as an animal feed ingredient in western Canada and in Misamis Oriental, Philippines, respectively. Since these were produced from different raw materials and

subjected to different processes and processing conditions, they differed in their physico-chemical characteristics. Wheat DDGS had higher protein and ash content but lower fat content than BSG. While both dried materials were very porous (68 – 83% porosity), wheat DDGS samples were relatively less porous than BSG since the former had smaller particle sizes and denser than the latter. Wheat DDGS samples were also darker and redder than the BSG samples. This could be due to differences in chemical composition and exposure to elevated temperatures. Wheat DDGS samples were dried in the ethanol plant, where higher temperatures were used compared to the temperature range employed in drying BSG.

9.2 CDS:WDG blending proportion and wheat DDGS variability

Both wheat DDGS and BSG showed differences in the chemical composition of sample materials obtained from two production batches in their respective processing plants. While causes of BSG variability were not investigated in this study, differences in the raw materials (such as the variety of barley and the type of adjuncts) and in the proportion of malted barley and adjuncts used during the mashing process of beer production could be two of the possible contributory factors.

In wheat DDGS, laboratory-scale investigations demonstrated that variation in the condensed distillers solubles (CDS) : wet distillers grain (WDG) blending proportion affected its chemical and physical characteristics. These suggest importance of the maintaining the CDS:WDG blending proportion within a specific range in ethanol plants to produce wheat DDGS with more consistent and more predictable quality characteristics.

9.2.1 Chemical composition

Study results had shown that as CDS level in the blend increased, protein and ash content increased while fat and fiber content decreased. These compositional trends were attributed to differences in the chemical composition of the two wheat DDGS parent streams: CDS and WDG. The CDS fraction had higher protein and ash content and lower fat and fiber content compared to WDG. Comparison of the proximate composition results between ethanol plant-sourced and laboratory-produced wheat DDGS samples suggests that the former may have been produced at CDS levels higher than those employed in the study (15-45% CDS).

Producing wheat DDGS with more consistent nutritional characteristics would enhance its marketability as an animal feed ingredient and thus, improve the ethanol plant's revenue generation potential. It would enable animal processors and nutritionists to forecast their raw material requirements more accurately and optimize their feed formulations more efficiently. Feed mixing procedures can also be standardized to ensure that the nutrient variability of their feed products is kept within allowable limits.

9.2.1.1 Protein quality

While high CDS level is desirable because of the associated high protein and mineral content, this could also potentially lead to increased production of heat-damaged proteins via Maillard reaction. With increased protein content, more lysine, and more free ϵ -amino groups of lysine would be available to react with the carbonyl compounds of reducing sugars. Coupled with elevated processing temperatures, such as those employed in drying and pelleting processes, use of high CDS blends would increase the rate of Maillard reaction and thus, increase the incidence of heat-damaged proteins. This would lower the nutritional value of wheat DDGS. Selection of

an appropriate range of CDS:WDG blending proportion and drying conditions would be imperative to maximize nutrient content but also minimize protein damage.

9.2.2 Physical properties

Consistent use of a specific range of CDS level during the blending process would also enable better predictability of its physical characteristics. This could facilitate better planning, control, and improvement of storage, handling and transport processes and enhance the marketability and utilization of wheat DDGS as an animal feed ingredient.

Particle size. In this study, particle size decreased as CDS level in the blend increased. Although grinding was employed to generate the bulk material after drying, controls were installed so that the effect of CDS level on particle size could still be observed. These include the use of a single screen size, four times larger than the geometric mean diameter of plant-sourced sample, for all sample types, use of the same grinding equipment and the same grinding time, and use of a similar amount of feed material for grinding. With increased CDS level in the blend, there is increased presence of the finer but heavier particles and decreased amount of the larger but lighter bran-related particles associated with the WDG fraction. Solids content of the CDS and WDG fractions, based on their moisture contents, was about 24.5% and 33.2%, respectively. Although CDS had lower solids content than WDG, it was heavier. This was observed in the bulk density values of the WDGS samples. As CDS level increased (and WDG level decreased), bulk density of the wheat WDGS increased.

The presence of small and large particles in varying degrees contributes to less particle size uniformity. This was reflected in the values of the uniformity coefficient and geometric standard

deviation of particle diameter by mass (S_{gw}) derived from particle size analysis data. Values of S_{gw} and uniformity coefficient were significantly higher at higher CDS level.

Wheat DDGS samples used in the laboratory investigations came from an ethanol plant which employed a ring dryer in its DDGS production. The plant-sourced wheat DDGS sample, sourced on the same day as the CDS and WDG used in laboratory-produced DDGS, had smaller particle size than the ones produced in the laboratory. One possible cause of smaller particle size could be the CDS level used in the ethanol plant. The plant-sourced sample showed higher protein and lower fiber content, suggesting that it may have been produced at higher CDS level than what was used in the laboratory. Results showed that blends with higher CDS level had higher protein and lower fiber content than those with low CDS and were associated with smaller particle sizes. However, since the laboratory conditions did not simulate the plant-scale operations, these results may not be reflected at the plant level, particularly in blending and drying systems that could promote particle agglomeration.

Plant-scale studies (Kingsly et al. 2010; Clementson and Ileleji 2012) on corn DDGS indicated that particle size increased with CDS level due to agglomeration during rotary drum drying. A laboratory-scale investigation by Ganesan et al. (2008a) showed significant differences in particle size as soluble level was varied but no distinct trends were observed. The term “solubles” in their investigation was defined differently from this study. Differences in the particle size-CDS level relationship between corn and wheat DDGS in these studies could be attributed to inherent differences in the chemical composition of the respective CDS and WDG fractions and in the processing conditions employed in generating the bulk material.

These laboratory wheat DDGS results, however, suggest the importance of maintaining a specific range of CDS:WDG blending proportion to control particle size variation. A number of animal feed studies reported shown that particle size of feed materials affected growth performance, nutrient digestibility, and gastric ulcer development in swine (Seerley et al. 1988; Healey et al. 1994; Wondra et al. 1995a). These studies recommended optimal particle sizes for specific feed materials. Wondra et al. (1995b) also reported that particle size uniformity also affected the apparent digestibility of nutrients in pigs. Meeting particle size requirements of animal feed processors would require better process control during DDGS production. Maintaining a specific range of CDS:WDG blending proportion could be one way toward meeting particle size requirements of customers.

Bulk density. With increased presence of smaller but heavier particles and decreased amount of the larger but lighter bran-related particles, bulk density increased as CDS level increased. The smaller solids can also easily penetrate inter-particle void spaces, thus, increasing bulk density.

Controlling bulk density variation would enable the ethanol plant to forecast its logistical requirements more accurately and plan on how to meet these at minimum cost. If the bulk density of wheat DDGS were to range from $339.7 \text{ kg}\cdot\text{m}^{-3}$ to $437.0 \text{ kg}\cdot\text{m}^{-3}$, producing the higher bulk density DDGS ($437.0 \text{ kg}\cdot\text{m}^{-3}$), for example, would enable the ethanol plant to place about 14 t more in a 147 m^3 hopper car (Greenbrier Companies n.d.) than if it were to produce and transport the lower bulk density DDGS ($339.7 \text{ kg}\cdot\text{m}^{-3}$). Loading 14 t more for every hopper car used would translate to considerable savings.

Pelleting. Increased presence of the smaller but heavier high protein solids in the blend also lead to denser and stronger pellets. Smaller particle sizes provided more surface area for contact and

chemical changes (such as protein denaturation, Maillard reaction) that may have occurred during densification may have promoted better binding. Denser pellets translate to lower unit storage and handling cost while difficult-to-break pellets would minimize product losses due to breakage as these are able to withstand the stresses during handling, transport, and storage better. However, pelleting may also exacerbate the protein damage that the wheat DDGS may have already incurred during drying because of the heat applied, along with friction, during densification. Additional tests would be necessary to verify its effect on nutrient content.

Flow properties. While higher CDS level in the blend led to higher bulk density and production of denser and stronger pellets, it could also adversely affect other processes. Higher CDS level in the blend, for example, could lead to flow problems. While wheat DDGS had been classified as a fairly flowable material that may sometimes require vibration to assure flow, high CDS samples showed lower flowability (higher compressibility, angle of repose, and angle of spatula values). These samples also showed higher coefficient internal friction, indicating that the particles in the bulk material present higher resistance to movement. Thus, if the CDS level is not controlled and allowed to increase beyond a certain threshold, no-flow or irregular flow incidents could occur during loading and unloading operations. Such incidents would cause unnecessary downtimes and higher costs.

Drying characteristics. Variation in the CDS:WDG blending proportion could also affect the subsequent drying process. Study results showed that effective moisture diffusivity and thermal diffusivity values were significantly lower at higher CDS level and could contribute toward lower drying rates and thus, longer drying times. Differences in the effective moisture diffusivity and thermal diffusivity values were attributed to differences in the chemical composition and porosity. Samples with lower CDS levels had higher porosity values compared to those with high

CDS level. Additional studies, however, are needed to distinguish the effect of composition from porosity.

These results highlight the need to determine or to verify the appropriate CDS:WDG blending proportion that can be practicably used in an ethanol plant that would also satisfy some if not most of the customer requirements. Maintaining the blending proportion within a specific range enables the production of wheat DDGS with more consistent quality characteristics.

In this project, the quantification of the effects of CDS:WDG blending proportion on the physico-chemical properties of wheat DDGS was based on laboratory-scale investigations. Plant-scale investigations need to be conducted to obtain a more accurate picture of its effects on wheat DDGS physical characteristics.

9.3 Drying the by-products of fuel ethanol and beer production

Both wheat DDGS and BSG were derived from high moisture processing residues. Drying enables their safe storage, handling, transport, and utilization. The contexts under which drying of these two materials was investigated differed. In wheat DDGS, laboratory-scale drying investigations were primarily conducted to address challenges relating to product quality and process efficiency. In BSG, drying was rarely practiced as the material was disposed, transported to farms, stored, and utilized in its wet form. A dryer was developed as a means of curbing BSG spoilage and promoting its safer use.

9.3.1 Drying and wheat DDGS product quality

In the proximate composition and physical attributes of wheat DDGS, the effect of drying conditions, while significant, can be considered secondary to that of the CDS:WDG blending

proportion. These can be inferred from the distribution of the ANOVA total sums of squares, where CDS:WDG blending proportion or CDS level in most cases accounted a major portion of the variation. This was not, however, seen in lysine, ADICP, and fat content and in the color parameters, where drying conditions, particularly temperature, accounted the major bulk of the variation.

Protein quality. Maximum protein and lysine content and minimum ADICP content was achieved when wheat DDGS was produced at the highest CDS level investigated (45% CDS) and dried under lower settings: 80°C under forced air convection, 676 W power level under microwave, and 150°C-30% power (316 W) under microwave convection. Forced-air convection drying of ethanol plant-sourced wheat WDGS samples also showed decrease in lysine content as drying temperature was increased. Lysine content of these laboratory-dried samples was (0.96% - 1.12%, db) also higher compared to the ethanol plant-dried sample (0.76%, db). Differences were attributed to the higher drying temperatures employed in the ethanol plant than in the laboratory.

Variations in lysine and ADICP contents were the result of Maillard reaction, which involved the binding of the amino groups of amino acids, such as lysine, with the carbonyl groups of reducing sugars (Fayle et al. 2002; Owusu-Apenten 2004). The rates of these reactions are affected by temperature, time, pH, water activity and chemical composition (Ames 1990; Owusu-Apenten 2004). Increase in temperature increases the Maillard reaction rate, and thus, the production of heat-damaged proteins. Since heat-damaged proteins are not nutritionally available to the animals, as these are not susceptible to enzymatic decomposition (Mao et al. 1993), their high incidence in wheat DDGS would lower the latter's nutritional value.

Figure 9.1 shows how drying conditions, CDS level, and their interaction accounted for the observed variations in lysine and ADICP content. This suggests the stronger influence of drying conditions (temperature, microwave power, and microwave convection setting) on lysine content variation. In ADICP content, temperature, rather than CDS level, seemed to be the stronger influence under forced-air convection drying.

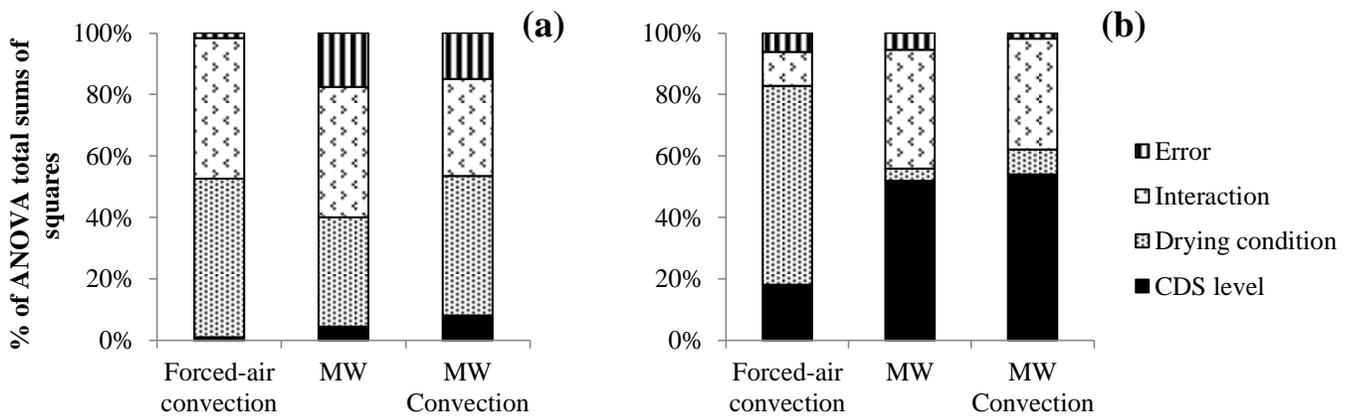


Fig. 9.1 Sources of variation in (a) lysine and (b) acid detergent insoluble crude protein (ADICP) of wheat DDGS under forced air convection, microwave (MW), and microwave convection drying

Fat content. While fat content of forced-air convection-, microwave-, and microwave convection-dried samples still generally decreased as CDS level was increased, the strength of these linear relationships was not as strong as that exhibited by the freeze-dried sample. This indicates the influence of drying conditions, particularly temperature, on fat content. This is seen in Fig. 9.2, where interaction effect of CDS level and drying conditions, particularly under forced-air and microwave convection methods accounted a higher proportion of the observed variation, which was not observed in the chemical constituents (protein, ash, neutral detergent fiber).

In the 15% CDS samples, where higher fat contents were observed compared to the other CDS levels investigated, those dried under 80°C had significantly higher fat content than those dried under 40°C and 120°C. Those dried under the lowest microwave convection setting (at constant microwave power) had highest fat content than those dried at the higher settings. Fat losses in samples dried under higher temperature or in those with longer exposure to a lower drying temperature could be due to lipid oxidation. In microwave-dried samples, those dried under the 805 W setting showed significantly higher fat content than those dried under the 420 to 701 W power levels. Shorter exposure time to higher microwave power may have retarded lipid oxidation rate. Additional tests, however, are needed to verify the effect of these drying conditions on lipid oxidation.

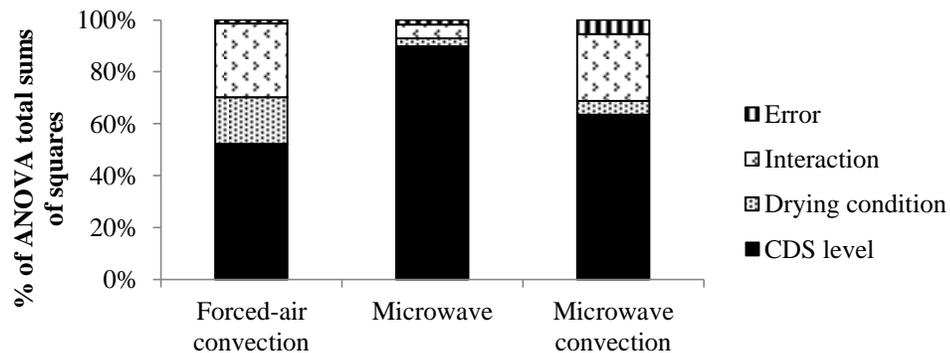


Fig. 9.2 Sources of variation in fat content of wheat DDGS under forced air convection, microwave, and microwave convection drying

Color parameters. The effect of CDS:WDG blending proportion on the color parameters of wheat DDGS was clearly observed in freeze-dried samples. As CDS level in the blend was increased, the freeze-dried samples became significantly darker and redder. However, when these samples were dried under forced-air convection-, microwave-, and microwave convection, these linear relationships were not observed, indicating the influence of drying conditions. Color changes during drying are attributed to Maillard reaction.

Results on the color parameters highlight the importance selecting the appropriate range of CDS:WDG blending proportion and milder drying conditions. Under forced-air convection, maximum Hunter L (lightness) and minimum Hunter a (redness) and b (yellowness) values were achieved under a high CDS (45%) – low temperature (40°C) combination. Under microwave and microwave convection, this was achieved in the combinations of 15% CDS-701 W microwave power level and 45% CDS – C4 (190°C-30% power), respectively.

Only a few significant linear relationships between color and protein quality parameters were observed in the study. In laboratory-produced wheat DDGS samples, there was (i) an inverse relationship between lysine content and redness of forced-air convection dried samples ($R^2 = 0.51$); and (ii) a positive relationship between lightness and ADICP content ($R^2 = 0.40$) of microwave-dried samples. In plant-sourced WDGS samples that were dried under laboratory conditions, a positive linear relationship between lightness and lysine content ($R^2 = 0.55$) was observed in forced-air convection-dried samples.

9.3.2 Use of microwave energy in drying wheat WDGS

Microwave and microwave convection methods achieved desirable product quality, comparable to the level associated with low temperature drying, at much shorter times. This was shown in Table 3.8, where optimum protein quality (highest protein and lysine content and lowest ADICP) was achieved under the 45% CDS – 80°C (forced convection), 45% CDS – P6 (676 W power level, microwave), and 45% CDS – C2 setting (150°C – 316 W, microwave convection) combinations. Drying under 80°C, however, took about an hour while drying under the P6 and C2 settings took only about 8 and 17 min, respectively. In terms of color, optimum

combinations, as previously mentioned, were: 45% CDS – 40°C (forced-air convection), 15% CDS-701 W (P8, microwave), and 45% CDS – C4 (190°C-332 W, microwave convection), respectively. Drying under 40°C took about 3 h while it took about 7 min under P8 and about 16-18 min under C4. Variation in microwave power level did not also cause as much variation in fat content, as shown in Fig. 9.2.

Rapid drying rates under the microwave method result from the volumetric heating of the material, which creates a high internal vapor pressure gradient between its core and surface that enabled faster moisture movement to the surface (Yaylayan and Roberts 2001). In conventional hot air drying, moisture removal is slower because heat transfer to cause evaporation is slow. Shorter exposure to heat under microwave drying could also hinder completion of Maillard reactions responsible for many product quality changes. Further, microwave drying does not also cause the formation of a dehydrated crust, which could impede moisture movement and promote Maillard reaction.

Rapid drying rates translate to lower energy consumption. This was also reflected in the results of the techno-economic evaluation, where microwave drying showed the least energy consumption in producing wheat DDGS with 10% moisture (wb), among the scenarios considered. However, in terms of cost, complete replacement of the conventional hot air drying with microwave drying is not yet economically feasible for wheat DDGS. The estimated cost of microwave drying equaled the 2011 average market price. Although DDGS prices in 2013 were higher, the estimated drying cost using high efficiency microwave system may not have enough elbow room for a profitable operation (Fig. 7.9) since the cost of other wheat DDGS production processes have yet to be factored in.

Under the present set of assumptions, high cost of electricity was seen as the major hindrance. It accounted about 61 - 73% of the drying cost per tonne, depending on drying system efficiency. While initial investment on equipment would be quite high, its cost contribution was only about 13-18% of the drying cost per tonne. Unless there are cheaper sources of electricity or very attractive market incentives of producing high protein quality DDGS, it would be very difficult to encourage ethanol producers to consider microwave drying in protein quality improvement efforts. Finish drying, which involved the use of microwave energy toward the end of the conventional hot air drying, was seen as the more viable option, since its costs were closest to that of rotary drying. The presence of very attractive market incentives or potential cost saving opportunities would be necessary to compensate for this additional investment.

9.3.3 Drying brewers spent grain

Drying was explored as a potential means toward enabling the safer storage and use of BSG and broadening the limited feed resource base of smallholder animal producers in Misamis Oriental, Philippines. Nutrition has been identified as one of the constraints in improving small-scale livestock production and productivity in Bangladesh, Vietnam, and Philippines, with animals commonly “fed with sub-maintenance and maintenance diet” (Food and Agriculture Organization 1999). Although the dairy cattle farmers interviewed in the study have more diversified feed sources, other smallholder producers largely depend on tethered grazing on grasses in neighboring lots to feed their beef cattle and goats and on grain milling by-products for swine feed (Mosqueda 2014). Making BSG and other cheaper alternative feed sources more available to these smallholder producers could enable them to improve their productivity and income generation potential. BSG spoilage, however, was a concern because this resource was commonly transported, stored, and utilized wet. To address this, a prototype solar- or biomass-

powered batch drying system was developed and evaluated for potential use by the smallholder producers. Ways to improve the technical performance of this dryer had been presented in Chapter 8.

There are a number of considerations that need to be assessed and addressed before introducing drying and other BSG spoilage prevention methods to smallholder producers, both existing and potential BSG users.

Firstly, drying is not commonly practiced by the BSG users interviewed in this study, with only one farm occasionally employing sun drying. Aside from being weather-dependent, sun drying requires considerable labor resources which smallholder producers cannot afford or would not want to invest in. This could be the same with the other BSG users in the province or in other parts of Mindanao. Thus, using a biomass-fueled dryer may not be an attractive option to them. Resource-poor smallholder farmers may only be able to engage on drying through a collective or cooperative arrangement with other farmers or with some support from the local government.

Use of ensiling technology for wet storage of BSG needs investigated as an alternative to drying. Ensilage could generate a more stable product than the current wet storage method employed in farms. Comparative laboratory-scale studies on the effect of these two wet storage methods on the feed quality of BSG need to be conducted.

Secondly, there is very limited published information in Misamis Oriental about BSG and other alternative feed sources, how these can be accessed or propagated, safely stored, and efficiently used. This could constrain smallholder farmers from considering readily available and accessible feed sources or from using these more efficiently and safely.

Partnerships with specific *barangay* (village) officials, El Salvador City government, farmers' organizations, academic institutions, and the regional field unit of the Department of Agriculture may be actively sought to assess and address these concerns. Creative research and learning programs can be developed and implemented to raise awareness or improve understanding on feeds nutrition and on BSG and other alternative feed resources, including the associated preparation and storage methods so these can be used more safely and efficiently.

Since the local government of El Salvador already operates a weekly livestock auction market, it can also consider strengthening animal production end of the chain so smallholder farmers would be able to participate more fully in this market. It can, for example, consider studying the feasibility of creating a village-level feeds processing enterprise program to support the need of smallholder producers for cheaper but high nutrient feed sources. Because of their low animal production and feed requirement volume, smallholder farmers need to consolidate so they can more economically access and utilize alternative feed materials like the BSG. Drying BSG and/or formulating more nutritionally-balanced, BSG-incorporated feeds could be a feasible undertaking at the village level rather than by individual farms. The local government unit can collaborate with academic institutions in assessing the feasibility of this village level enterprise program.

Chapter 10

Conclusions and Recommendations

The main objectives of this PhD project were: (i) to evaluate alternative methods of drying wheat distillers grain with solubles that would minimize protein damage, energy consumption, and associated costs; (ii) to describe the physico-chemical characteristics of wheat distillers dried grain with solubles (DDGS); and (iii) to assess the effect of drying methods/conditions and the blending process on the physical and chemical characteristics of wheat DDGS. A secondary aim was to apply the knowledge and skills acquired from accomplishing these main objectives in addressing a related issue within the context of a developing country (Philippines). The general conclusions drawn from these laboratory-scale investigations and Philippine case study and recommendations for future research are presented below.

10.1 Conclusions

10.1.1 Evaluation of drying technologies for, and physico-chemical characterization of, wheat distillers grain with solubles

- a. Condensed distillers solubles (CDS): wet distillers grain (WDG) blending proportion, or CDS level in short, affected the chemical composition of wheat DDGS. As CDS level increased, protein and ash content increased while fat and fiber content decreased. Samples with high CDS level had higher lysine and lower acid detergent insoluble crude protein (ADICP) content.

- b. CDS level also influenced the physical properties of wheat DDGS. Wheat DDGS samples with higher CDS level were smaller, denser, less uniform in size, less flowable, and less dispersible. These also had significantly lower thermal diffusivity, higher angle of internal friction, and led to the production of pellets with higher density and failure stress. Effective moisture diffusivity linearly decreased with increase in CDS level.
- c. Aside from CDS level, drying conditions also markedly affected lysine, ADICP, and fat content and the color parameters of wheat DDGS.
 - i. Maximum lysine and minimum ADICP content was achieved in high CDS level samples dried under low temperature, microwave power, and microwave convection settings.
 - ii. Fat content decreased as drying temperature increased. High temperature settings at constant microwave power, longer exposure to lower microwave power level and shorter exposure to higher microwave power level also adversely affected fat content.
 - iii. Increased CDS level resulted to darker, redder freeze-dried wheat DDGS. Drying under lower temperature and microwave convection setting produced lighter samples. Microwave- and microwave convection-dried samples with higher CDS were redder and yellower.
- d. Page model best described the thin layer drying behavior of wheat-based wet distillers grain with solubles under forced-air convection, microwave, and microwave convection methods. Drying rates increased with temperature and microwave power.

- e. It is not yet economically viable to replace conventional hot air drying with microwave drying in commercial wheat DDGS production, despite the following advantages:
 - i. Desirable protein quality (high lysine content, low acid detergent insoluble crude protein), associated with low temperature drying, was achieved under microwave drying at a much shorter time.
 - ii. Microwave drying showed the least energy consumption compared to hot air (rotary), boost, and finish drying

High cost of electricity was a major hindrance. Finish drying, which involved the use of hot air during the first stage of drying followed by the use of microwave energy toward the end of the drying process, was seen as the more feasible option.

- f. Ethanol plant-sourced wheat DDGS samples from two production batches differed in their physico-chemical characteristics.
 - i. Moisture content affected some of their physical properties. When moisture content was increased, bulk density increased while particle density and compressibility decreased. Lower moisture samples showed higher porosity, higher pellet failure stress, higher energy consumption during compression, higher thermal diffusivity and dispersibility.
 - ii. Their drying, moisture sorption, and compression characteristics were best described by the Page, GAB, and Kawakia-Ludde models, respectively. Under the Carr classification system, the samples were considered fairly flowable, sometimes

requiring vibration to ensure flow, and floodable, requiring some measures to prevent flushing.

- iii. In comparison with laboratory-produced wheat DDGS samples with 15-45% CDS level, plant-sourced samples had higher protein and lower fiber content and were smaller and darker in color. In comparison with brewers spent grain produced in Misamis Oriental, Philippines, wheat DDGS had higher and ash content but lower fat content, and was smaller, denser, and darker and redder in color.

10.1.2 Philippine case study

- a. Brewers spent grain is a cheap, low bulk density, high protein high fiber resource. It is commonly used as animal feed by cattle farmers in Misamis Oriental. It is transported, stored, and utilized in its wet form. Drying spent grain is rarely practiced.
- b. A forced-air convection prototype batch dryer was developed and was able to reduce moisture of the spent grain from 71% to 10%, wb. Accumulation of wet material on the blades of the rotating wheel inside the drying chamber lowered its product yield. Drying using the biomass furnace was feasible with some design improvements. Design of the solar heat collector needs to be improved to shorten drying time.

10.2 Recommendations for future work

10.2.1 Wheat DDGS

- a. Physico-chemical characteristics of wheat DDGS produced under combined hot air-microwave (finish) drying. The cost analysis showed that finish drying was the more

feasible way of incorporating microwave energy in wheat DDGS production at present. It would be interesting to see how this combination would affect protein quality, fat content, color, and other characteristics.

It would also be worthwhile to study the dielectric properties of wheat CDS, WDG, WDGS at varying CDS:WDG blending ratio, and DDGS as well as the flow properties of the microwave finish-dried material. It may be necessary to fluidize the dried material as it could form a hard mat during microwave-finish drying.

- b. Physico-chemical characteristics of wheat DDGS produced from other ethanol plants. Raw materials used in the study came from only one ethanol plant. It would be interesting to compare, for example, how different plant processes would affect these characteristics.
- c. Plant-scale investigations on the effect of CDS:WDG blending proportion using different drying systems (rotary and ring dryers). This research project was limited only to laboratory-scale investigations, which were not able to approximate the actual blending and drying conditions.
- d. Assessment of protein quality, fat, and degradation kinetics under high temperature drying to enable better control of the drying process. Studies done in this research undertaking were limited to dried product evaluation and drying temperatures were lower than those employed in ethanol plants.
- e. Determination of the effect of pelleting on the protein quality of wheat DDGS as this heat treatment could exacerbate the protein damage that the bulk material may have sustained during drying.

- f. Extension of these studies to include animal feeding trials to obtain a more complete, more concrete picture of the effect of ethanol plant processes (such as blending and drying) on animal growth performance.

10.2.2. Philippine case study on brewers spent grain

- a. Investigations to improve the technical performance of the prototype dryer, such as assessing sticky behavior of brewers spent grain vis-à-vis its properties and drying system variables so product yield can be improved, and design improvements of the drying and heating system to improve thermal efficiency and unloading efficiency.
- b. Feasibility of a local government unit operating a village-level feed processing (dryer-feedmill module) enterprise program to assist smallholder farmers in accessing cheaper feed alternatives.
- c. Shelf-life studies on wet and dried brewers spent grain under local conditions and the use of alternative wet storage methods, such as ensilage could be investigated to minimize BSG spoilage. Various phases of the ensiling process, for example, could be investigated to optimize silage quality. Feed quality of ensiled BSG samples and those stored using the method currently employed in cattle farms can also be compared. Effect of these two storage methods on animal growth performance and final animal product quality can also be assessed.

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Appendix A

Physico-chemical properties of microwave vacuum-dried wheat distillers grain with solubles

A similar version of this chapter would be submitted to a peer-reviewed journal and would be presented in a conference.

A.1 Abstract

Microwave vacuum drying was evaluated as an alternative to the conventional hot air drying of wheat distillers grain with solubles. Drying kinetics, proximate composition, physical attributes, thermal and mechanical properties of microwave vacuum-dried samples were assessed under four microwave power levels at constant vacuum pressure and compared with a commercial sample dried by a ring dryer. Page model adequately described the drying behavior, with its drying rate constant increasing positively with microwave power. Microwave power significantly affected acid detergent insoluble crude protein content, particle size, bulk and particle densities, and static friction coefficient, and the compression characteristics. The microwave vacuum-dried samples showed higher fat, lower acid detergent insoluble crude protein, higher bulk density, larger particle sizes, and were also lighter, less red and more yellow compared to the commercial sample. Their protein, ash, and fiber contents, particle and pellet density, porosity, thermal conductivity, and thermal diffusivity values were numerically close, if not statistically similar, to those reported for the commercial sample.

A.2 Introduction

Reduced protein quality is one of the current challenges that confront the utilization of wheat distillers dried grain with solubles (DDGS) in western Canada, particularly as feed for monogastric animals (University of Saskatchewan 2010). Despite its higher crude protein and amino acid content, animal feed-related studies showed that wheat DDGS had low lysine content and digestibility values (Nyachoti et al. 2005; Widyaratne and Zijlstra 2007; Lan et al. 2008; Cozannet et al. 2010). Since lysine is usually the limiting essential amino acid in animal feed ingredients, its loss lowers the nutritive value of a feed material (Fayle and Gerrard 2002), ultimately affecting animal growth performance. High temperature drying has been identified as one of the major causes of protein damage during wheat DDGS production (Nyachoti et al. 2005; Widyaratne and Zijlstra 2007). Protein damage is one of the consequences of Maillard reaction, which is invariably accelerated by elevated processing temperatures, among other factors (Ames 1990; Owusu-Apenten 2004). In addition, conventional hot air drying is also a highly energy intensive process, estimated to account about one-third of the total thermal demand of wheat ethanol plants (Murphy and Power 2008). Previous studies on the effect of forced air convection, microwave and microwave convection drying on energy consumption, lysine damage and physical properties demonstrated the potential of using microwave energy in wheat DDGS production (Mosqueda et al. 2013a; 2013b; 2013c).

Microwave vacuum drying had been investigated as an alternative drying method for a number of materials where exposure to high drying temperatures for long periods of time can seriously damage their nutritional characteristics and other quality attributes. It combines the advantages of microwave drying (rapid heat transfer rates) and vacuum drying (lower process temperature,

enhanced moisture transfer) and offers the potential of improving product quality and energy efficiency of the process (Yongsawatdigul and Gunasekaran 1996; Drouzas and Schubert 1996; Mousa and Farid 2002; Meda et al. 2008; Song et al. 2009). Some of these investigations reported that materials dried under microwave vacuum or microwave vacuum in combination with other methods exhibited higher retention rates of pigments (Lin et al. 1998; Cui et al. 2004; Yanyang et al. 2004), nutrients (Lin et al. 1998; Yanyang et al. 2004), and other bioactive substances (Cui et al. 2006). Improvements in drying rates (Sunjka et al. 2004; Yanyang et al. 2004), color (Drouzas et al. 1999), rehydration (Drouzas and Schubert 1996; Lin et al. 1998), and in other quality characteristics (Krokida and Maroulis 1999; Cui et al. 2003) relative to high temperature forced-air drying were also reported. Materials used in these investigations include apple, banana, carrot, Chinese chive leaves, cranberries, *Ganoderma lucidum* extract, garlic, pectin gels, potato, and wild cabbage.

There were also studies that assessed the effect of process variables (microwave power and vacuum pressure) on the drying kinetics and quality characteristics of selected products. Careful selection of these process variables is important for optimal process performance and product quality improvement. Studies drying banana (Drouzas and Schubert 1996), Saskatoon berries (Meda et al. 2008), and potato slices (Song et al. 2009) demonstrated that drying rate was significantly influenced by microwave power level than by vacuum pressure. Chauhan and Srivastava (2009) found that although both microwave power and vacuum significantly affected selected quality attributes of dried green peas, the effect of microwave power level on linear shrinkage, apparent density, rehydration, and green color ratios, overall acceptability, drying time, and drying efficiency was more pronounced. Meda et al. (2008) reported that total color difference observed in Saskatoon berries was dependent on microwave power and vacuum

pressure levels. In cranberries, darker samples were observed at higher microwave power levels (Sunjka et al. 2004).

Considering the potential of microwave vacuum drying in improving product quality and energy efficiency, it could be an alternative for drying wheat-based wet distillers grain with solubles (WDGS). There were no published studies found on microwave vacuum drying of corn or wheat WDGS. This study, then, investigated the effect of microwave vacuum drying conditions on the drying kinetics and selected physico-chemical characteristics of wheat DDGS. Resulting physico-chemical characteristics of the dried product were also compared to those obtained from a commercial wheat DDGS sample.

A.3 Materials and Methods

Samples of wheat WDGS were obtained from a south Saskatchewan fuel ethanol plant, packed in plastic bags, and stored in a freezer (-18°C to -16°C) until these were used in the study. Bags were thawed overnight at room temperature (22-24°C) prior to the conduct of experimental drying runs. Initial moisture content was obtained using the Association of Official Analytical Chemists (AOAC) Official Method 920.36 (AOAC 2003a). Wheat DDGS sample materials were also obtained from the same ethanol plant on the same day as the WDGS material. These were placed in sealed bins and were stored in a 3-4°C cold storage room. It was assumed that both these materials (DDGS and WDGS) were derived from the same WDGS batch of the ethanol plant.

A.3.1 Drying characteristics

An Enwave® microwave vacuum dryer, Model VMD 1.8 (Enwave® Corp., Vancouver, BC) was used in the experiments (Fig. A.1). Four microwave power levels were used in determining the drying characteristics of wheat WDGS and in generating the material for the physico-chemical characterization. These power (P) levels are subsequently referred to in this paper as P4, P6, P8, and P10, with the corresponding power output estimated at 295 W, 489 W, 601 W, and 745 W, respectively (Tripathy 2009; Meda et al. 2008). Vacuum pressure was set at 88 kPa, which is the maximum setting for the equipment.

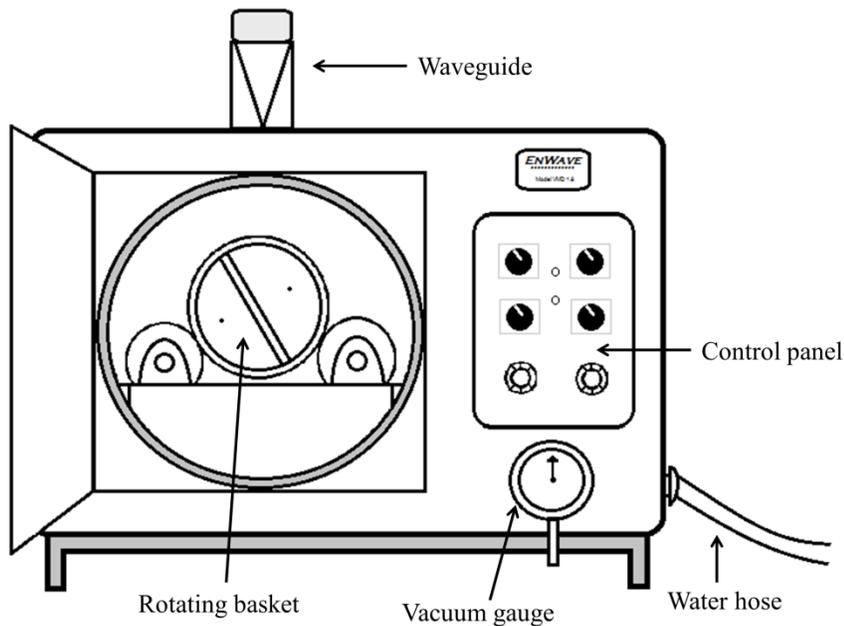


Fig. A.1 Schematic diagram of the microwave vacuum dryer used in drying wheat distillers grain with solubles.

The dryer has a cylindrical drying chamber, equipped with a rotating, perforated high density polyethylene basket as sample holder. Cloth bags were used to contain the 100 g samples before these were placed into the drying basket. Three bags were used for each drying run. Average

initial sample mass was 308.85 g with a standard deviation of 7.64 g. Two drying runs were conducted under each of the 4 power levels. Drying basket rotation speed was held constant (level 5 setting).

Sample mass was monitored every 3 min until the moisture content reached about 10.0-12.0%, wet basis (w.b.). This was done by switching off the microwave power, releasing the vacuum, taking out the sample bags from the basket, and weighing each bag. Taking out the basket, weighing the bags, placing them back into the basket, adjusting the vacuum, and turning on the microwave power took about 3 min. Weighing the sample bags took less than 30 s.

Drying data were fitted to the Page model (Eq. A.1). This model adequately described the drying characteristics of plant-sourced and laboratory-prepared wheat distillers grain with solubles under forced air convection, microwave, and microwave convection methods (Mosqueda and Tabil 2011; Mosqueda et al. 2013a). Equilibrium moisture content was assumed to be zero (Maskan 2000; Meda et al. 2008). Goodness of fit was assessed using the coefficient of determination (R^2) and the standard error of the estimate (SEE) (Eq. A.2).

$$MR = \exp(-kt^N) \quad (A.1)$$

$$SEE = \sqrt{\frac{\sum(MR_{\text{Observed}} - MR_{\text{Predicted}})^2}{n-2}} \quad (A.2)$$

In Eqs. A.1 and A.2, MR stands for moisture ratio, k and N are the Page model parameters, t is time in min, SEE stands for standard error of the estimate, and n is the total number of observations.

A.3.2 Chemical composition

Dried samples for proximate analysis were ground using a precision grinder equipped with a 0.8 mm sieve. The proximate analysis was conducted in duplicate runs. Detailed procedures are presented in the referenced AOAC documents. Moisture content was determined using the AOAC Official Method 920.36 (AOAC, 2003a). Crude protein was estimated using the Kjeldahl method, AOAC Official Method 984.13 (AOAC 2003b). The acid detergent fiber (ADF) and neutral detergent fiber (NDF) were estimated through AOAC Official Method 973.18 (AOAC 2003c) and through the method laid out by van Soest et al. (1991), respectively. The acid detergent insoluble crude protein (ADICP) was determined from the residue of ADF analysis using AOAC official method 984.13 (AOAC 2003b) and was expressed as a percentage of crude protein (dry matter basis). ADICP is used as a measure of heat damage in animal feeds and forages (van Soest and Mason 1991; Perry et al. 2003; Coblenz 2013). Crude fat was determined using the Goldfish fat extractor (Labconco Corporation, Kansas City, MO), following the AOAC Official Method 920.39 (AOAC 2003d), using anhydrous diethyl ether as extraction solvent. Crude ash was determined using AOAC Official Method 942.05 (AOAC 2003e). A lamb starter feed sample, AAFCO 0728 (Association of American Feed Control Officials (AAFCO) Check Sample Program, AAFCO, Champaign, IL), was used as a check sample.

A.3.3 Physical properties

To generate the bulk material for physical properties determination, dried samples were ground using a Thomas-Wiley mill (Thomas Scientific, Swedesboro, NJ), equipped with a 2.68 mm screen, a size about four times larger than the observed geometric mean diameter of commercial wheat DDGS samples. Samples, in 150-200 g batches, were ground for about 2 min. Moisture

content of the ground samples used for physical properties determination ranged from 11.8% to 12.2%, w.b.

A.3.3.1 Particle size and size distribution

Sieve sizes 16, 20, 30, 40, 60, 80, 100, 120, 140, 170, 200 and 270 were used for particle size analysis, following ANSI/ASAE S319.4 (ASABE 2008). The calculated geometric mean diameter was used to represent particle size in this study.

A.3.3.2 Bulk and particle density

A grain bulk density apparatus (SWA951, Superior Scale Co. Ltd., Winnipeg, MB) was used to determine the bulk density of wheat DDGS. Sample material was placed in a funnel and was allowed to freely flow into a 0.5 L steel cup. The discharge point in the funnel was about 55 mm above the brim of the steel cup. The contents in the cup were leveled by rolling a steel rod across the top edges of the cup. Bulk density was obtained by dividing sample mass contained in the cup with the cup volume. Particle density was determined using a gas multipycnometer (QuantaChrome, Boynton Beach, FL). Bulk porosity (ε), expressed as a percentage, was determined as a function of bulk (ρ_b) and particle densities (ρ_p) using Eq. A.3 below.

$$\varepsilon = 1 - \frac{\rho_b}{\rho_p} \quad (\text{A.3})$$

A.3.3.3 Color

Color of the samples was determined using the HunterLab spectrophotometer (Hunter Associates Laboratory Inc., Reston, VA) and was expressed in terms of the Hunter L, a, and b parameters, which represent lightness, redness, and yellowness, respectively.

A.3.3.4 *Compression characteristics*

About 1 g wheat DDGS samples were pelleted using a die-plunger assembly. The die (6.35 mm diameter and 135.34 mm length) was heated at 90°C while the plunger was attached to an Instron Model 3366 testing machine (Instron, Norwood, MA). Samples were compressed at 4400 N and crosshead speed was set at 30 mm·min⁻¹. Five pellets were produced from each of the P4, P6, P8, and P10-dried material. The 1 g plant-sourced DDGS samples were pelleted using an Model 1011 Instron testing machine and compressed at 4000 N with a crosshead speed of 50 mm·min⁻¹.

Pellet density was also determined by measuring the mass, length, and diameter of the resulting pellet immediately after it was extruded from the die. Specific energy consumption during compression and extrusion was estimated by calculating the area under the corresponding force-displacement curve using the trapezoidal rule and dividing it by the pellet mass.

Compression data were fitted to the Kawakita-Ludde model (Eq. A.4). This model adequately described the compression characteristics of laboratory-prepared wheat DDGS with varying CDS level (Mosqueda et al. 2013b) and that of plant-sourced wheat DDGS sample (Mosqueda et al. 2013c).

$$\frac{P}{C} = \frac{1}{d \cdot f} + \frac{P}{d} \quad (\text{A.4})$$

In Eq. A.4, P is the applied compressive pressure; C is the degree of volume reduction, $C = \frac{V_0 - V}{V_0}$ where V_0 is the volume of compact at zero pressure; V is volume of compact at pressure P (MPa); d and f are the model parameters. Model parameter d represents the porosity of the feed

material while the reciprocal of f (f^{-1}) relates to failure stress (MPa) of the pelleted sample (Mani et al. 2004).

A.3.3.5 Frictional properties

A tilting table apparatus was used to determine the coefficient of static friction of microwave vacuum-dried wheat DDGS samples on stainless steel, concrete, and wood. The angle at which the material begins to slide was recorded. The tangent of this angle represents the coefficient of static friction.

The coefficient of internal wall friction and cohesion was determined using the Wykeham Farrance shear box apparatus (Wykeham Farrance International Ltd., Slough, U.K.), described in detail by Mani et al. (2004) and by Emami and Tabil (2008). Four normal loads (200, 400, 600, and 1000 N) were used and the samples were sheared at a constant speed of $0.4 \text{ mm}\cdot\text{min}^{-1}$. Values of the internal friction coefficient and cohesion were obtained using Eq. A.5 (Mani et al. 2004; Emami and Tabil 2008), where τ is the shear stress (kPa), σ is the normal stress (kPa), θ is the angle of internal friction, $\tan \theta$ is the coefficient of internal friction, and C_c is cohesion (kPa).

$$\tau = \sigma \tan\theta + C_c \quad (\text{A.5})$$

A.3.3.6 Thermal properties

Thermal conductivity and thermal diffusivity of dried samples at ambient room condition were determined using a KD 2 Thermal Properties Analyzer (Decagon Devices, Inc., Pullman, WA). Bulk density of the samples was determined immediately before the thermal properties

measurement. A 0.5 L container was used to contain the sample for thermal properties measurement. Sample mass ranged from 219.0 to 265.2 g.

A.3.3.7 Comparison with a plant-sourced wheat DDGS sample

The physico-chemical characteristics of microwave vacuum-dried samples were compared with those obtained from a plant-sourced wheat DDGS sample (Mosqueda et al. 2013c). Both the WDGS sample used in microwave vacuum drying and the plant-sourced DDGS sample were obtained from the same ethanol plant on the same day. It was assumed that the plant-sourced and microwave vacuum-dried DDGS samples were derived from same WDGS batch in the ethanol plant.

A.3.4 Statistical analysis

Data were analyzed using the one-way analysis of variance, general linear model univariate, and regression procedures of SPSS 14.0 for Windows (SPSS Inc., Chicago, IL). Tukey's test was used to further evaluate statistically significant effects. All tests were conducted at the 0.05 significance level.

A.4 Results and Discussion

A.4.1 Drying characteristics

Figure A.2 shows the drying behavior of wheat DDGS, expressed in terms of moisture ratio over time. Drying time decreased as microwave power level was increased. With an initial moisture content of 70.7% (w.b.), it took about 42-45 min to dry about 300 g to 10.0-12.0% moisture

(w.b.) under P4 and about 21-24 min under P10. The figure also illustrates how the Page model (Eq. B.1) fitted the drying data generated under the four microwave power levels.

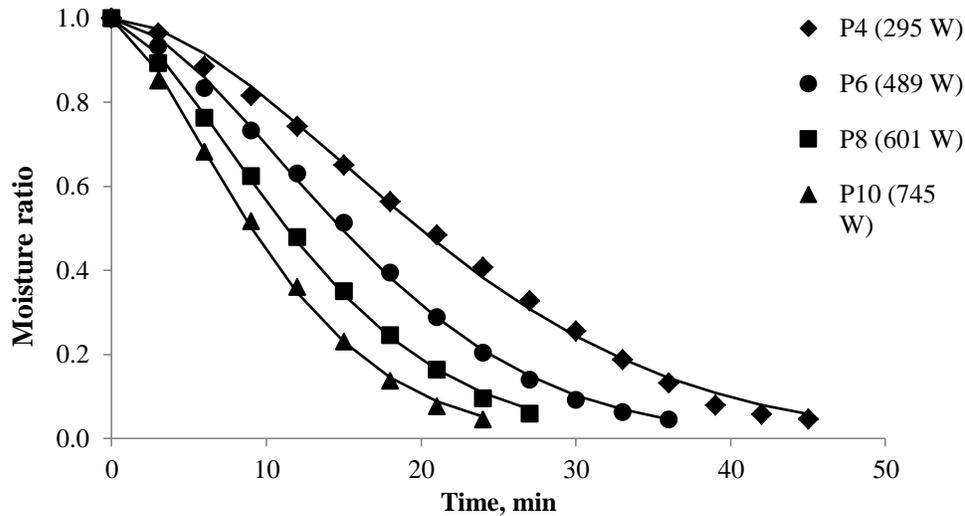


Fig.A.2 Drying behavior of microwave vacuum-dried wheat distillers grain with solubles under four microwave power levels at 88 kPa vacuum pressure. Initial sample mass was about 300 g.

Table A.1 presents the Page model parameters, k and N , and the statistical parameters used in testing goodness of fit. The high R^2 and small standard error of estimate values indicate that the model fitted very well with the experimental data. The model parameter k , which is the drying rate constant, varied from 0.005 to 0.026 min^{-1} and was found to increase linearly with microwave power level ($R^2 = 0.90$). Samples dried under P10 had significantly higher k values than those under P4 and P6. Higher values of k indicate steeper curves, and thus, faster drying. Values of the model parameter N , on the other hand, did not show significant difference.

A.4.2 Chemical composition

Table A.2 shows the proximate composition of the microwave vacuum (MWW)-dried and plant-sourced wheat DDGS samples. Protein, ash, fat, and fiber were expressed in dry matter basis

Table A.1 Page model parameters of microwave vacuum-dried wheat distillers grain with solubles. Values in parentheses represent standard deviation (n = 2)

Microwave power level	Page model parameters [*]		Coefficient of determination	Standard error of estimate
	k, min ⁻¹	N		
P4 (295 W)	0.005 (0.001) ^a	1.650 (0.114) ^a	0.997	0.019
P6 (489 W)	0.011 (0.006) ^{a,b}	1.591 (0.132) ^a	0.997	0.018
P8 (601 W)	0.017 (0.001) ^{a,b,c}	1.521 (0.038) ^a	0.998	0.016
P10 (745 W)	0.026 (0.001) ^{c,d}	1.509 (0.033) ^a	0.999	0.013

^{*}Tukey's test at 5% significance level. Values followed by the same letter/s are not significantly different

Table A.2 Proximate composition¹ of microwave vacuum (MWV)-dried and plant-sourced wheat distillers dried grain with solubles (DDGS). Values in parenthesis represent standard deviation (n = 2)

DDGS sample	Moisture, % wet basis	% dry matter					Acid detergent insoluble crude protein ²
		Protein	Ash	Fat	Neutral detergent fiber	Acid detergent fiber	
MWV-dried							
P4 (295 W)	10.52 (0.42)	37.94 (0.09) ^a	6.23 (0.01) ^a	6.36 (0.17) ^a	49.13 (2.21) ^a	15.71 (1.00) ^{a,e}	14.41 (0.52) ^a
P6 (489 W)	10.61 (0.05)	38.86 (0.04) ^{b,e}	6.26 (0.01) ^b	6.87 (0.37) ^a	48.07 (1.18) ^a	14.68 (0.16) ^{a,e}	11.20 (0.17) ^b
P8 (601 W)	10.82 (0.14)	38.40 (0.06) ^c	6.30 (0.01) ^c	7.24 (0.19) ^a	47.79 (0.08) ^a	13.43 (1.14) ^a	10.70 (0.80) ^b
P10 (745 W)	10.22 (0.24)	38.24 (0.08) ^c	6.05 (0.00) ^d	6.72 (0.04) ^a	47.54 (1.05) ^a	13.16 (0.47) ^a	10.87 (0.53) ^b
Plant-sourced ³	13.32 (0.23)	38.81 (0.14) ^e	7.10 (0.01) ^e	4.89 (0.18) ^e	46.55 (0.80) ^a	17.45 (0.27) ^e	18.37 (1.71) ^e

¹Tukey's test at 5% significance level. Tests were done in 2 stages: (1) differences between microwave power levels, (2) differences between MWV and plant-sourced samples. Values followed by the same letters are not significantly different

²Expressed as a % of crude protein, dry matter basis

³Source of data: Mosqueda et al. (2013b). The plant-sourced DDGS sample and the raw material used in microwave vacuum drying were obtained on the same day from the same ethanol plant.

while the acid detergent insoluble crude protein was expressed as a percentage of crude protein content, also in dry matter basis. The plant-sourced wheat DDGS sample and the wheat WDGS material used in microwave vacuum drying were obtained on the same day from the same ethanol plant.

Protein and ash contents of MWV-dried samples ranged from 37.9% to 38.9% and from 6.0% to 6.3%, respectively. Although there were statistical differences in the protein and ash content due to microwave power, these differences were numerically small as seen from the very narrow value ranges. These results were close to the protein (38.8%) and ash (7.1%) content reported for the plant-sourced DDGS sample, suggesting that both sample types were produced from the same source material. This was further supported by the NDF content of both samples. The NDF content of the MWV-dried DDGS was statistically similar with the plant-sourced sample, with the former about 2-6% higher than the latter.

No significant difference was found in the fat, NDF, and ADF content of microwave vacuum-dried samples due to microwave power level (Table A.2). Exposure to higher microwave power level for a shorter time produced the same effect on these chemical constituents as exposure to lower microwave power level for a longer time did. Fat content of the MWV-dried samples (6.4 - 7.2%), however, was about 30-48% higher than the plant-sourced sample (4.9%). High drying temperatures employed in the ethanol plant may have promoted lipid oxidation and lowered the fat content of the plant-sourced sample. While the study did not evaluate the free fatty acid composition of the samples, literature, however, indicated that increases in temperature accelerate the rate of lipid oxidation (Kolakowska 2002; Márquez-Ruiz et al. 2013), with deterioration of sensory quality (off odor, off flavor, discoloration) and nutritional value (losses

in polyunsaturated fatty acids and vitamins, protein damage) as among its many undesirable effects (Kolakowska 2002; Kolakowska and Bartosz 2013). Laboratory drying conditions employed in the study were milder compared to the drying conditions in the source ethanol plant. In the source ethanol plant, inlet and outlet temperatures of the ring dryer typically ranged from 255°C to 275°C and from 110°C to 130°C, respectively.

The ADICP content of MWV-dried samples ranged from 13.2% to 15.7% and was significantly affected by microwave power (Table A.2). Samples dried under the P4 setting had significantly higher ADICP content than those dried under the P6, P8, and P10 settings. Although drying under P4 subjected the samples to lower energy levels (and therefore, lower temperatures) compared to the other settings, drying time was longer, and thus, may have caused higher heat damage. These ADICP values (13.2-15.7%) were also 22-42% lower than that of the plant-sourced sample and were significantly different from the latter. These indicate that the MWV-dried samples had lower percentage of heat-damaged proteins compared to the plant-sourced sample.

Incidence of heat-damaged proteins is attributed to Maillard reaction, which is “a chemical reaction between amino groups and reducing sugars” (van Boekel 1998) affected by several factors including temperature and time (Ames 1990). Schroeder et al. (1996), for example, had reported increased acid detergent insoluble nitrogen (ADIN) in sunflower oilcake as processing temperature and time were increased. In their study, sunflower oil cake was roasted at 6 different temperature settings (110-210°C) and 5 time intervals (10-120 min). The combined effect of temperature and time on Maillard reaction was also demonstrated by Stamp and Labuza (1983). They plotted the extent of browning as a function of time for two model systems

(glucose/glycine and glucose/aspartame). To achieve a browning value of 0.1, the glucose/aspartame model system held at 80°C took about 5.3 h while the one held at 100°C took only about an hour. A similar trend was observed in the glucose/glycine model system, where the system held at 80°C took only 0.58 h while the one at 100°C took about 0.12 h to achieve the same extent of browning.

A.4.3 Physical properties

Table A.3 presents the particle size, bulk density, particle density, and color parameters of MWV-dried and plant-sourced wheat DDGS samples.

A.4.3.1 Particle size and size distribution

The average particle size of MWV-dried samples varied from 0.45 mm to 0.64 mm and was significantly affected by the microwave power level. Samples dried under the P4, P6, and P8 power levels had significantly larger particle sizes than those dried under P10. The P6-dried samples also had significantly larger particle sizes than those dried under P4. The particle sizes of P6- and P8-dried samples, as well as those dried under P4 and P8, were not significantly different.

Figure A.3 shows the particle size distribution of the samples. Those dried under P10 had higher percentage of particles passing through each of the sieves and thus, had finer particles than those dried under the lower microwave power levels. The P6-dried sample, on the other hand, was made up of coarser particles. The P4-, P6-, and P8-dried samples had particle sizes that were 28-49% larger than the plant-sourced sample and were significantly different from the latter. The

Table A.3 Physical attributes¹ of microwave vacuum (MWV)-dried and plant-sourced wheat distillers dried grain with solubles. Values in parenthesis represent standard deviation

Sample	Moisture, % wet basis	Particle size, mm	Bulk density, kgm ⁻³	Particle density, kgm ⁻³	Porosity, %	Color parameters		
						Lightness (L)	Redness (a)	Yellowness (b)
MWV-dried								
P4 (295 W)	11.80 (0.15)	0.55 (0.02) ^{a,c}	450.1 (5.0) ^a	1357.0 (1.6) ^a	66.8	44.6 (1.1) ^a	6.7 (0.2) ^a	14.9 (0.3) ^a
P6 (489 W)	11.99 (0.22)	0.64 (0.00) ^b	440.3 (3.4) ^{a,c}	1361.9 (1.1) ^b	67.7	44.8 (1.4) ^a	6.7 (0.1) ^a	15.2 (0.6) ^a
P8 (601 W)	12.18 (0.24)	0.58 (0.03) ^{b,c}	507.9 (1.0) ^c	1368.3 (0.4) ^c	62.9	42.9 (1.2) ^a	6.6 (0.1) ^a	14.3 (0.5) ^a
P10 (745 W)	12.00 (0.15)	0.45 (0.01) ^d	517.1 (4.6) ^c	1367.9 (1.1) ^c	62.2	43.6 (1.4) ^a	6.5 (0.2) ^a	14.3 (0.6) ^a
Plant-sourced ²	13.2 (0.0)	0.43 (0.02) ^c	437.0 (3.4) ^c	1320.5 (2.9) ^c	66.9	35.4 (0.0) ^c	7.7 (0.0) ^c	12.6 (0.0) ^e

¹Tukey's test at 5% significance level. Tests were done in 2 stages: (1) differences between microwave power levels, (2) differences between MWV and plant-sourced samples. Values followed by the same letters are not significantly different.

²Source of data: Mosqueda et al. (2013c)

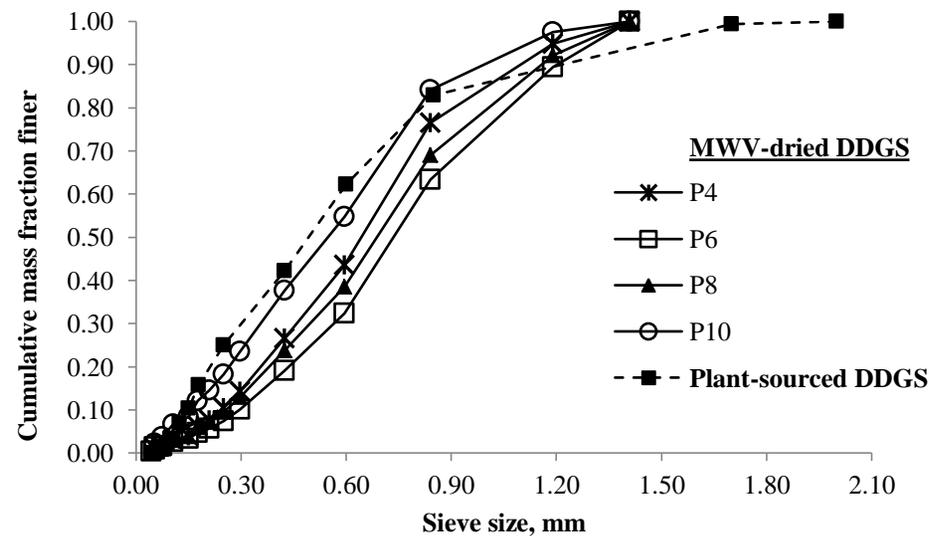


Fig. A.3 Particle size distribution of microwave vacuum (MWV)-dried and plant-sourced wheat distillers dried grain with solubles. MWV samples were dried under 4 microwave power levels, P4 (295 W), P6 (489 W), P8 (601 W), P10 (745 W) at 88 kPa vacuum.

particle size of the P10-dried sample was statistically similar to the particle size of the plant-sourced sample.

Particle size differences may be attributed to the changes in the microstructure of DDGS particles during drying. Studies have shown electromagnetic energy-based drying methods produced materials that have more porous and puffy microstructures than those which were air-dried (Lin et al. 1998; Krokida and Maroulis 1999; Witrowa-Rajchert and Rzaca 2009; Setiady et al. 2009). Chauhan and Srivastava (2009), in their study of microwave vacuum drying green peas at 100-300 W and 6.7-53.3 kPa vacuum pressure, reported that higher microwave power resulted to higher linear shrinkage, lower apparent density, and higher rehydration ratios, all indicating more porous structure of the dried product. In the wheat DDGS samples, drying under the highest microwave power setting (P10) produced significantly finer particles than drying under the lower power settings (P4, P6, P8) did. This could suggest that the more rapid evaporation of moisture under the P10 setting may have damaged or collapsed the cellular structure of the DDGS particles while drying under the lower microwave power settings did not damage the microstructure, thus resulting to puffier particles. Additional tests, however, would be necessary to verify this.

Differences could also be due to the way the bulk material was generated. The MWV-dried samples were passed through a knife mill equipped with a screen size that was about four times larger than the average particle size of the plant-sourced sample. These MWV-dried samples were ground using the same grinding time and similar feed amount. The plant-sourced DDGS sample, on the other hand, was dried using a ring dryer and was not ground.

A.4.3.2 Bulk and particle density

Differences in particle size led to differences in bulk and particle density values. This is shown in Table A.3 where bulk and particle density values were significantly affected by microwave power level. The P8- and P10-dried samples were significantly denser, and their porosity (Eq. B.4) values numerically lower, than the P4- and P6-dried samples. The sample dried under the P10 setting, for example, had smaller particle size compared to those dried under P4 and P6. In a heterogeneous mix of particles, the finer particles can easily penetrate the inter-particle spaces in a bulk material and thus, could translate to higher bulk density and lower porosity values.

A.4.3.3 Color

Values of the lightness (L), redness (a), and yellowness (b) parameters were 42.9 - 44.8, 6.5 - 6.7, and 14.3 - 15.2, respectively, and were not significantly affected by microwave power level (Table A.3). The L, a, b values of the MWV-dried samples were about 21-27% higher, 13-16% lower, and 13-21% higher than the plant-sourced sample, respectively. This means that the MWV-dried samples were lighter, less red, and more yellow than the plant-sourced wheat DDGS sample and could be attributed to milder drying conditions.

Color darkening is one of the consequences of Maillard reaction, which involves the binding of amino groups with the carbonyl compounds of reducing sugars (Owusu-Apenten 2004; Nyachoti et al. 2005; Widyaratne and Zijlstra, 2007). During the initial stages of Maillard reaction, colorless products are formed while highly colored polymers are formed during the final stages (Hodge 1953; Yaylayan and Roberts 2001). The rate of Maillard reaction is affected by such factors as temperature, time, water activity, and chemical composition (Owusu-Apenten 2004; Ames 1990). Higher drying temperature, for example, could increase the rate of Maillard

reaction and intensify color darkening. It had also been reported that microwave heating does not favor Maillard reaction (Ames 1990; Yaylayan and Roberts 2001; Reineccius and Whorton 1990). In conventional heated air drying, the surface of the material usually dries out first, producing a crust with a low water activity and favoring Maillard reaction, while in microwave heating, the lack of the hot, dry air surface surrounding the product does not favor crust formation and Maillard reaction, and thus, the development of colors and flavors may not occur (Ames 1990). The use of vacuum, which translates to lower drying temperature, further contributed to less color darkening.

A.4.3.4 *Compression characteristics*

Table A.4 shows the pellet density, specific energy consumption during compression and extrusion processes, and the values of the Kawakita – Ludde model parameters (Eq. B.4) for both MWV-dried and plant-sourced wheat DDGS samples.

For 1 g MWV-dried DDGS samples, pellets formed had average diameter and length of 6.7 mm and 23.1-23.6 mm, respectively. Equivalent pellet density values ranged from 1210.8 kg·m⁻³ to 1232.4 kg·m⁻³ and were not significantly affected by the microwave power level used in drying the feed material. The pellet density values were about 10-11% lower than the particle density values of bulk DDGS (Table A.4).

Specific energy consumption in compressing the MWV-dried DDGS to the pellet densities presented in Table A.4 varied between 7.7 MJ·t⁻¹ to 8.6 MJ·t⁻¹, accounting about 94-97% of the total specific energy consumption. Only the P6- and P8-dried materials showed significant difference in their specific energy consumption. The rest gave statistically similar results.

Table A.4 Compression characteristics¹ of microwave vacuum (MWV)-dried and ethanol plant-sourced wheat distillers dried grain with solubles. Values in parentheses represent standard deviation (n = 5)

Sample	Moisture, % wet basis	Pellet density, kgm ⁻³	Specific energy consumption, MJ·ton ⁻¹		Kawakita – Ludde model ²			
			Compression	Extrusion	d	f ¹	R ²	SEE
MWV-dried								
P4 (295 W)	11.80 (0.15)	1210.8 (10.9) ^a	8.29 (0.13) ^{a,e}	0.52 (0.11) ^a	0.66 (0.00) ^{a,e}	0.80 (0.03) ^{a,e}	1.00	0.39
P6 (489 W)	11.99 (0.22)	1215.1 (14.6) ^a	8.60 (0.57) ^{a,b,e}	0.34 (0.08) ^b	0.67 (0.00) ^b	0.87 (0.07) ^{a,b,c,e}	1.00	0.33
P8 (601 W)	12.18 (0.24)	1232.4 (5.1) ^a	7.73 (0.18) ^{a,c,e}	0.44 (0.06) ^{a,b,d}	0.63 (0.00) ^c	0.94 (0.03) ^{c,d,e}	1.00	0.38
P10 (745 W)	12.00 (0.15)	1215.2 (15.2) ^a	8.00 (0.52) ^{a,e}	0.28 (0.09) ^d	0.62 (0.00) ^d	0.97 (0.11) ^{b,d,e}	1.00	0.38
Plant- sourced ³	13.2 (0.0)	1208.2 (22.9) ^a	8.64 (1.10) ^e	2.54 (0.54) ^e	0.66 (0.01) ^e	0.96 (0.22) ^e	1.00	0.38

¹Tukey's test at 5% significance level. Tests were done in 2 stages: (1) differences between microwave power levels, (2) differences between MWV and plant-sourced samples. Values followed by the same letters are not significantly different.

² d and f¹ are the Kawakita-Ludde model parameters associated with initial porosity and failure stress, respectively. R² and SEE represent coefficient of determination and standard error of the estimate, respectively.

³Source of data: Mosqueda et al. (2013c).

Specific energy consumption during the extrusion process ranged from $0.28 \text{ MJ}\cdot\text{t}^{-1}$ to $0.52 \text{ MJ}\cdot\text{t}^{-1}$ and was also significantly affected by microwave power level. Significantly higher amount of energy was consumed in extruding the pellets made of the P4-dried material than when extruding pellets derived from P6- and P10-dried materials.

The Kawakita-Ludde model (Eq. B.4) adequately described the compression characteristics of the MWV-dried samples, similar to what was reported for wheat DDGS dried under forced air convection, microwave, and microwave convection conditions (Mosqueda et al. 2013b; 2013d). Values of the model parameter d , which represents initial porosity of the feed material, ranged from 0.62 to 0.67 and were similar to the bulk porosity values presented in Table A.3. These d values were significantly affected by microwave power level, wherein those dried under the lower microwave power levels being significantly more porous. The P4-dried DDGS, for example, was significantly more porous than those dried under P8 and P10. Similarly, the P6 was more porous than the P8 material, which, in turn, was also more porous than the P10-dried material. These differences were attributed to particle size differences, which consequently affected the density values.

In terms of the Kawakita-Ludde model parameter f^{-1} (Eq. B.4), which relates to failure stress, the values varied from 0.80 MPa to 0.97 MPa and was significantly affected by the microwave power. Samples dried under higher microwave power levels (P8, P10) produced significantly stronger pellets (higher failure stress) than those dried under lowest microwave power level (P4). The P10-dried DDGS, for example, was smaller, denser, and less porous compared to P4. These characteristics may have facilitated greater inter-particle contact area for bonding, thus, contributing to greater yield strength.

The plant-sourced DDGS was pelleted using a different process equipment and slightly different process parameters. The resulting pellet density, however, was statistically similar to the MWV-dried samples. Its compression characteristics were also adequately described by the Kawakita Ludde model (Eq. B.4), with a porosity value (model parameter d) similar to the one obtained from Eq. B.3 using bulk and particle density. Failure stress (model parameter f^{-1}) was statistically similar to those obtained from MWV-dried samples.

A.4.3.5 Frictional properties

Table A.5 presents the static friction coefficients of MWV-dried and plant-sourced wheat DDGS samples on three surfaces and the coefficient of internal friction of the MWV-dried samples. The static friction coefficients of MWV-dried samples were 0.33-0.39 on stainless steel, 0.47-0.50 on concrete, and 0.57-0.61 on wood. Microwave power level used in drying the samples and surface type significantly affected the values of the static friction coefficient. Surface type, however, was seen as having a more dominant influence on the observed variation, based on the ANOVA sums of squares distribution and F-values. MWV-dried samples on wood gave the highest static friction coefficient, followed by concrete and stainless steel.

Under the stainless steel and concrete surfaces, the plant-sourced wheat DDGS sample had significantly higher static friction coefficient than the MWV-dried samples. This could be due to differences in their moisture contents. The plant-sourced sample had a slightly higher content (14.0% w.b.) than compared to the MWV-dried samples (11.8 – 12.2%, w.b.).

Coefficient of internal friction ranged from 0.77 to 0.79 while cohesion varied from 2.92 kPa to 3.62 kPa (Table A.6) for the microwave vacuum-dried samples. These coefficients were higher compared to those reported for wheat DDGS samples that were prepared at 15-45% condensed

Table A.5 Frictional properties of microwave vacuum (MWV)-dried and ethanol plant-sourced wheat distillers dried grain with solubles. Values in parentheses represent standard deviation

Sample	Moisture, % wet basis	Static friction coefficient ¹			Coefficient	Internal friction ²		SEE
		Stainless steel	Concrete	Wood, parallel to grain		Cohesion, kPa	R ²	
MWV-dried								
P4 (295 W)	11.80 (0.15)	0.39 (0.01) ^{Aa}	0.50 (0.01) ^{Ba}	0.60 (0.02) ^{Ca,Ce}	0.77	2.92	0.999	0.80
P6 (489 W)	11.99 (0.22)	0.34 (0.02) ^{Aa,Ab}	0.50 (0.02) ^{Ba,Bb}	0.61 (0.01) ^{Ca,Cb,Ce}	0.79	3.62	0.992	2.09
P8 (601 W)	12.18 (0.24)	0.37 (0.02) ^{Aa,Ab}	0.47 (0.02) ^{Ba,Bb}	0.59 (0.02) ^{Ca,Cb,Ce}	NA	NA		
P10 (745 W)	12.00 (0.15)	0.33 (0.02) ^{Ab}	0.47 (0.02) ^{Bb}	0.57 (0.01) ^{Cb}	0.78	3.51	0.998	1.10
Plant-sourced ³	13.97	0.48 (0.01) ^{Ae}	0.54 (0.01) ^{Be}	0.61 (0.02) ^{Ce}	NA	NA		

¹Tukey's test at 5% significance level for the same microwave power level at different surfaces (A,B,C) and for the same surface at varying microwave power levels (a,b,c,d). Values followed by the same letters are not significantly different.

²R², SEE and NA represent coefficient of determination, standard error of the estimate, and "no data available", respectively.

³Source of data: Mosqueda et al. (2013d)

distillers solubles (CDS) level and dried under the microwave and microwave convection methods (0.63 – 0.74). Differences between the internal friction coefficients of the MWV-dried and the laboratory-prepared samples could be attributed to the level of condensed distillers solubles (CDS) incorporation during DDGS production. The laboratory-prepared samples had 15-45% CDS (wet basis) while MWV-dried samples were derived from a plant-sourced WDGS, believed to have CDS content higher than 45% (wet basis) (Mosqueda et al. 2013b). In corn DDGS, angle of internal friction varied from 12° to 54° for laboratory-prepared samples with varying solubles (10-25%, dry basis) and moisture (10-30%, dry basis) levels (Ganesan et al. 2008). For commercial corn DDGS, angles of internal friction between 31° to 52° were reported (Bhadra et al. 2009) reported angles of internal friction between for commercial corn DDGS. Hargreaves et al. (2010) reported lower angles of internal friction (14.4 – 28.9° or equivalent to 0.26 – 0.55 coefficient of internal friction) for superheated steam-dried DDGS samples derived from a mixture of corn and wheat stillage and prepared at varying CDS:WDG blending proportions.

A.4.3.6 Thermal properties

Thermal conductivity and diffusivity of MWV-dried samples were 0.05-0.06 W·m⁻¹K⁻¹ and 1.3-1.4 x 10⁻⁷ m²·s⁻¹, respectively (Table A.6). The thermal properties were not significantly affected by the power levels used during drying. The MWV-dried and the plant-sourced samples were also not significantly different in their thermal properties. These could be attributed to almost similar porosity values. Porosity of the MWV-dried samples ranged from 62% to 68% while that of plant-sourced sample was about 67% (Table A.3). These values were also close to those reported for commercial corn DDGS. Thermal conductivity and thermal diffusivity values of

commercial corn DDGS varied from 0.06 to 0.08 $\text{W}\cdot\text{m}^{-1}\text{K}^{-1}$ and from 1.30×10^{-7} to 1.50×10^{-7} $\text{m}^2\cdot\text{s}^{-1}$, respectively (Rosentrater 2006).

Table A.6. Thermal properties of microwave vacuum (MWV)-dried and plant-sourced wheat distillers dried grain with solubles at 23°C

Sample	Moisture, % wet basis	Bulk density, kgm^{-3}	Thermal conductivity, $\text{Wm}^{-1}\text{K}^{-1}$	Thermal diffusivity, $\times 10^{-7} \text{m}^2\cdot\text{s}^{-1}$
MWV-dried				
P4 (295 W)	11.80 (0.15)	455.4 (5.9)	0.06 (0.00) ^a	1.3 (0.0) ^a
P6 (489 W)	11.99 (0.22)	440.7 (4.4)	0.05 (0.01) ^a	1.4 (0.0) ^a
P8 (601 W)	12.18 (0.24)	514.5 (1.8)	0.06 (0.00) ^a	1.3 (0.0) ^a
P10 (745 W)	12.00 (0.15)	529.6 (0.9)	0.06 (0.00) ^a	1.3 (0.0) ^a
Plant-sourced ²	13.2 (0.0)	437.0 (3.4)	0.05 (0.01) ^a	1.4 (0.1) ^a

¹Tukey's test at 5% significance level. Values followed by the same letters are not significantly different.

²Source of data: Mosqueda et al. (2013c).

A.5 Conclusions

This study investigated the drying kinetics and physico-chemical characteristics of wheat wet distillers grain with solubles (WDGS) dried under microwave vacuum conditions. The Page model adequately described the drying characteristics of wheat DDGS under microwave vacuum. Drying rate increased with microwave power level. Drying times for 300 g samples under MWV drying ranged from 21 to 45 min.

Increases in microwave power level significantly increased the bulk density and decreased the bulk porosity of the granular material. It also significantly increased the failure stress of the pelleted DDGS and decreased the specific energy consumption in removing the pellet out of the die.

Microwave-vacuum dried wheat DDGS samples had lower amount of heat-damaged proteins, higher fat content, and were significantly lighter in color compared to the commercial sample.

Careful selection of drying process conditions to optimize energy efficiency and protein quality and an economic evaluation of its incorporation in fuel ethanol plants would be necessary.

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Appendix B

Sample calculations: Techno-economic evaluation of drying wheat distillers grain with solubles using microwave energy

Model Plant: 25 million liters per year (MLPY)

Scenarios: Rotary drying (base case, low efficiency)
Microwave drying (scenario 1, high efficiency)

B.1 Mass flow rates

B.1.1 Assumptions

Item	Value
1. Utilization, % of nameplate capacity	92
2. Annual operating hours	8000
3. Product generation from 1 t of wheat grain	
a. Ethanol, L	375
b. DDGS, kg	370
4. Moisture content, % wet basis	
a. WDGS entering the dryer	70
b. DDGS leaving the dryer	10

B.1.2 Calculations

1. Amount of wheat DDGS produced per hour, m_d

$$m_d = \frac{\text{Nameplate cap.} * \% \text{ utilization}}{\text{Annual operating hours}} * \frac{\text{DDGS produced per ton of grain}}{\text{Ethanol produced per ton of grain}}$$

$$m_d = \frac{(25 \times 10^6 \text{ L}) * 0.92}{8000 \text{ h}} * \frac{370 \text{ kg}}{375 \text{ L}} = 2836.7 \text{ kg/h}$$

2. Amount of wheat WDGS entering the drying system per hour, m_p

$$m_p = \frac{(m_{DDGS} - m_{DDGS} * \text{DDGS final moisture})}{1 - \text{WDGS initial moisture}}$$

$$m_p = \frac{(2836.7 \text{ kg/h} - (2836.7 \text{ kg/h} * 0.10))}{1 - 0.70} = 8510.1 \text{ kg/h}$$

3. Total amount of water to be evaporated per hour, m_w

$$m_w = m_p - m_{DDGS} = 8510.1 \text{ kg/h} - 2836.7 \text{ kg/h} = 5673.4 \text{ kg/h}$$

B.2 Energy requirement

B.2.1 Assumptions

Item	Value
1. Temperature of WDGS entering the drying process, °C	80
2. Product temperature during drying, °C	
a. Rotary drying	250
b. Microwave drying	100
3. Rotary drying-related efficiencies	
a. Dryer thermal efficiency, e_D	0.50
b. Combustion efficiency, e_C	0.85
4. Microwave drying-related efficiencies	
a. Applicator efficiency, e_A	0.95
b. Generator efficiency, e_G	0.85

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B.2.2 Calculations

1. Energy requirement under rotary drying

a. Total heat load, E_T

$$E_T = m_w L_v + m_p c_p (T_f - T_i)$$

$$E_T = [(5673.4 \text{ kg/h} \times 2260 \text{ kJ/kg}) + 8510.1 \text{ kg/h} \times 3.21 \text{ kJ/kg}^\circ\text{C} (250^\circ\text{C} - 80^\circ\text{C})] \times \frac{1\text{h}}{3600 \text{ s}}$$

$$E_T = 4851.6 \text{ kW}$$

b. Energy input for drying

$$E_{RD} = \frac{E_L}{e_D} = \frac{4851.6 \text{ kW}}{0.50} = 9703.2 \text{ kW}$$

c. Energy requirement to be supplied by natural gas

$$E_{NG} = \frac{E_{RD}}{e_C} = \frac{9503.2 \text{ kW}}{0.85} = 11415.5 \text{ kW}$$

d. Total energy requirement, E_T

$$E_T = E_{NG} + E_{\text{Electric motors}} = 11415.5 \text{ kW} + 168.75 \text{ kW} = 11584.2 \text{ kW}$$

2. Energy requirement under microwave drying

a. Total heat load, E_T

$$E_T = m_w L_v + m_p c_p (T_f - T_i)$$

$$E_T = [(5673.4 \text{ kg/h} \times 2260 \text{ kJ/kg}) + 8510.1 \text{ kg/h} \times 3.21 \text{ kJ/kg}^\circ\text{C} (100^\circ\text{C} - 80^\circ\text{C})] \times \frac{1\text{h}}{3600 \text{ s}}$$

$$E_T = 3713.4 \text{ kW}$$

b. Energy input for drying (in applicator/ovens)

$$E_G = \frac{E_T}{e_A} = \frac{3713.4 \text{ kW}}{0.95} = 3908.8 \text{ kW}^*$$

*This was later adjusted to 3950 kW to suit commercially available microwave generator sizes. See B.3.2, item 2d.

c. Energy requirement supplied from source to microwave generators

$$E_S = \frac{E_G}{e_G} = \frac{3950 \text{ kW}}{0.85} = 4647 \text{ kW}$$

3. Energy requirement under booster and finish drying

This requirement is calculated by combining items 1 and 2 above. The only change is the amount of material to be dried under each drying method (rotary and microwave drying).

B.3 Equipment size and number

B.3.1 Assumptions

Item	Value
1. Rotary drying	
a. Evaporation rate per m ² , kg/h	14.64
b. Commercially available sizes	1.8 x 14; 2.2 x 18; 2.4 x 20; 2.8 x 24
2. Microwave drying system	
a. Range of available sizes	75 kW – 1 MW
b. Size of microwave generators	75 kW, 100 kW
c. No. of microwave generators per oven	2

B.3.2 Calculations

1. *Size and number of rotary dryers*

a. *Area of rotary dryer required, A_{RD}*

$$A_{RD} = \frac{\text{Amount of water to be evaporated per hour}}{\text{Evaporation rate per unit area of dryer}}$$

$$A_{RD} = \frac{5673.4 \text{ kg/h}}{14.64 \text{ kg/h-m}^2} = 387.53 \text{ m}^2$$

b. *Dryer length (L), if diameter (D) is 2.4 m*

$$L = \frac{A_{RD}}{\pi D} = \frac{386.53 \text{ m}^2}{\pi(2.2 \text{ m})} = 56.07 \text{ m}$$

c. *Number of rotary dryers, N_{RD}*

Commercial size chosen: 2.4 m x 20 m

$$N_{RD} = \frac{\text{Required dryer length}}{\text{Commercially available length}} = \frac{56.07 \text{ m}}{20 \text{ m}} \approx 3 \text{ units}$$

2. Size and number of microwave drying units

a. Energy requirement at the microwave applicators/ovens

$$E_G = \frac{E_L}{e_A} = \frac{3713.4 \text{ kW}}{0.95} = 3908.8 \text{ kW}$$

b. No. of microwave drying units

$$\text{No. of 1 MW units} = \frac{3908.8 \text{ kW}}{1000 \text{ kW}} = 3$$

$$\text{No. of 800 kW units} = \frac{908.8 \text{ kW}}{800 \text{ kW}} = 1$$

$$\text{No. of 150 kW units} = \frac{108.8 \text{ kW}}{150 \text{ kW}} \approx 1$$

Total energy supplied = 3950 kW

c. No. of microwave generators

$$\text{No. of 100 kW generators} = \frac{3800 \text{ kW}}{100 \text{ kW}} = 38$$

$$\text{No. of 75 kW generators} = \frac{150 \text{ kW}}{75 \text{ kW}} = 2$$

Total no. of microwave generators = 40

d. No. of waveguide assembly units = no. of generators = 40

e. No. of applicators/ovens

$$\text{No. of ovens} = \frac{\text{no. of generators}}{2 \text{ generators per oven}} = \frac{40}{2} = 20$$

B.4 Equipment costs

B.4.1 Assumptions

Item	Value
1. Installation cost, % of purchase price	40
2. Foreign exchange rate, US\$: CAN \$	1:1.03
3. Cost of microwave drying unit, US\$	
a. 1 MW unit	1,200,000.00
b. 75 kW unit	150,000.00
4. Rotary drying unit	
a. Drying gas factor (direct contact), f_g	0.12
b. Material factor (stainless steel), f_m	1.4
5. Annual interest rate, %	15
6. Economic life, L, years	10
7. Salvage value, S_v	0
8. Annual operating hours, t	8000
9. Other costs:	
a. US sales tax	4%
b. Canada custom duties	8%
c. Canadian Goods & Services Tax (GST)	5%
d. Freight cost per mile	
i. Within USA, US\$	2.56
ii. Within Canada, CAN\$	2.99
10. Equipment cost capacity factor, x	0.60
11. Distance (New Orleans, LO to Unity, SK), miles	2245

B.4.2 Calculations

1. *Initial cost of the microwave drying units*

$$a. \text{ Cost of 3 – 1 MW units} = \frac{\text{US \$1200000}}{\text{unit}} * 3 \text{ units} = \text{US\$ 3,600,000}$$

b. *Cost of 950 kW unit, I_{950}*

$$I_2 = I_1 * (Q_2 / Q_1)^x$$

$$I_{950\text{kW}} = 1200000 * (0.95\text{MW} / 1\text{MW})^{0.6} = \text{US\$1,163,631}$$

c. Total, in CAN\$

$$C_{MD} = (\text{US\$ } 3600000 + 1163631) * \frac{1.03 \text{ CAN\$}}{1 \text{ US\$}} = \text{CAN\$ } 4,906,541.00$$

2. Initial cost of the rotary drying units

$$C_{RD} = 1.218(1 + f_g + f_m) * \exp(4.9504 - 0.5827 \ln(A_s) + 0.0925 (\ln(A_s))^2), 1000 \text{ US\$}$$

A_s = dryer lateral surface area, sq. ft = 1622.33

$$C_{RD} = \left[1.218(1 + 0.12 + 1.4) * \exp(4.9504 - 0.5827 \ln(1622.33) + 0.0925 (\ln(1622.33))^2) \right]$$

$$C_{RD} = \text{US\$ } 914,707.74 * 3 \text{ units (see B.3.2 item 1d)} = \text{US\$ } 2744123.21$$

$$C_{RD} = \text{US\$ } 2744123.21 * \frac{1.03 \text{ CAN\$}}{1 \text{ US\$}} = \text{CAN\$ } 2826446.90$$

3. Other costs (calculations for microwave drying units only)

a. Freight costs

Transporting microwave drying units from the US to SK using 40 ft container vans)

$$\text{No. of vans needed} = \frac{\text{no. of ovens to be transported}}{\text{length of van/length of oven}} + 20\% \text{ allowance}$$

$$\text{No. of vans needed} = \frac{20}{40 \text{ ft}/12 \text{ ft}} * 1.20 \approx 8$$

$$\text{Freight cost} = \text{Cost per mile} * \frac{\text{no. of miles (return)}}{\text{van}} * \text{no. of vans} = \text{CAN\$ } 74,185.34$$

b. US sales tax, Canadian custom duties and GST

$$\text{Sales tax and custom duties} = \text{Initial cost (US \$)} * \% \text{ US sales tax} * \frac{1.03 \text{ CAN \$}}{1 \text{ US \$}}$$

$$+ \text{initial cost (CAN\$)} * \text{Canadian custom duties \& GST rate}$$

$$\text{Sales tax and custom duties} = \text{CAN\$ } 828,395.61$$

4. Installation cost

$$\text{Installation cost} = \text{Initial cost} * 40\% = \text{CAN\$ } 1,962,616.40$$

5. Total equipment cost, C_E (microwave drying)

$$C_E = \text{Initial cost of equipment} + \text{other costs} + \text{installation cost}$$

$$C_E = 4,906,541.00 + (74,185.34 + 828,395.61) + 1,926,616.40 = \text{CAN\$ } 7,771,738.35$$

6. Depreciation cost per hour, C_{DEP} (microwave drying)

$$C_{DEP} = \frac{(C_E - S_V) * (1+i)}{L_E * t_A} = \frac{(7771738.35 - 0) * 1.15}{10 * 8000} = \text{CAN\$ } 111.72$$

B.5 Operating costs

B.5.1 Assumptions

Item	Value
1. Electricity, CAN\$ per kWh	0.082
2. Cooling water	
a. Cost, CAN\$ per m ³	0.6201
b. Tax rate, %	7
c. Consumption, L·min ⁻¹ per generator	30
3. Natural gas, US\$ per million BTU	3.70
4. Magnetron replacement	
a. Life, h	8000
b. Cost, including freight and taxes, CAN\$	
i. 75 kW	8485
ii. 100 Kw	9806
5. General maintenance, % of purchase price	2
6. Personnel	
a. Number of personnel	10
b. Average annual salary, CAN\$	90,000
c. Share of drying process in salaries, % of total	10
d. Training, CAN\$ per person per year	1800

B.5.2 Calculations

1. Operating costs for microwave drying, CAN\$ per hour

a. Electricity, C_P

Power required from main supply line = 4647 kW

$$C_P = 4647 \text{ kW} * \frac{\text{CAN\$ } 0.082}{\text{kWh}} = \text{CAN\$ } 381.06$$

b. Water, C_W

$$C_W = \frac{30 \text{ Lpm}}{\text{generator}} * \frac{1 \text{ m}^3}{1000 \text{ L}} * \frac{60 \text{ min}}{1 \text{ h}} * \frac{\text{CAN\$ } 0.6201}{\text{m}^3} * 20 \text{ generators} * 1.07$$

$$C_W = \text{CAN\$ } 50.40$$

c. Magnetron replacement, C_M

$$C_M = \frac{N_{75\text{kW}} * U_{75\text{kW}} + N_{100\text{kW}} * U_{100\text{kW}}}{t}$$

$$C_M = \frac{2 \text{ units} * \frac{\text{CAN\$ } 8485}{\text{unit}} + 38 \text{ units} * \frac{\text{CAN\$ } 9806}{\text{unit}}}{8000 \text{ h}} = \text{CAN\$ } 48.24$$

d. General maintenance cost, C_{GM}

$$C_{GM} = \frac{0.02 * \text{Initial equipment cost}}{t} = \frac{0.02 * \text{CAN\$ } 4,906,541.00}{8000 \text{ h}} = \text{CAN\$ } 17.17$$

e. Personnel salaries and training cost, $C_{S\&T}$

$$C_{S\&T} = \frac{0.10 * \left(10 \text{ employees} * \frac{\text{CAN\$ } 90000}{\text{employee}} \right) + \left(10 \text{ employees} * \frac{\text{CAN\$ } 1800}{\text{employee}} \right)}{8000 \text{ h}}$$

$$C_{S\&T} = \text{CAN\$ } 13.50$$

2. Operating costs for rotary drying, CAN\$ per hour

a. Cost of natural gas, C_{NG}

Energy to be supplied by natural gas = 11415.45 kJ·s⁻¹

$$C_{NG} = 11415 \frac{\text{kJ}}{\text{s}} * \frac{\text{US\$ } 3.70}{1 \times 10^6 \text{ BTU}} * \frac{1 \text{ BTU}}{1.055 \text{ kJ}} * \frac{\text{CAN\$ } 1.03}{\text{US\$ } 1} * \frac{3600 \text{ s}}{1 \text{ h}} = \text{CAN\$ } 148.45$$

b. Other operating costs (electricity, general maintenance, and personnel salaries) are computed in the same manner as presented under item 1 above.

B.6 Converting cost per hour to cost per tonne

$$\text{Cost per tonne DDGS} = \frac{\text{Cost}}{\text{hour}} * \frac{\text{hour}}{\text{kg of DDGS produced}} * \frac{1000 \text{ kg}}{1 \text{ tonne}}$$

$$\text{Depreciation cost per tonne DDGS} = \frac{\text{CAN\$ } 111.72}{\text{hour}} * \frac{\text{hour}}{2836.7 \text{ kg DDGS}} * \frac{1000 \text{ kg}}{1 \text{ tonne}} = \text{CAN\$ } 39.38$$

B.7 Cost Summary

Cost	Drying cost, CAN\$			
	Rotary (Base case, low efficiency)		Microwave (Scenario 1, high efficiency)	
	Per hour	Per tonne DDGS	Per hour	Per tonne DDGS
1. Depreciation	63.96	22.55	111.72	39.38
2. Electricity	13.84	4.88	381.06	134.33
3. Water	-	-	50.40	17.77
4. Magnetron replacement	-	-	48.24	17.01
5. Natural gas	148.45	52.33	-	-
6. General maintenance	49.46	17.44	17.17	6.05
7. Personnel	11.25	3.97	13.50	4.76
Total cost	286.96	101.16	622.09	219.30

Appendix C: Case study details

C.1 Interview Guides

1. Brewery personnel

SUPPLY

- a. No. of brews per week
 - i. Peak months
 - ii. Lean months
 - iii. Average
- b. Amount of spent grain generated per brew
- c. How spent grain is disposed to buyers (schedule, procedure overview)

BUYERS

- a. Requirements to be able to buy spent grain
- b. Common spent grain buyers and where they come from

2. Livestock farmers

ANIMALS RAISED

a. Number of dairy cattle raised

- i. Adults _____
- ii. Young _____

b. Other animals (kind and number)

Kind	Number
_____	_____
_____	_____
_____	_____

c. Where/how present stock was procured

d. Where/how animals/animal products (milk) are sold/marketed

MAIN SOURCE OF TECHNICAL SERVICES SUPPORT

City Veterinary Office _____ Others, pls specify _____

BREWERS SPENT GRAIN (BSG) SUPPLY

a. How spent grain was obtained from brewery

b. Frequency of purchase _____

c. Quantity procured for each purchase _____

d. Cost:

- i. Spent grain _____
- ii. Transportation (vehicle, gas, labor) _____

STORAGE AND DRYING OF BSG

- a. How BSG is stored

- b. Is drying done? (If yes, how? If no, why not?)

COMMON FEED SOURCES AND PRACTICES

- a. What are the common feed/feed types used and where these were sourced?

- b. Describe how spent grain is prepared as feed (mixed it with other feeds? use in wet/dry form?)

MAIN ADVANTAGES AND DISADVANTAGES OF USING BREWERS SPENT GRAIN

- a. Advantages

- b. Disadvantages

3. Markets

LIVESTOCK AUCTION MARKET

- a. Parties involved in its overall management
- b. Overview of transactions and procedures
 - i. Requirements for participation (sellers, buyers, middlemen)
 - ii. Types and volume of animals traded
 - iii. Procedures: from receipt of animals until these are sold
- c. Buyers and sellers
 - i. Where they come from

DAIRY PROCESSING PLANT

- a. Overview
 - i. Northern Mindanao Federation of Dairy Cooperatives
 - ii. Dairy processing plant operations, its products, markets
- b. Dairy farmer-suppliers
 - i. Where they come from
 - ii. Milk supply arrangements (delivery schedule, buying price, standard procedures)

C.2 Details of the prototype dryer for brewers spent grain

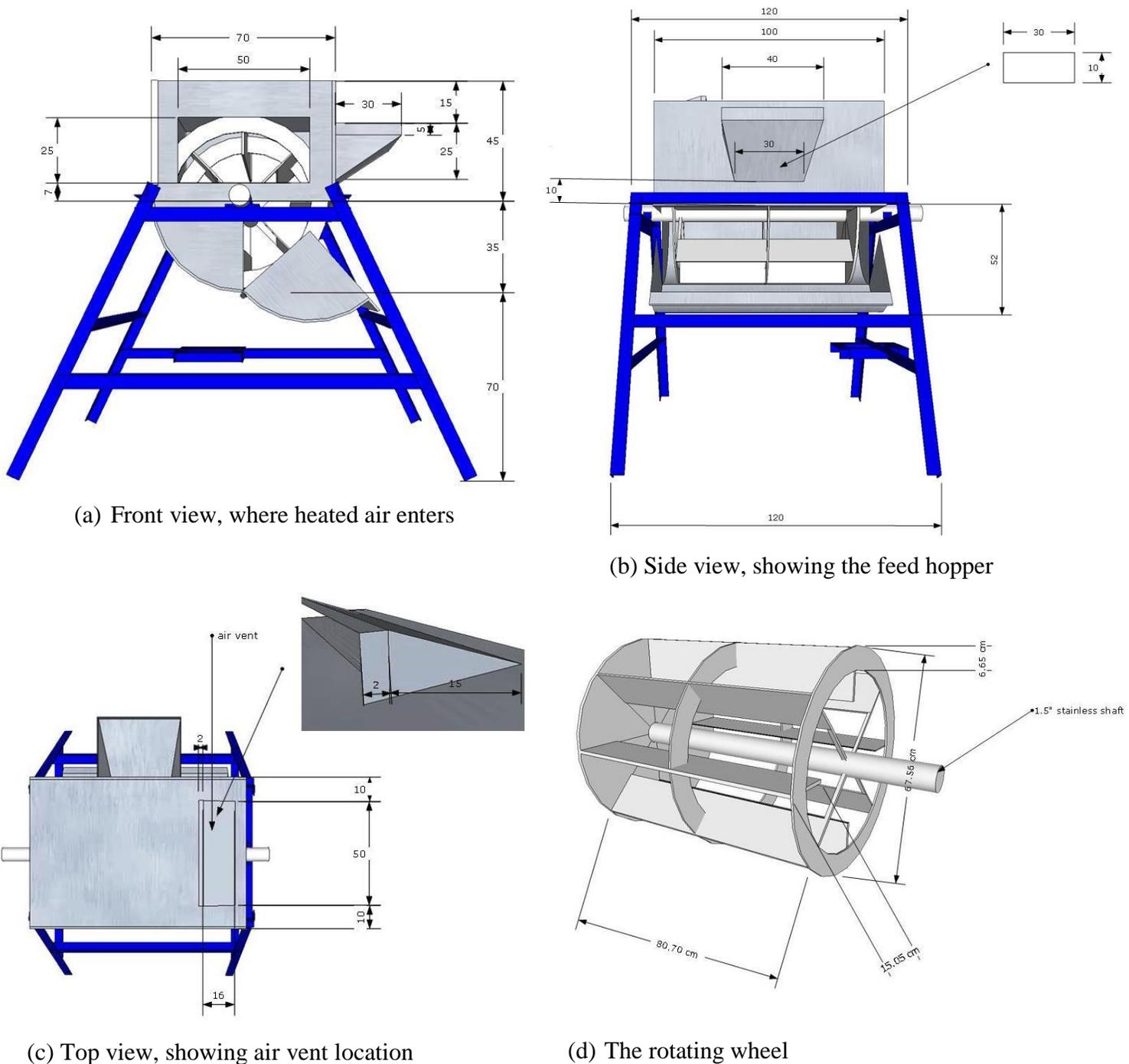
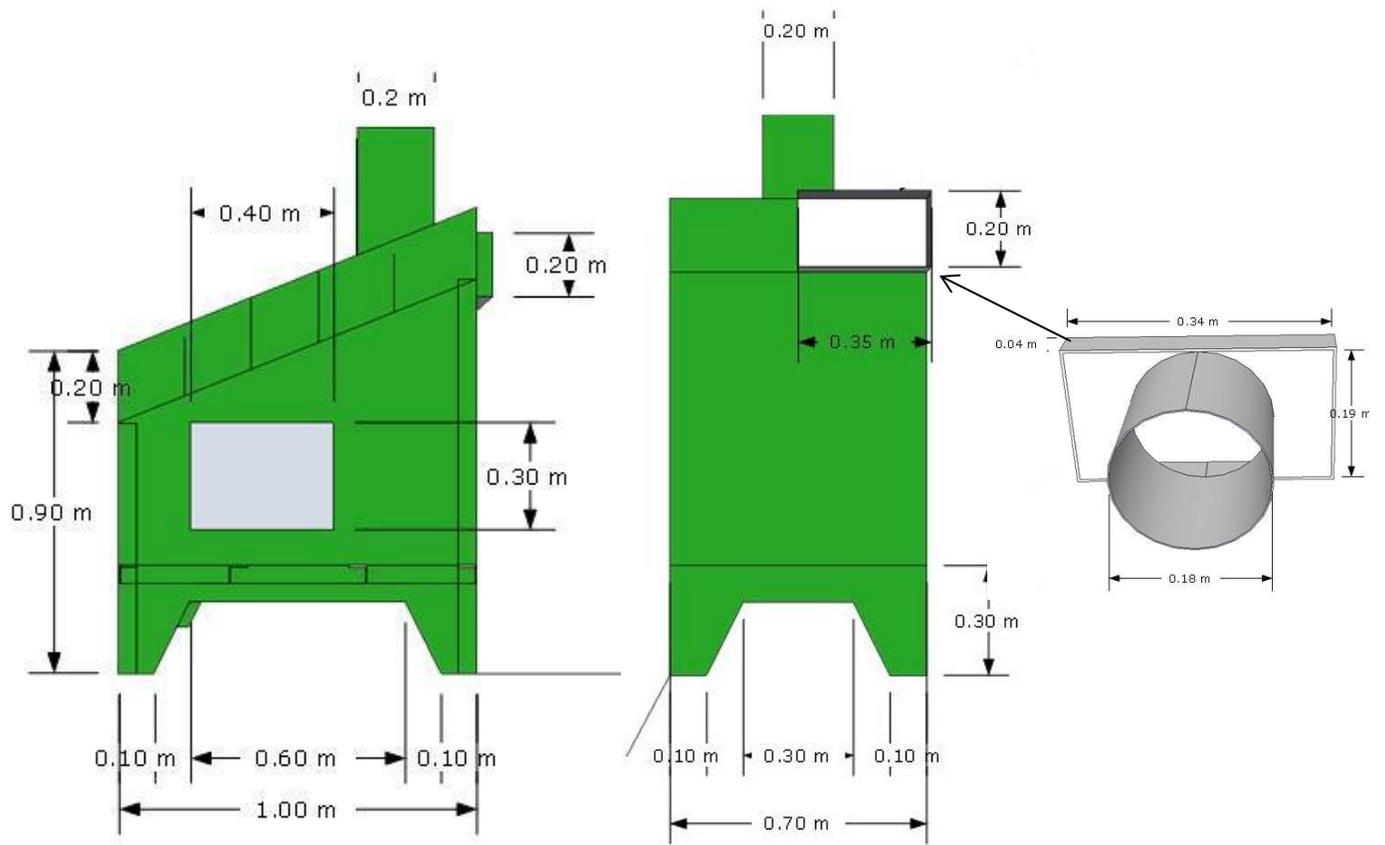
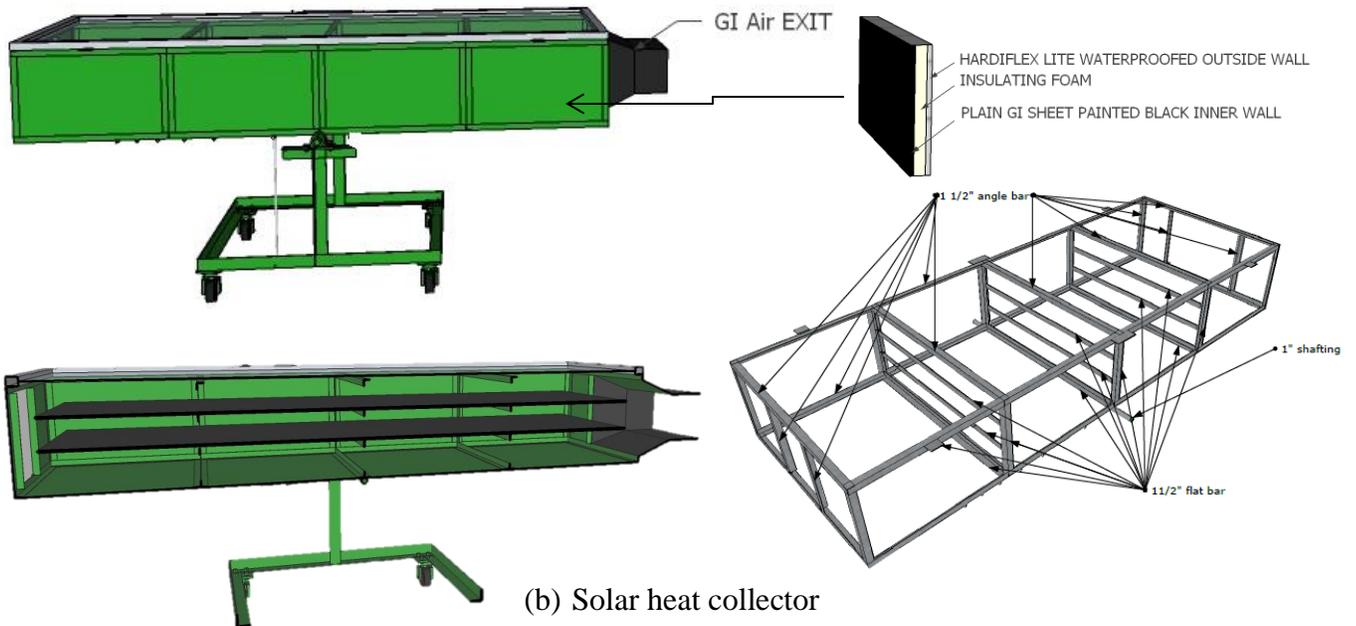


Fig. C.2.1 Details of the drying chamber developed to dry brewers spent grain and similar bulk materials. Measurement data reflected are in terms of centimeters.



(a) Furnace



(b) Solar heat collector

Fig. C.2.2 Details of the air heating systems used in conjunction with the drying chamber: (a) biomass furnace, and (b) solar heat collector.

C.3 The livestock auction market in El Salvador City, Misamis Oriental, Philippines

Figure C.3.1 shows a typical day at the livestock auction market, where local farmers, other sellers, and buyers from various parts of the province, Mindanao, and even as far as Luzon converge every Wednesday. The livestock auction market, which started in 1952, is managed by *barangay* (village) Cogon and supported by the city government of El Salvador.



Fig. C.3.1 The livestock auction market in Barangay Cogon, El Salvador City, Misamis Oriental, Philippines, October 2012.

Sales volume. Figure C.3.2 shows the monthly sales volume for cattle from Jun 2011 – Jul 2012. Most of those sold are destined for slaughter and for farm work, accounting about 66% of the total volume. Cattle sold for fattening or breeding comprised the remaining 34% of the volume. Average selling prices per kg for cattle varied, depending on the intended use of the animal. For Jul 2011 – Jun 2012, the average prices per kg were PHP 72.81 (for slaughter), PHP 83.01 (for work), PHP 85.73 (for fattening) and PHP 85.11 (for breeding). While cattle were the most traded animals, swine, chicken, horse, goat, and *carabao* (domestic water buffalo, *Bubalus bubalis*) were also sold in the market.

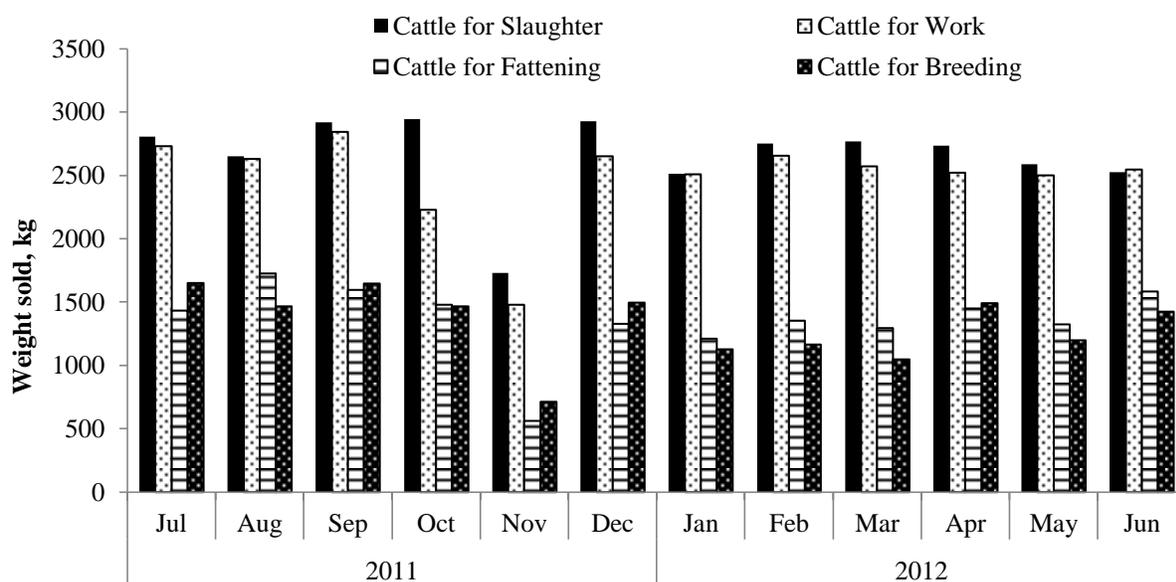


Fig. C.3.2 Categories of cattle sold monthly at the El Salvador Livestock Auction Market, Misamis Oriental, Philippines, from Jul 2011 to Jun 2012 (Source: Barangay Cogon, El Salvador City, Misamis Oriental, 2012).

Sources of cattle. Figure C.3.3 shows that about 46% of the cattle brought to the livestock auction market in 2011 came from El Salvador City. About 50% came from neighboring municipalities of Alubijid, Laguindingan, Gitagum, and Opol and Cagayan de Oro City. The remaining 4% came from other municipalities of Misamis Oriental, Lanao del Norte, and Bukidnon. Average weight of cattle brought in ranged from 35 kg to 520 kg, while that of carabao ranged from 70 kg to 580 kg. Body weights of small animals like swine, goat, and chicken were not measured when these are brought inside the auction market.

Market participants. *Barangay* (village) Cogon provides the overall management of the auction market, such as ticket issuance (sellers with large animals are collected PHP 10.00 per head brought to the market), animal weighing, and peace and order maintenance. The treasurer's office of the El Salvador city government, on the other hand, facilitates on-site transfer of animal

ownership. Sellers include both the farmers and the ‘*capitalistas*’ who are both sellers and buyers at the same time. These *capitalistas* buy from individual farmers and sell their stock to other auction markets, to supermarkets and public markets in Cagayan de Oro, or during the next auction market day in El Salvador. The *cabidors* act as middlemen between the buyers and sellers. At the time of the visit, there were 47 *capitalistas* and 145 *cabidors* registered with barangay Cogon. Buyers that served customers outside of Cagayan de Oro and Misamis Oriental are also present. These buyers serve Bukidnon, Iligan City, Agusan del Norte, Agusan del Sur, Surigao del Norte, Surigao del Sur, Zamboanga del Norte, Zamboanga del Sur, and even as far as Batangas and Pangasinan provinces in Luzon.

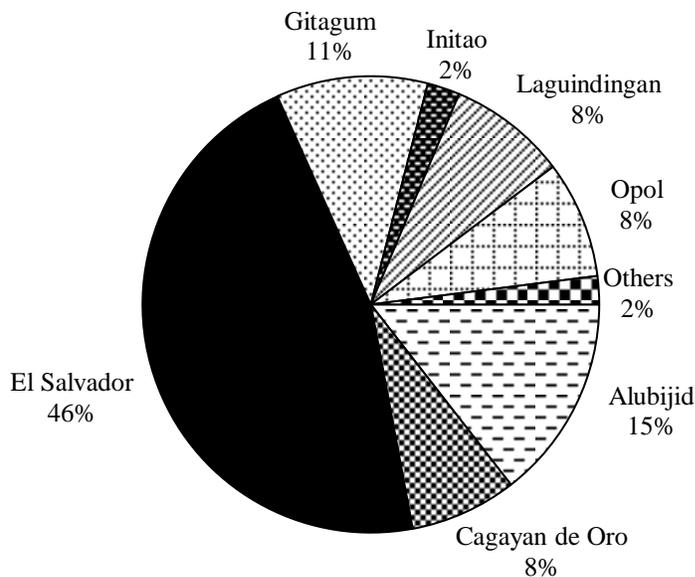


Fig. C.3.3 Sources of cattle brought to the El Salvador Livestock Auction Market, Jan – Dec 2011 (Source: Barangay Cogon, El Salvador City, Misamis Oriental, 2012).

Appendix D

Permission from companies to use selected diagrams

1. From GEA Barr-Rosin

The diagrams appeared as Figs. 1.3 and 7.2 (rotary drying system) and Fig. 1.5 (ring dryer) in the report.

RE: Query - Ring Dryer for Wheat Based DDGS - University of Saskatchewan

Dee, Adrian [adrian.dee@gea.com]

Sent: Tuesday, November 12, 2013 11:23 AM

To: Mosqueda, Maria Rosario

Cc: Kanellis, Kosta [kosta.kanellis@gea.com]; Chartrand, Francis [francis.chartrand@gea.com]; Skaarenborg, Mads [mads.skaarenborg@gea.com]

Attachments: 244-GSBCX-01.pdf (433 KB) ; 244-GSBCX-01.jpg (497 KB) ; 144-GDBCX-02.jpg (492 KB) ; 144-GDBCX-02.pdf (433 KB)

Maria,

Please find pdf and jpg versions of both the requested flow diagrams attached herewith.

Please label the drawings with:

Copyright © 2013 GEA Barr Rosin. All rights reserved.

Please also send me a copy of your paper to review before publishing.

If I can be of any further assistance in the meantime, please do not hesitate to contact me.

Best regards

Adrian Dee
Market Manager, GEA Barr-Rosin

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From: Mosqueda, Maria Rosario [mailto:mrm548@mail.usask.ca]
Sent: November/12/13 10:06 AM
To: Dee, Adrian
Subject: RE: Query - Ring Dryer for Wheat Based DDGS - University of Saskatchewan

Hi Adrian,

I would like to request permission to use the following diagrams that appeared in the 'Dryhouse technologies and DDGS production' chapter (Chapter 21 of the Alcohol Textbook, 5th ed):

Figure 22 - Rotary direct-fired PGR dryer

Figure 23 - Ring PGR dryer

Thanks very much again for your generous assistance.

Maria

From: Dee, Adrian [adrian.dee@gea.com]
Sent: Tuesday, November 12, 2013 8:36 AM
To: Mosqueda, Maria Rosario
Subject: RE: Query - Ring Dryer for Wheat Based DDGS - University of Saskatchewan

My pleasure Maria.

The easiest thing would be for me to issue you GEA Barr-Rosin versions. Just let me know which ones you would like and I'll get them arranged. Provided you reference us with something like "Printed with permission by GEA Barr-Rosin" or similar, we should be good. I'll check on the wording.

Should you have any queries in the meantime, please do not hesitate to contact us.

Best regards

Adrian Dee
Market Manager, GEA Barr-Rosin

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2. From Cellencor, Inc.

The diagram on an industrial microwave drying system appeared as Fig. 7.3 in the report.

Re: Request for permission to use a diagram

Ken Kaplan [kbkaplan@cellencor.com]

Sent: Thursday, November 14, 2013 10:05 PM

To: Mosqueda, Maria Rosario

Maria,

Please feel free to use the drawing in your report. Thank you for asking. I would very much enjoy reading the report when it's available.

Best Regards,

Ken Kaplan

On 11/14/2013 3:21 PM, Mosqueda, Maria Rosario wrote:

Hello, Dr Kaplan

I am the graduate student who wrote to you sometime in July 2013 about the use of microwave energy in drying wheat DDGS. You also generously shared some of your materials to me.

May I ask your permission to use the attached diagram of a microwave drying system in my report (economic evaluation)? The diagram was drawn using MS Paint but was based entirely on page 8 of the microwave drying powerpoint material that you provided (also attached).

I would be happy to send you a copy of my draft report once it is completed and edited by my research supervisor.

I hope for your approval.

Thanks,
Maria Rosario

Ken Kaplan, ASABE, IEEE

President

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