

**INVESTIGATION OF WATER REPELLENCY AND CRITICAL WATER
CONTENT IN UNDISTURBED AND RECLAIMED SOILS FROM THE
ATHABASCA OIL SANDS REGION OF ALBERTA, CANADA**

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In Partial Fulfillment of the Requirements for the Degree of

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By

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ABSTRACT

Ecosystems are disturbed to extract synthetic crude oil from the Athabasca Oil Sands Region (AOSR) in northern Alberta, Canada. Successful reclamation of mined oil sands sites depends on maximizing water storage and minimizing the potential for erosion. Soil water repellency in the AOSR affects undisturbed sites and consequently reclamation materials. Extreme water repellency may lead to low infiltration rates and hinder reclamation. There is a lack of information about the naturally occurring and pre-existing levels of soil water repellency in the AOSR. Thus, questions arise about the degree of naturally occurring water repellency and the potential for severe water repellency in reclamation soils.

Studies were conducted on nine sites in the AOSR in the summers of 2008 and 2009. A range of undisturbed and reclaimed sites, as well as mineral and organic reclamation materials were examined. Five undisturbed Jack Pine stands (classified as A ecosites), four reclaimed sites and reclamation materials including mineral soil, peat and leaf and lichen covering the forest floor (LFH) were studied. For a comparison of methods, one grasslands site in central Saskatchewan was included.

Mini and standard tension infiltrometers were compared as a means of measuring soil water repellency index (RI). There was strong variability in RI values between the infiltrometer methods. The mean RI values from the mini infiltrometers were higher than from the standard infiltrometer (9.61 and 3.46, respectively). The variability within sites dominated the variability in RI for the two methods. Despite these obvious trends, RI values between infiltrometer sizes were statistically different for only two individual sites. Increasing the number of sampling points in the second field season did not reduce the variability. The simpler, less expensive mini infiltrometer is as effective as the standard infiltrometer in measuring soil water repellency. This will enable more efficient and extensive monitoring of soil water repellency in reclaimed and undisturbed sites in the AOSR.

Soil water repellency of reclaimed and undisturbed sites was investigated *in situ* using RI, the water droplet penetration time (WDPT) test, and the molarity of ethanol droplet (MED) test. These measures showed similar trends. Variability in soil water repellency was high at both reclaimed and undisturbed sites. The average RI value for the surface of reclaimed sites was

higher than that of the subsurface at reclaimed sites; however, there were no statistical differences between RI values of surface reclaimed and undisturbed sites ($P = 0.213$) due to high spatial variability.

The critical water content (CWC) of reclamation materials was determined by measuring the contact angle (CA) and WDPT. Generally, CA and WDPT were inversely related to water content, though variability was high and the relationship between water content was weak. The clearest relationship between water repellency and water content was present for the mineral soil samples. Reclaimed mineral soil was generally wettable above gravimetric water contents of 5-10 %, while the coarse textured tarball affected materials were only subcritically water repellent. There was no relationship between water repellency and water content for peat and LFH. The degree of water repellency was statistically higher for peat materials with increasing decomposition levels. The average WDPT was 44, 128 and 217 s for fibric, mesic, and humic peat, respectively.

With careful management and monitoring, water repellency may not be a major limitation to reclamation success. The mini tension infiltrometer is an effective method for monitoring soil water repellency in the AOSR.

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LIST OF ABBREVIATIONS

AE1	A ecosite 1
AE2	A ecosite 2
ALFH	Aurora LFH capping study
AOSR	Athabasca Oil Sands Region
CA	contact angle
CC	coke cover capping study
CCME	Canadian Council of Ministers of the Environment
CONRAD	Canadian Oil Sands Network for Research and Development
CV	coefficient of variation
CWC	critical water content
F	fragmented litter layer
FP	fibric peat
H	humus layer
HP	humic peat
L	leaf litter layer
LFH	leaf, litter and humus layer on soil surface
MED	molarity of an ethanol droplet
MP	mesic peat
RI	water repellency index
SS	shallow-stripping study
SV10	soil vegetation plot 10
SV26	soil vegetation plot 26
SV27	soil vegetation plot 27
SW30	south west 30
WDPT	water droplet penetration time
WOTB	coarse textured soils without tarballs
WTB	coarse textured soils with tarballs

1. INTRODUCTION

Observations of low infiltration rates at some reclaimed sites have prompted the following investigation into soil water repellency in the Athabasca Oil Sands Region (AOSR) of northern Alberta, Canada. One such observation occurred on the Aurora LFH capping study, which has been further studied in this research. Rain on the surface of this reclaimed site beaded up, rather than infiltrating the soil (personal communication, C. Dubyk, May, 2008). This occurrence raised questions about the possible occurrence of water repellency at reclaimed sites. The following thesis is a result of these observations and is intended to answer the questions that were raised.

The AOSR contains nearly 81% of Alberta's bitumen deposits (Government of Alberta, 2008). Bitumen is excavated in open-pit mines and upgraded to synthetic crude oil by removing carbon and sulfur and adding hydrogen (Johnson and Minanishi, 2008). The oil sands are a major component of the provincial and national economy. Consequently, the number and scale of mining operations in the region is increasing at exceptional speed, leaving large tracts of land to be reclaimed now and in the future.

Mined sites are reclaimed by careful placement of reclamation materials that have been salvaged from sites before mining. These materials are stored in stockpiles during the mining process. Coarse textured and organic soils dominate the AOSR and are the primary materials available for reclamation. These materials are especially susceptible to soil water repellency, or hydrophobicity (Tschapek, 1984; de Jonge et al., 1999; Doerr et al., 2000; Woche et al., 2005). Soil water repellency, the reduced wettability of soil, may inhibit water infiltration and increase erosion. Water repellency is caused by organic coatings on soil particles. This is a dynamic condition, changing with water content, temperature, relative humidity and physical disturbance (Savage et al., 1969; DeBano, 1981; Lichner et al., 2007). As such, the degree of water repellency of reclamation materials may depend on the handling and storage practices used.

Water content is a major control on the degree of soil water repellency. Dry soil is generally more water repellent while moist soil is more wettable. Critical water content (CWC) describes the water content at which materials change from being water repellent to wettable

(Dekker and Ritsema, 1994). Information about the CWC of reclamation materials will help to guide decisions about handling and storage practices of reclamation materials.

The overall intent of this work is to provide information about the state of water repellency in reclaimed and undisturbed sites and provide tools to monitor and minimize water repellency in reclamation. Information gained from these studies will help inform decisions about mining, handling of reclamation materials and reclamation practices. The objectives of these studies are to:

1. Compare the water repellency indices measured by the mini and standard sized tension infiltrometers.
2. Determine the current state of soil water repellency in reclaimed and undisturbed sites in the AOSR.
3. Investigate the relationship between water content and soil water repellency in order to determine the CWC of mineral and organic reclamation materials.

To meet these objectives, a series of field and laboratory studies were conducted along with a thorough investigation of the literature on oil sands mining, soil water repellency and methods for its assessment. These investigations are presented in five chapters. The first chapter introduces the topic and outlines the observations that prompted this research as well as the objectives of these studies. A comprehensive review of oil sands mining and reclamation practices, soil water repellency, critical water content, methods for describing soil water repellency and previous research that is pertinent to this study is provided in Chapter Two. Chapters Three and Four summarize the studies investigating soil water repellency in the AOSR, followed by an overall summary and conclusion in Chapter Five.

The results addressing objective 1 are discussed in Chapter Three. In this study, the mini tension infiltrometer was compared to the standard tension infiltrometer as a means of determining soil water repellency index (RI) (Tillman et al., 1989; Wallis et al., 1991). By showing the effectiveness of the less expensive and time consuming mini tension infiltrometer, more effective and extensive monitoring may be used in the AOSR.

Objectives 2 and 3 are met by the studies described in Chapter Four. For these studies, the RI, water droplet penetration time (WDPT) test (Van't Woudt, 1959; Letey, 1969; Doerr, 1998), and molarity of an ethanol droplet (MED) test (Watson and Letey, 1970; King, 1981; Doerr, 1998; Roy and McGill, 2002) were used *in situ* to determine the range of soil water repellency in both undisturbed and reclaimed sites. Knowledge of soil water repellency in natural conditions will provide a basis of comparison for reclaimed sites, while understanding of soil water repellency in existing reclaimed sites will show whether soil water repellency is exacerbated by the current practice. The CWC of reclamation materials were examined *ex situ* using the WDPT test and the angle (CA) using digital images captured using a PG-X goniometer (FIBRO System AB, 2006). Understanding of CWC of reclamation materials will aid decisions about the handling, storage and placement of reclamation materials.

This thesis is presented as a series of peer-reviewed manuscripts. As such, repetition is unavoidable and each chapter may be viewed as a stand-alone article. Chapter Three was published in the Canadian Journal of Soil Science in February, 2011 (Hunter et al., 2011) and Chapter Four is hoped to be published as two separate papers after completion of this thesis.

2. LITERATURE REVIEW

2.1. Oil Sands Operations

Synthetic crude oil is produced from bitumen deposits from the Athabasca Oil Sands Region (AOSR) in northern Alberta, Canada. Bitumen is a viscous petroleum product that is mined and processed to yield synthetic crude oil (Johnson and Miyanishi, 2008). The AOSR contains nearly 81% of Alberta's bitumen deposits (Government of Alberta, 2008) of which, 43 000 km² is currently leased from the province of Alberta for mining development (Johnson and Miyanishi, 2008). The number and size of these mines continues to increase, leaving large tracts of land to be reclaimed.

In the AOSR, most bitumen occurs in near-surface deposits which are extracted in open-pit mines from up to 100 m below the soil surface. Though this method is the most economical in this case, it also has the most severe environmental impact as whole ecosystems are removed (Johnson and Miyanishi, 2008). Before mining takes place, surface materials are salvaged and then stored in stockpiles while sites are mined. These reclamation materials are then used to create new, self-sufficient ecosystems that support natural cycles and provide habitat for biotic communities. The overall goal of reclamation is to restore sites to their original land capacity, rather than re-creating them exactly as they were (Johnson and Miyanishi, 2008). Materials are divided according to site and materials type. Materials such as organics, mineral soil, overburden and tailings sand are available for reclamation.

Surface organic matter is classified as leaf litter (L), fragmented litter (F) and humus (H) horizons, or simply LFH (Soil Classification Working Group, 1998). The L horizon is the top layer in which original leaf and twig structures are remain intact; F is the middle, partially decomposed horizon; and H is the bottom layer containing mainly decomposed humic materials. LFH is an important resource in reclamation, as it contains plant materials for asexual reproduction and seeds. The use of LFH as an independent reclamation material is quite recent. Though salvaging LFH poses logistical challenges, it has proven to be a valuable propagule bank for newly reclaimed sites (Mackenzie and Naeth, 2007; Mackenzie and Naeth, 2010).

Organic soils are also prevalent in the AOSR region. These are soils with greater than 30% organic matter by weight to a depth of 40 cm or more. Organic materials are categorized by

their decomposition level as fibric, mesic and humic. Fibric peat is the least decomposed, where original structures of leaves and twigs are still discernable. Mesic peat is the mid-range of decomposition, where the peat is less heterogeneous than fibric but some original structures are present. No original structures are present in humic peat and it is the consistency of thick jelly. The von Post scale of decomposition ranges from 0 – 10 with 0 being not decomposed and 10 being completely decomposed. On the von Post scale, fibric peat is 1-4, mesic peat is 5-6 and humic peat is 7-10 (Soil Classification Working Group, 1998). Peat materials are salvaged and stored, then often mixed with mineral soils in reclamation prescriptions.

Mineral soils contain less than 30% organic matter by weight. They are salvaged and stored according to soil horizon. The A horizon is the top layer in mineral soils, where most biotic activity and weathering takes place. The B horizon is below the A and is characterized by a change in structure, texture and / or organic content (Soil Classification Working Group, 1998). Overburden is the parent material and bedrock that lies between living soil and bitumen deposits. It is also stored during mining and used to form landscapes in reclamation. In the production of synthetic crude oil, petroleum is removed from the bitumen. Mine tailings, a combination of water, clay and some residual bitumen, are left after oil is extracted from the bitumen.

Bands and chunks of bitumen deposits are naturally occurring in some surface soils (0-6m) in the AOSR (Fleming et al., 2011). When these deposits are present in chunks, they are referred to as tarballs (Fleming et al., 2011). Tarballs range from a few centimeters to a few meters in size. These naturally occurring tarballs often exceed clean soil guidelines but have been shown to be recalcitrant enough to resist leaching, posing little to no risk for hydrocarbon contamination of groundwater (Fleming et al., 2011). The exterior of tarballs serve to protect the internal bitumen from weathering (Fleming, 2011). Tarballs affected materials may also be used in reclamation covers.

When mining is complete, the stockpiled reclamation materials are used to create new landscapes. Reclamation materials come from a variety of sources and are affected by handling, storage and processing before they are used in reclamation. The source and handling of reclamation materials are outlined in Figure 2.1.

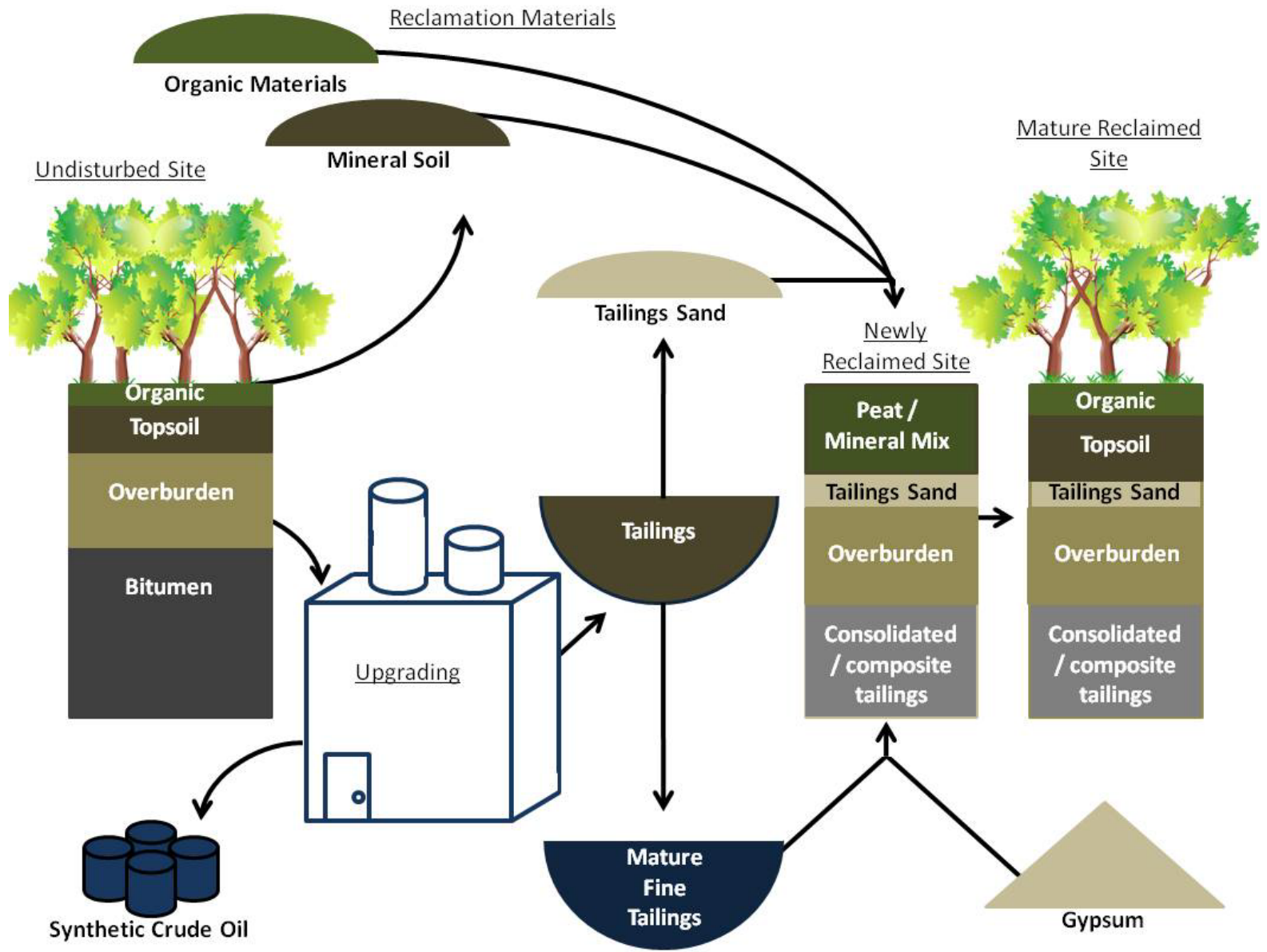


Figure 2.1. Flow chart of mining and reclamation process.

Success of reclamation is dependent on numerous interdependent factors. The health of microbial, fungal, plant and animal communities are dependent, in part, on the ability of soil to absorb and retain water. Without sufficient water infiltration and stabilizing plant communities, soil is vulnerable to wind and water erosion (DeBano, 1981; King, 1981; Dekker and Ritsema, 1994; Ellies et al., 2005). Water infiltration and storage is related to soil texture, organic matter, structure, slope, and water repellency. While the roles of these factors are well known, the cause, effect and management of soil water repellency, especially in the context of oil sands reclamation requires further investigation.

2.1. Soil Water Repellency

Soil water repellency, or hydrophobicity, occurs when soil is not completely wettable. In severe cases, no water infiltrates regardless of how long the soil is exposed to water. This is rare in nature, but occurs as shown in Figure 2.2. Sub-critical soil water repellency, a reduction in infiltration rate, is nearly ubiquitous in natural ecosystems (Hallett et al., 2001; Dekker et al., 2005).

A small degree of soil water repellency is important for stabilizing soil structure (Tisdall and Oades, 1982) and soil aggregates (Hallett and Young, 1999), improving soil water storage capacity (Dekker and Ritsema, 1996; Kobayashi and Shimizu, 2007), and preventing dispersion and erosion (Ellies et al., 2005). Severe soil water repellency, however, has negative impacts on water infiltration (DeBano, 1971; Wallis et al., 1993) and retention (DeBano, 1981; Hendrickx et al., 1993), leaving soil vulnerable to wind (Ravi et al., 2009) and water erosion (King, 1981; Dekker and Ritsema, 1994; Shakesby et al., 2000; Ellies et al., 2005; Cerdà and Doerr, 2007) and hindering seed germination (Osborn et al., 1967). Furthermore, heterogeneous wetting patterns in water repellent soils may result in preferential flow and contribute to groundwater contamination (Hendrickx et al., 1993; Dekker and Ritsema, 1994; Bauters et al., 2000; Buczko and Bens, 2006; Carrick et al., 2011).

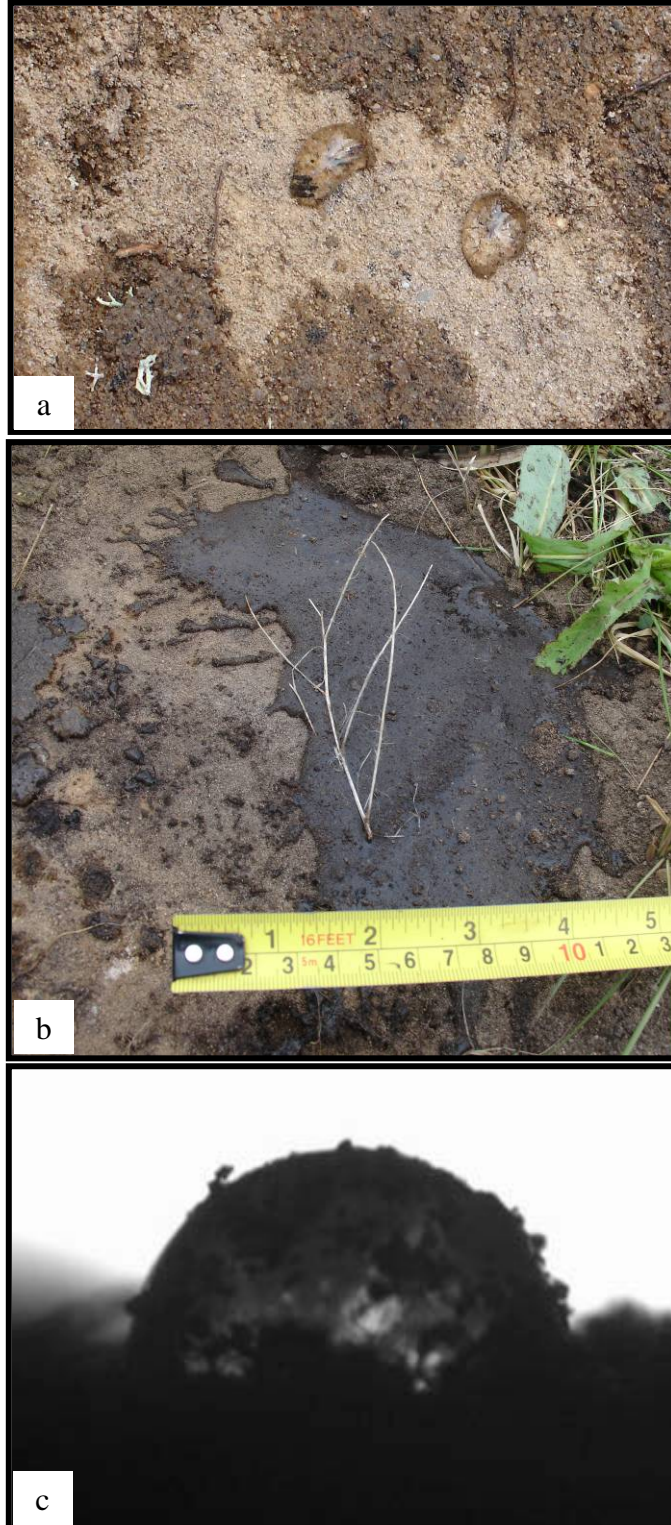


Figure 2.2. Examples of soil water repellency from the Athabasca Oil Sands Region in Alberta, Canada a) at an undisturbed site, b) at a reclaimed site and c) as a magnified water droplet on reclaimed mineral soil.

2.1. Causes of Soil Water Repellency

Organic matter is the leading and most documented contributor to soil water repellency (e.g. Bond, 1968; Tschapek, 1984; Ma'shum et al., 1988; Doerr et al., 2000; Ellies et al., 2005; Karunaratna et al., 2010). Bare mineral particles have a high affinity for water (Tschapek, 1984), but are coated with hydrophobic or amphiphilic organic residues of many varieties (Hudson et al., 1994; Ellies et al., 2005; Buczko and Bens, 2006). These organic compounds originate from vegetation (Bond, 1964; Franco et al., 2000), fungi (Bond and Harris, 1964; Savage et al., 1969; Dekker and Ritsema, 1996), microorganisms (Schaumann et al., 2007; Fisher et al., 2010), humic acids (Roberts and Carbon, 1972; Chen and Schnitzer, 1978), decomposed plant material (McGhie and Posner, 1987; Ellies et al., 2005) and more. The relationship between organic matter content and soil water repellency is inconsistent and is suspected to be dependent on the chemical nature of the particle surface, strength of organic-soil interaction, abundance and quality of organic materials and the chemistry of soil solution (Horne and McIntosh, 2000; Graber et al., 2009).

Because petroleum hydrocarbons have a low solubility in water, they contribute to soil water repellency when they coat soil particles, (Davis, 1952; Ellis and Adams, 1961; Karickhoff et al., 1979; de Jonge et al., 1997). The impact of hydrocarbon content on soil water repellency is dependent on factors such as type and age of hydrocarbon (de Jonge et al., 1997), quality and quantity of organic matter (Hudson et al., 1994) and soil texture (Karickhoff et al., 1979) to name only a few. The implications of crude oil spills in Alberta, Canada on soil water repellency have been studied (Roy and McGill, 1998; Roy et al., 2003); however, no studies on the relationship between the naturally occurring hydrocarbons in the AOSR and soil water repellency were located in this review.

Though soil water repellency has been found in many soils around the world in all soil textures (Wallis et al., 1991; Dekker et al., 2005; Leelamanei et al., 2010), sandy soils are especially susceptible to soil water repellency (Tschapek, 1984; Harper and Gilkes, 1994; de Jonge et al., 1999, Woche et al., 2005; Karunaratna et al., 2010). Coarse textured soils have a lower particle surface area per unit volume than fine textured soils, meaning that less organic matter is required to coat surfaces (Doerr et al., 2000). These coatings may be abraded by

physical disturbance such as shaking the soil in a laboratory shaker (King, 1981) or cultivation (Hallett et al., 2001).

Atmospheric conditions such as air temperature (Dekker et al., 1998; Dekker et al., 2001; Diehl and Schaumann, 2007) and relative humidity (Bisdorf et al., 1993; Dekker et al., 2001; Doerr et al., 2002) affect soil water repellency. Soil water repellency may be underestimated when measured at high ambient air temperatures as increased temperatures reduce the surface tension of the test liquid (King, 1981; Dekker et al., 1998; Dekker et al., 2001). Increased relative humidity before a rain event may increase soil water repellency (Jex et al., 1985; Doerr et al., 2002). Furthermore, increased atmospheric CO₂ has been associated with decreased soil water repellency. Newton et al. (2003) speculated that this is a result of chemical alterations to soil carbon pools, but identified a need for further research to confirm the mechanism for this result.

Soil water repellency is a dynamic phenomenon that changes with water content (Dekker and Ritsema, 1994). Heating and drying cause hydrophilic portions of amphiphilic organic compounds to bind to themselves and soil particles, leaving mainly hydrophobic areas exposed (Savage et al., 1969; DeBano, 1981; Doerr, 1998; Lichner et al., 2007). As such, forest fires are a major cause of severe soil water repellency (DeBano and Krammes, 1966; DeBano et al., 1970; DeBano, 1981; DeBano, 2000; Ferreira et al., 2005; Shakesby and Doerr, 2006). Just as drying induces soil water repellency, prolonged exposure to water weakens repellency by re-exposing hydrophilic portions of organics (Doerr et al., 2000).

Critical water content (CWC) is the threshold at which soils change from being water repellent to wettable (Dekker and Ritsema, 1994; Ritsema and Dekker 1994; Dekker et al., 2001). This was originally identified as one water content; however, many studies have reported a transition zone for CWC (e.g. de Jonge et al., 1999; Dekker et al., 2001). The degree of soil water repellency is dependent on whether soil is in a wetting trend or a drying trend through a phenomenon called hysteresis (Dekker et al., 2001; Regalado and Ritter, 2005; Shang et al., 2008). As wettable soils dry, they retain their wettability and water repellent soils resist wetting in the presence of water.

Given the numerous, dynamic and heterogeneous causes of soil water repellency, it is expected that soil water repellency itself is spatially and temporally variable (Angulo-Jaramillo

et al., 2000; Nunan et al., 2002; Hallett et al., 2004; Leighton-Boyce et al., 2005; Sepaskhah et al., 2005; Lamparter et al., 2006; Regalado and Ritter, 2006). The relationship between water repellency and water content of mineral soil is shown in Figure 2.3. In this figure, an arbitrary ‘degree of soil water repellency’ is used to illustrate the severity of soil water repellency, as various methods could be used to describe this.

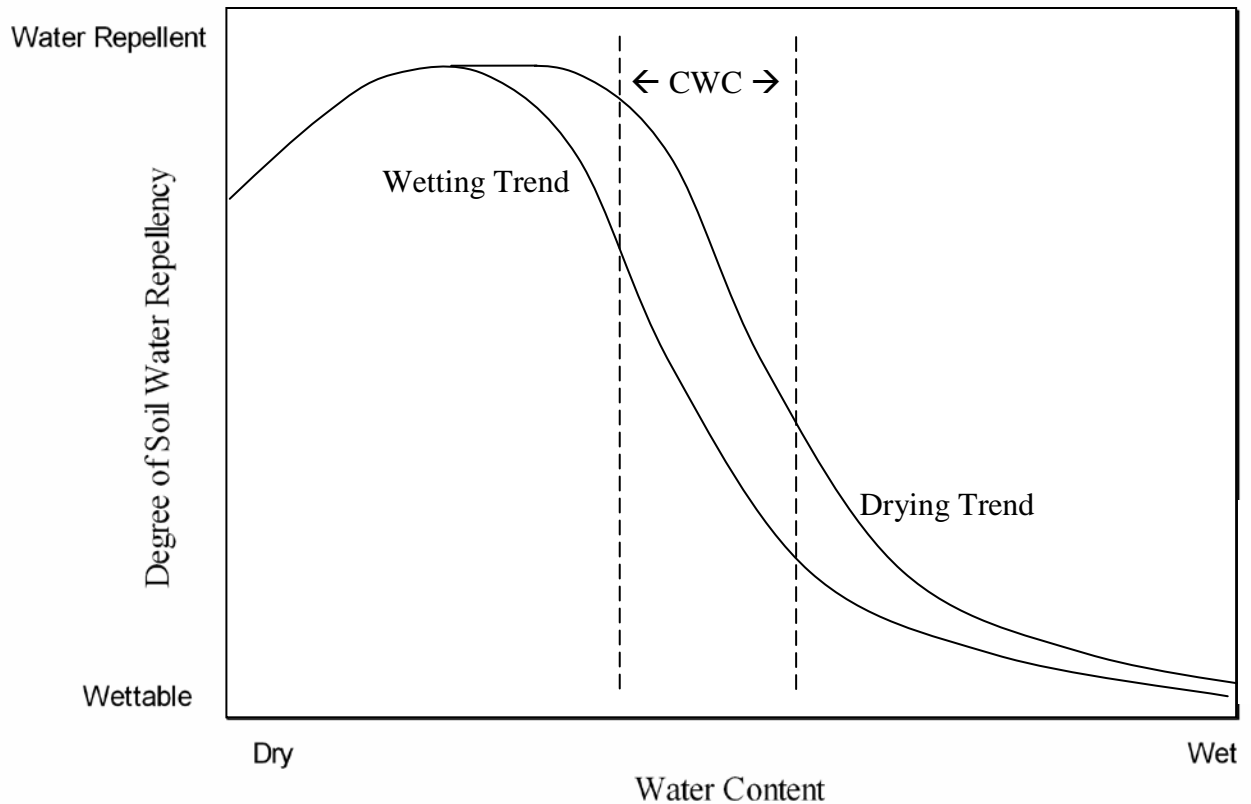


Figure 2.3. Theoretical relationship between soil water repellency and water content illustrating the critical water content (CWC), adapted from de Jonge et al. (1999) and Dekker et al. (2001).

Though there have been many studies on CWC of mineral soils, none were located that examined the CWC of organic materials. The existing information shows a strong relationship between soil water repellency and water content for mineral materials and implicates organic materials as the leading cause. This suggests that organic materials themselves are likely to follow a similar trend.

The study of soil water repellency is challenging and pertinent because of its many inter-related causes. These causes are summarized in Table 2.1.

2.2. Measures of Soil Water Repellency

Soil water repellency may be measured on both dried and field moist samples. Dekker and Ritsema (1994) described ‘actual’ water repellency from measures taken *in situ* or on field moist samples and ‘potential’ water repellency as measures taken from dried samples. Actual water repellency indicates current *in situ* conditions, while potential water repellency has been used as a proxy to the most extreme case. Recent studies have shown that drying samples by air and oven are not indicative of field conditions (Dekker et al., 1998; Buczko et al., 2002; Newton et al., 2003; Dekker et al., 2009).

Severity of soil water repellency describes how strongly water is initially repelled. This provides information about the risk of runoff and erosion in a single rainfall event. Because soil water repellency is temporally variable, its longevity, or persistence, is also of interest. An understanding of the persistence of water repellency provides information about wettability in the long term. Both of these measures provide different information that is required in order to gain a clear picture of the state of water repellency. For example, severe water repellency may not be a large concern if it is not very persistent. Likewise, a moderate degree of water repellency may be of greater concern if it is very persistent. The level of concern raised by these two scenarios is dependent on the type of material, the grade of the landscape and the level of sun and wind exposure that will be received by the soil.

Because the causes of soil water repellency are so numerous and complex, standardized measures are needed to produce comparable results (Dekker et al., 2009). Of particular importance are drying temperature (Dekker et al., 1998), air temperature (Dekker et al., 1998; 2001; Doerr et al., 2002) and relative humidity (Jex et al., 1985; Bisdom et al., 1993; Dekker et al., 2001). At higher temperatures and relative humidities water repellency may be artificially elevated. Furthermore, drying above room temperatures may further increase water repellency. However, drying at extreme temperatures may remove soil water repellency as organic matter is burned off. Storage, preparation and handling of samples should be carefully considered and clearly stated in studies about soil water repellency.

Table 2.1. Causes, effects and mechanisms of soil water repellency and references.

Cause	Effect	Mechanism	References
Organic matter	Between 2 – 14% carbon content, water repellency and carbon content are correlated	Hydrophobic organic compounds coat soil particles	Bond, 1968 Karunarathna et al., 2010
Vegetation	Presence and distribution of vegetation influences soil water repellency	Waxes from plant tissues are deposited into the soil and coat soil particles.	Bond, 1963 Franco et al., 2000
Fungi	Presence of some fungi increases water repellency	Hydrophobic exudates coat soil particles	Bond and Harris, 1964
Microbial activity	Biological crusts prevent infiltration	Non-polar exudates prevent wetting	Schaumann et al., 2007 Fisher et al., 2010
Humic acids	Humic acids in carbon pool increase water repellency	Humic acids coat soil particles	Chen and Schnitzer, 1978
Decomposed plant material	Inputs of decomposed plant materials may increase soil water repellency	Waxes from plant materials coat soil particles	McGhie and Posner, 1987 Ellies et al., 2005
Petroleum hydrocarbons	Increased dichloromethane-insoluble organics may increase soil water repellency	Hydrophobic hydrocarbons from crude oil and natural gas spills bind to soil particles	Ellis and Adams, 1961 Roy and McGill, 2003
Soil texture	Coarse textured soils are more water repellent	Lower soil particle surface area requires less organic matter to coat	DeBano et al., 1970 Karunarathna et al., 2010
Physical disturbance	Soils may lose water repellency after physical disturbance	Physical disturbance may abrade organics on soil particles	King, 1981 Hallett et al., 2001
Ambient air temperature	Higher air temperatures cause falsely low soil water repellency measures	Increased temperatures reduces the surface tension of test liquid	Dekker et al., 2001 Diehl and Schaumann, 2007
Relative humidity	Soil water repellency increases after high humidity	Displaced organics expand in pore spaces	Doerr et al., 2002
Elevated atmospheric CO ₂	Soil water repellency is decreased under elevated CO ₂	Increased CO ₂ changes the chemical composition of soil carbon pool	Newton et al., 2003
Water content	Dry soil is more repellent, wet soil is more wettable	Drying causes organic matter to orient on soil particles, leaving hydrophobic portions exposed.	Dekker and Ritsema, 1994
Extreme heat	Soil is water repellent after exposure to extreme heat such as forest fires	Heating causes organics to condense onto soil surfaces	DeBano et al., 1970 DeBano, 2000
Drying temperature	Increased drying temperature increases repellency	Higher temperatures cause organics to condense onto soil particles	Dekker et al., 1998
Hysteresis	Wettable soils retains wettability when drying, water repellent soils resist wetting		Dekker et al., 2001 Regalado and Ritter, 2005

For a liquid to infiltrate into soil, its surface energy must be less than that of the soil (Doerr, 1998). Thus, the infiltration of water will be impeded if the soil has a high surface energy, as is the case for water repellent soils. The test liquid will infiltrate if the surface energy (tension) is sufficiently lowered (Van't Woudt, 1959). Ethanol, with a much lower surface energy than water behaves in water repellent soil in a similar way that water would in the same soil if it were wettable (Letey et al., 1962). Because soil water repellency impedes infiltration of water but not ethanol, various comparisons of their behaviors in soil are used to characterize soil water repellency.

The most direct way to determine soil water repellency is to measure the contact angle (CA) of water on the soil surface. Because soil particles are not flat, direct measurement of CA is difficult and often inconsistent (Letey et al., 2000). Several methods have been used to calculate or directly measure CA, including capillary rise (Emerson and Bond, 1963) and modified capillary rise (Bachmann et al., 2003), Wilhelmy plate (Bachmann et al., 2003) and sessile drop (Bachmann et al., 2000) methods.

The capillary rise method indirectly calculates the initial CA by measuring the rate of rise of water in a sand column. Letey et al. (1962) describes the capillary rise equation as:

$$Q'' = \frac{\phi r (\rho r G h + 2\gamma \cos CA)}{8L\eta} \quad [2.1]$$

where Q'' is the rate of water entry at the soil surface ($\text{m}^2 \text{s}^{-1}$), Φ is porosity ($\text{m}^3 \text{m}^{-3}$), r is the capillary radius (m), ρ is density of the solution (kg m^{-3}), G is the gravitational constant ($6.67 \times 10^{-11} \text{ m}^3 \text{kg}^{-1} \text{s}^{-2}$), h is capillary length plus depth of solution above the capillary (m), γ is surface tension of the solution (dyn m^{-1}), CA is liquid-solid contact angle ($^\circ$), L is capillary length (m) and η is viscosity (Pa s). By measuring either the height of rise (Emerson and Bond, 1963; Bond, 1968) or the mass gained by the column (Bachmann et al., 2003) with time, all but the pore radius and the CA are known or assumed. The only variable that is not easily measured is CA. A treatment that assumes that $CA = 0$ is used to calculate r , and the CA for water can then be calculated. This can be done by eliminating the effects of soil water repellency by removing it from the test soil, usually by burning it (Emerson and Bond, 1963, Bond, 1968) or by using ethanol as the test liquid (Letey et al., 1962; Bachmann et al., 2003).

A major limitation of the capillary rise method is that it is only applicable to subcritically water repellent soils ($CA < 90^\circ$). A modification by Bachmann et al. (2003) allows for this method to be applied to water repellent soils ($CA > 90^\circ$) by using mixtures of varying surface energies as the wetting agent. This modification has broadened the range of measurement of the capillary rise method to CA 0- 180° with a precision of approximately $<5^\circ$ but is very labor intensive (Bachmann et al., 2003).

Bachmann et al. (2003) also adapted the Wilhelmy plate method to determine the soil-liquid CA . In this method, a single-grain layer of soil is adhered to a plate using double sided tape. This plate, suspended from an electronic balance, is lowered then raised out of a test liquid in order to determine the advancing and retreating CA . The forces that act on the plate are gravity, buoyancy and pressure from the meniscus of the water (or wetting force). Given the knowns about the forces of gravity and buoyancy, the weight change of the plate can be used to derive the CA using the following relationship (Bachmann et al., 2003):

$$\cos(CA) = \frac{(F_t + V\rho g)}{l_w \sigma_{lv}} \quad [2.2]$$

where CA is the contact angle, F_t is the total force on the plate, V is the volume of the plate that is immersed (m^3) ρ is density of the fluid ($kg\ m^{-3}$), g is acceleration due to gravity ($m\ s^{-2}$), l_w is the wetted length of sample (m) and σ_{lv} is surface energy of the test liquid. ($mJ\ m^{-2}$). F_t can be found using the relationship (Bachmann et al., 2003):

$$F_t = W - F_b + F_w = W - V\rho g + l_w \sigma_{lv} \cos(\theta) \quad [2.3]$$

where W is the weight of the plate (kg), F_b is the buoyancy force and F_w is the wetting force. F_w is found using a linear regression of the weight as a function of time. This method allows for angles 0- 180° to be measured with an accuracy of approximately 3- 5° (Bachmann et al., 2003). The benefit of this method is that it may be automated, allowing for easy replication of many samples. However, the cost of equipment may be prohibitive.

The sessile drop method (Bachmann et al., 2000) is used to determine the CA directly from a photograph of a droplet on the soil surface. This method allows for measurement of CA

0-180° within accuracy of $<6^\circ$ (Bachmann et al., 2003). This is applied to a single-grain layer of dried, sieved soil mounted on double sided tape, providing the flattest possible plane for measurement. The droplet size must exceed the diameter of soil particles in order to be measurable (Bachmann et al., 2003). The photograph is captured with a camera fitted to a microscope (Bond, 1968; Bachmann et al., 2000) or a PG-X Gonimeter (FIBRO System AB, 2006) (Figure 2.4). The CA is either measured by hand using a protractor (Bond, 1968) or electronically using software such as multi-platform java image processing program, Image J (available at: <http://rsbweb.nih.gov/ij/>) using the Low Bond Axisymmetric Drop Shape Analysis Model of Drop Shape Analysis plug in (available at <http://bigwww.epfl.ch/demo/dropanalysis/>). This model utilizes image gradient energy and cubic spline interpolation to obtain contact angle image measurements (Stalder et al., 2006; Stalder et al., 2010). This direct measurement of CA is simple, accurate and requires the least extrapolation. In a comparison of methods for determining CA values, Shang et al. (2008) found that the sessile drop method yielded the most consistent results.

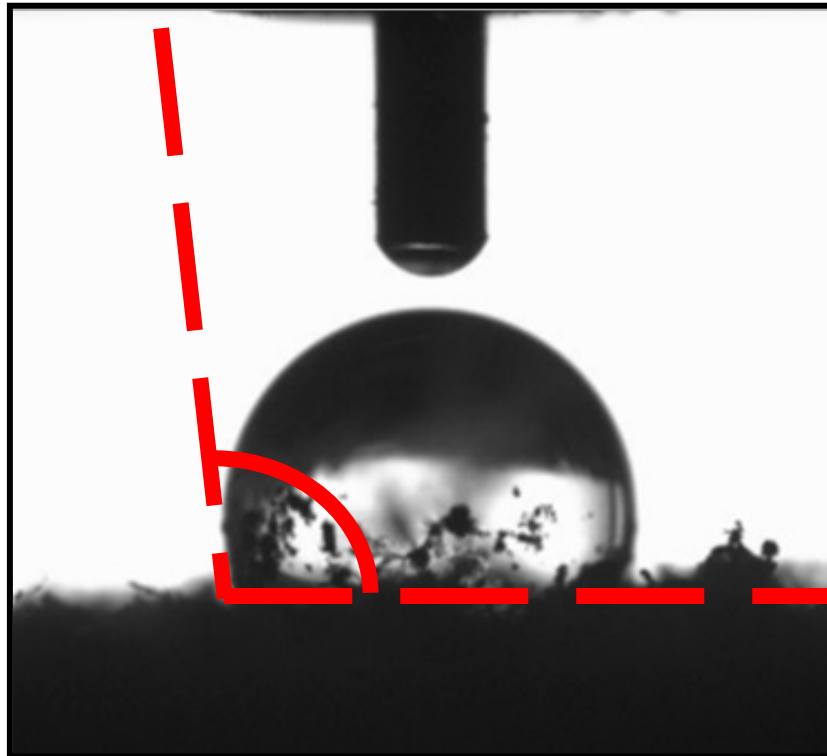


Figure 2.4. Photo of a 4 μ l droplet of water on a thin layer of soil captured by PGX-Giometer demonstrating the way that contact angle is determined.

The water droplet penetration time (WDPT) test, originally proposed by Van't Woudt (1959) and modified by Letey (1969) and Doerr (1998), provides a measure of the persistence of soil water repellency. This is a simple method of applying a droplet onto the soil surface and recording the time for infiltration. Several studies (e.g. Watson and Letey, 1970; King, 1981; Dekker and Ritsema, 1994; Bachmann et al., 2003) have reported WDPT values under five seconds as water repellent. These times have no physical meaning and are arbitrarily used to describe the degree of soil water repellency (Dekker and Ritsema, 1994).

Water droplet penetration times may be reported as averages. Many have divided WDPT results into a series of categories. King (1981) divided WDPT into 5 categories that have been used by Bisdorf et al. (1993), Chenu et al. (2000), Leelamenei et al. (2010) and more. Dekker and Jungerius (1990) divided WDPTs into 7 categories that have been used in many studies such as Dekker et al. (1998) Dekker et al. (2001). Doerr (1998), further divided times into 11 categories for higher resolution. These categories are outlined in Table 2.2.

This method is simple enough to be widely applied both *in situ* and *ex situ*. While Wessel (1988) preferred the WDPT over the CA because of the challenges with measuring contact angle of a droplet on an uneven surface, and the ability to divide WDPT values into subcategories, other researchers found that WDPT values were not easily reproduced. The time of infiltration of a water droplet is directly relevant to the erosion potential and water runoff (Wessel, 1988). As such, this method, in conjunction with other methods can be used to clearly describe soil water repellency.

In the molarity of an ethanol droplet (MED) test (Watson and Letey, 1970; King, 1981; Doerr, 1998), dilutions of ethanol, with known surface tensions (or energies), are applied to the soil. This allows the surface energy of the soil to be extrapolated. Results from the MED test have been reported in many ways. Droplets have been described by their liquid surface tension (dyn cm^{-1}) (Watson and Letey, 1970; de Jonge et al., 1999; Letey et al., 2000), molarity (mol m^{-3}) (King, 1981; Doerr, 1998) and volumetric ethanol percentage (cm^3 ethanol cm^{-3} water) (Dekker and Ritsema, 1994). All of these descriptors of ethanol concentration are correlated and can easily be converted between units (Letey et al., 2000). The 90° surface tension is the surface tension of liquid that produces a 90° contact angle with the soil surface (Watson and Letey, 1970;

Table 2.2. Water droplet penetration time test time categories.

Descriptor	King (1981)	Dekker and Jungerius (1990)	Doerr (1998)
	Time Range (s)	Time Range (s)	Time Range (s)
Non repellent	≤ 1	0 – 5	≤ 5
Slightly repellent	1 – 60	5 – 60	5 – 10
			10 – 30
			30 – 60
Strongly repellent	60 – 600	60 – 600	60 – 180
			180 – 300
			300 – 600
Severely repellent	600 – 3600	600 – 3600	600 – 900
			900 – 3600
Extremely repellent	≥ 3600	3600 – 10800	3600 – 18000
		10800 – 21600	≥ 18000
		≥ 21600	

Letey et al., 2000). Many have reported MED results as the lowest ethanol percentage to penetrate the soil in less than five seconds (e.g. Dekker and Ritsema, 1994), while others used three seconds (e.g. Crockford et al., 1991; Doerr, 1998; Doerr and Thomas, 2000; Cofield et al., 2007). In a report standardizing the method, Leelamanie et al. (2008) suggested that 10 s be used and cautioned that lower times may underestimate soil water repellency. Furthermore, King (1981) and Roy and McGill (2002) found that the MED test is not reliable at field moist conditions, as such this method should only be used on dried samples (Dekker et al., 2009).

The soil water repellency index (RI) compares hydraulic behavior of water and ethanol in soil. This uses the sorptivity, calculated from the unsaturated flow rate in soil, determined using the disc infiltrometer (Perroux and White, 1988). The disc infiltrometer infiltrates a test liquid (water and ethanol for these studies) into soil under a negative tension through a disc. Generally, sorptivity is the slope of the curve when infiltration is plotted against the square root of time. This relationship is illustrated in Figure 2.5.

Initial or early time sorptivity, calculated from infiltration and time measurements is described by Philip (1969) as:

$$S = \frac{i_2 - i_1}{\sqrt{t_2 - t_1}} \quad [2.4]$$

where S is sorptivity ($\text{m hr}^{-1/2}$), i is infiltration (m) and t is time (hr).

Zhang (1997) outlined a method for determining the steady state sorptivity by fitting the data to the formula:

$$i = Ct + S\sqrt{t} \quad [2.5]$$

where i is cumulative infiltration (m), C is the slope of the curve (m s^{-1}), t is time (hr) and S is the sorptivity ($\text{m hr}^{-1/2}$). C is calculated as:

$$C = KA \quad [2.6]$$

where K is the conductivity of the soil (m s^{-1}) and A is a non-dimensional coefficient relating to the van Genuchten parameters α and η for soil type, applied suction rate and the disc radius.

Soil-water sorptivity is impeded by soil water repellency, whereas soil-ethanol sorptivity is not (Letey et al., 1962). As such, the corrected soil-ethanol sorptivity is used as the benchmark against which the impeded soil-water sorptivity is compared. Water repellency index (RI) is calculated as:

$$RI = 1.95 \frac{S_E}{S_W} \quad [2.7]$$

where S_E and S_W are the sorptivities ($\text{cm hr}^{-1/2}$) of soil to ethanol and water respectively and 1.95 accounts for the difference in density and viscosity between water and ethanol (Tillman et al., 1989; Wallis et al., 1991). Though many studies reference Tillman et al. (1989) as having derived the above relationship, it was not expressed in this simplified form until it was used by Wallis et al. (1991), who also identified 1.95 as the threshold between wettable and water repellent.

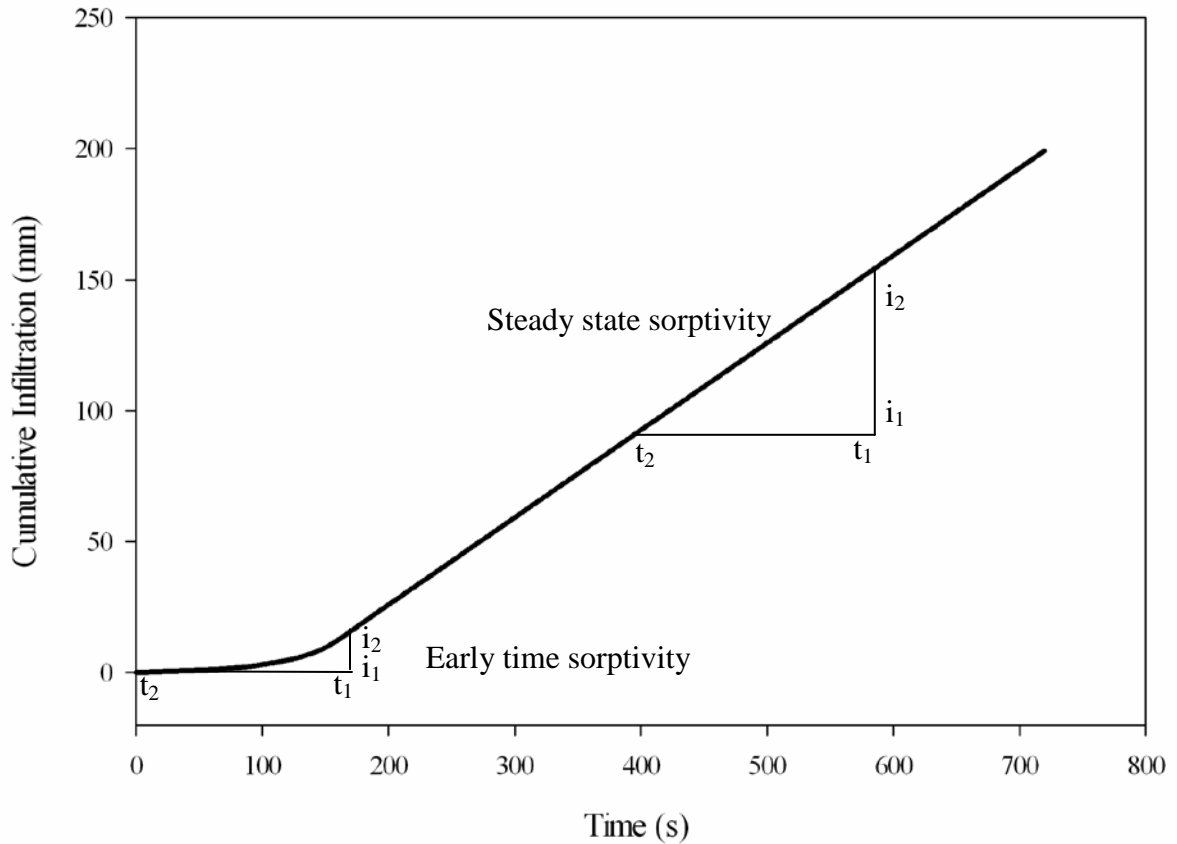


Figure 2.5. Relationship between cumulative infiltration and square root of time for water repellent soils.

The RI is more sensitive than other measures of soil water repellency and is able to describe sub-critical soil water repellency well. Water repellency index may be used to measure larger areas than WDPT, MED and CA and is able to capture effects of soil structure on soil water repellency (Wallis et al., 1991). The standard infiltrometer uses discs of interchangeable size to infiltrate the test liquid (White et al., 1992). A mini infiltrometer infiltrates liquid through a fixed 4.5 cm diameter plate (Figure 2.6). Leeds-Harrison et al., (1994) and Hallett et al. (2004) both developed miniaturized infiltrometer methods to determine RI at the aggregate scale.



Figure 2.6. Photos of a) standard tension infiltrometer and b) mini tension infiltrometer.

Table 2.3. Summary of methods for measuring soil water repellency.

Method	Output Units	Range	Application	Advantages	Limitations	References
Capillary Rise Method	Contact Angle Degrees (°)	< 90 °	<i>Ex situ</i>	<ul style="list-style-type: none"> • sensitive and accurate 	<ul style="list-style-type: none"> • subcritical repellency only • time consuming • expensive 	Emerson & Bond, 1963
Modified Capillary Rise Method	Contact Angle Degrees (°)	0 – 180 °	<i>Ex situ</i>	<ul style="list-style-type: none"> • sensitive • measures subcritical and severe repellency 	<ul style="list-style-type: none"> • labor intensive 	Bachmann et al., 2003
Wilhelmy Plate Method	Contact Angle Degrees (°)	0 – 180 °	<i>Ex situ</i>	<ul style="list-style-type: none"> • sensitive • may be automated 	<ul style="list-style-type: none"> • sensitive equipment is expensive 	Bachmann et al., 2003
Sessile Drop Method	Contact Angle Degrees (°)	0 – 180 °	<i>In situ</i> and <i>Ex situ</i>	<ul style="list-style-type: none"> • least extrapolation • can use software to calculate contact angle 	<ul style="list-style-type: none"> • cost of equipment may be limiting • measuring by hand may be inaccurate 	Bachmann et al., 2000
Water Droplet Penetration Time (WDPT) Test	Time (s)	Unlimited	<i>In situ</i> and <i>Ex situ</i>	<ul style="list-style-type: none"> • provides a measure of persistence • inexpensive • closely related to erosion risk 	<ul style="list-style-type: none"> • can be time consuming • results are not always reproducible 	Letey, 1969 King 1981 Wessel, 1988 Doerr, 1998
Molarity of an Ethanol Droplet (MED) Test	Liquid surface tension of droplets (dyn cm ⁻¹) Volumetric ethanol percentage (ethanol cm ³ water cm ⁻³) Molarity of ethanol droplet (mol m ⁻³) 90° surface tension	0 – 180 °	<i>In situ</i> and <i>Ex situ</i>	<ul style="list-style-type: none"> • inexpensive 	<ul style="list-style-type: none"> • not reliable on field moist samples 	Watson & Letey, 1970 King, 1981 Doerr, 1998 Dekker et al., 2009
Water Repellency Index (RI)	Unitless ratio	Unlimited	<i>In situ</i> and <i>Ex situ</i>	<ul style="list-style-type: none"> • range of scales available 	<ul style="list-style-type: none"> • standard infiltrometer requires large volumes of liquid to be transported to the field. 	Tillman et al., 1989 Perroux & White, 1998 Hallett et al., 2004

There are many ways to quantify soil water repellency, severity and persistence. These all measure different aspects of soil water repellency. As such, each method has advantages and limitations. Many studies have concluded that multiple methods of analysis are required to get a clear picture of the state of soil water repellency. Furthermore, the conditions under which soil water repellency are tested should be carefully controlled and clearly reported. Methods should be thoughtfully chosen based on the objectives of the research as each method describes different aspects of soil water repellency. The methods for measuring soil water repellency are summarized in Table 2.3.

2.1. Conclusion

Soil water repellency is naturally occurring in the AOSR in northern Alberta, Canada as a result of the dominance of coarse textured soils and organic materials. Observations of low infiltration rates at some reclaimed oil sands sites have led to suspicions that naturally occurring soil water repellency may be exacerbated as reclamation materials are salvaged, transported, stored or placed in reclamation (personal communication, C. Dubyk, May 2008). Existing knowledge about soil water repellency does not clearly support or reject this hypothesis. Soil water repellency may be more severe in reclamation materials if they dry out during handling or after use in reclamation in the absence of a protective plant community; however, soil water repellency of reclamation materials may be reduced as organic coatings are abraded by physical disturbance and organic inputs from active plant communities are decreased in newly reclaimed sites.

Soil water repellency is dependent on many inter-related and dynamic factors including soil organic matter content and quality, water content and more. The interactions between organic coatings and soil particle surfaces are complex and inconsistent. Of particular interest is the CWC or water content dependent repellency. The methods for investigating soil water repellency are just as diverse as its causes. CA, RI, WDPT, and MED tests are available to describe soil water repellency. They all have unique value and describe slightly different characteristics of soil water repellency. Furthermore, the conditions under which these measures are used greatly impact results obtained.

Water repellency has most commonly been studied as a result of forest fire (e.g. Ferreira et al., 2005). Studies have been conducted on soil water repellency associated with crude oil spills in Alberta, Canada, indicating that organic carbon of petroleum origin may lead to soil water repellency; however, similar studies on undisturbed or reclaimed sites in the AOSR have not been located. Information is needed about the naturally occurring level of soil water repellency in undisturbed sites in the AOSR. Improving the understanding of the CWC of reclamation materials may be used to inform decisions about their handling and placement. Moreover, there is a possibility that issues with soil water repellency can be prevented by using informed practices for handling and storage of reclamation materials.

3. IMPACT OF TENSION INFILTRATOR DISC SIZE ON MEASURED SOIL WATER REPELLENCY INDEX¹

Preface

The large selection of methods available for measuring soil water repellency poses challenges in designing studies and monitoring methods. Water repellency index (RI) is a reliable and thorough method to measure water repellency. Furthermore, there are many ways to measure RI itself. Is the more cumbersome, expensive and time consuming standard tension infiltrometer the most descriptive method for measuring RI? Is the simpler, more economical mini tension infiltrometer as effective at describing water repellency? The data collected at the undisturbed and reclaimed sites in the AOSR presented an opportunity to explore these questions and fill in this gap in knowledge.

3.1. Introduction

Soil water repellency occurs when the soil is not completely wettable. When the contact angle between water and soil exceeds 90° water infiltration is inhibited and the soil is termed water repellent. In extreme cases this contributes to erosion (Ellies et al., 2005), preferential flow and reduced water storage capacity (Dekker and Ritsema, 1994). Water repellency occurs in many soil types, climates and management regimes. Subcritical water repellency is used to describe the situation where a low degree of repellency impedes infiltration, but does not prevent it. Severe water repellency is the situation where water infiltration is hindered to the extent that site productivity is diminished. Critical water repellency occurs when no water infiltrates the soil. Subcritical water repellency is present in most sites around the world (Dekker et al. 2005). Previous research has focused on the challenges with severe soil water repellency with little

¹ This work has been previously published in Hunter, A.H., H.W. Chau and B.C. Si. 2011. Impact of tension infiltrometer disc size on measured soil water repellency index. *Can. J. Soil Sci.* 91:77-81. Minor modifications have been made for consistency.

emphasis on the more common subcritical water repellency. Subcritical soil water repellency is beneficial for stabilizing soil structure, preventing dispersion and minimizing erosion (Tisdall and Oades, 1982; Ellies et al., 2005). Soil water repellency has implications for soil hydraulic properties that are critical for ecosystem productivity, biodiversity, and health, especially in semi-arid environments.

Organic coatings on soil particles are the leading cause of soil water repellency (Ellies et al. 2005). Coarse textured soils have a lower surface area per unit volume than finer-textured soils. This means that coarse textured soils are more easily coated with organic matter and are more susceptible to soil water repellency (Dekker et al., 2005). Heating and drying of soil cause wetttable (hydrophilic) heads of amphiphilic organic molecules to bond to themselves and soil particles, leaving mainly hydrophobic portions exposed and exacerbating soil water repellency (Dekker and Ritsema, 1994). As such, the degree of soil water repellency is dependent on water content. However, a study by Doerr and Thomas (2000) found that drying soil after complete saturation effectively removes water repellency. Though it is generally accepted that water repellency and water content are inversely related, the relationship between the two is not completely understood. Furthermore, soil disturbance such as sieving, grinding and tillage abrade the organic coatings and may reduce or eliminate soil water repellency (Hallett et al., 2001).

Strong spatial and temporal variability of soil water repellency pose challenges in its measurement (Dekker et al., 2001; Hallett et al., 2004). Due to the dynamic nature of soil water repellency it is described in terms of both its persistence and degree. Persistence is how long soil remains repellent in the presence of water. This can be determined using the water droplet penetration time test (Doerr, 1998). The degree of soil water repellency describes how strongly infiltration is inhibited. Molarity of an ethanol droplet test (Doerr, 1988), water repellency index (RI) (Tillman et al., 1989), and several methods of measuring the contact angle (e.g. Bachmann et al., 2003) are used to describe the degree of soil water repellency.

Water repellency index is determined by comparing the soil-water and soil-ethanol sorptivities in an adjusted ratio (Tillman et al., 1989). Tension infiltrometers are used to determine the sorptivity. Tension infitrometers apply liquids at negative tensions where sorptivity drives flow (rather than gravity) and the macropore influence is negligible. Available

tension infiltrometers include miniaturized (Leeds-Harrison et al., 1994), mini and standard tension infiltrometers (Perroux and White, 1988). The miniaturized infiltrometer can be used to determine water repellency at the aggregate scale, while the mini and standard infiltrometers are most commonly applied *in situ*. The standard tension infiltrometer requires large volumes of liquid and time (up to 1 hour); however, it is adaptable both in the range of tensions and disc sizes. Alternatively, the mini tension infiltrometer is more compact, less expensive and requires less liquid and time (up to 20 min) but is limited to the 4.5 cm disc size and range of tensions. Little is known about the comparability of measured water repellency indices from these methods. Therefore, the objective of this study was to compare the mini and standard infiltrometers as means of determining RI. This information will be especially useful when choosing methods for site assessment of soil water repellency for reclamation purposes.

3.2. Materials and Methods

3.2.1. Site Description

This study was carried out on eight sites in northern Alberta and central Saskatchewan, Canada in the summers of 2008 and 2009 (Table 3.1). Seven of the eight study sites were located in northern Alberta. Four of these were undisturbed jack pine (*pinus banksiana*) stands with a lichen covered forest floor on coarse textured, nutrient poor soils, classified as A ecosites (Beckingham and Archibald 1996), including A ecosite 1 (AE1) and Soil Vegetation Plot 10, 26 and 27 (SV10, SV26 and SV27) (Table 1). Three were reclaimed from open pit oil sands mining including the Shallow-Stripping Trial (SS), Coke Cover Capping Study (CC) and the Aurora LFH Capping Study (ALFH). The eighth site, St. Denis National Wildlife Area (SD), was undisturbed grassland on loam textured Chernozemic soil located in the Dark Brown soil zone of central Saskatchewan. The loam site was included in this study to provide contrast of soil texture.

3.2.2. Sampling Design

In the summer of 2008, five points from an area approximately 10 m² at each of AE1, SV10, SV27, SS, CC, and SD sites were studied (n = 30). At each point, an area of approximately 1 m² was cleared of vegetation and surface debris and leveled with a hand shovel and straight edge, taking care to minimize compaction and disturbance. The RI was calculated using the early time sorptivity of both tap water and 95% (v/v) ethanol using the standard

Table 3.1. Study sites, location, initial soil water content, texture, density and total carbon and nitrogen.

Site	Location	Year of analysis	Initial water content (wt wt ⁻¹)	Soil texture	Bulk Density (g cm ⁻³)	Total Carbon (wt wt ⁻¹)	Total Nitrogen (wt wt ⁻¹)
<i>Undisturbed A Ecosites in northern Alberta</i>							
A Ecosite 1 (AE1)	N 57° 16' 01" W 111° 33' 29"	2008	2.9%	Sand	1.26	0.62%	0.04%
Soil Vegetation Plot (SV10)	N 57° 04' 31" W 111° 35' 40"	2008	5.7%	Sand	1.24	1.11%	0.01%
Soil Vegetation Plot (SV26)	N 57° 30' 39" W 111° 25' 48"	2009	4.0%	Sand	1.21	1.10%	0.05%
Soil Vegetation Plot (SV27)	N 57° 30' 21" W 111° 26' 12"	2008	6.9%	Sand	1.31	0.76%	0.06%
<i>Reclaimed Oil Sands Sites in northern Alberta</i>							
Shallow-Stripping Study (SS)	N 57° 15' 33" W 111° 31' 21"	2008 & 2009	4.2% (2008) 8.8% (2009)	Sandy Clay Loam	1.21 0.96	6.12%	0.33%
Coke Cover Capping Study (CC)	N 57° 00' 34" W 111° 30' 10"	2008	24.5%	Sandy Loam	0.91	8.41%	0.35%
Aurora LFH Capping Study (ALFH)	N 57° 04' 31" W 111° 30' 40"	2009	1.3%	Sand	1.23	0.96%	0.05%
<i>Grassland Site in Central Saskatchewan</i>							
St. Denis (SD)	N 52° 12' W 106° 05'	2008	25.3%	Loam	0.92	4.84%	0.33%

infiltrometer with a 20 cm diameter disc and the mini infiltrometer with a 4.5 cm diameter disc. The standard and mini infiltrometers were used according to the methods outlined by White et al. (1992) for 20 min and 10 min, respectively, at a matric potential of -3 cm. Early time sorptivity was calculated from the first two measurable movements in liquid level.

In the summer of 2009, ten points at SV26, SS and ALFH were sampled 4 m apart along a 40 m transect (n = 30). Early time sorptivity was estimated for water and ethanol to determine RI using the mini and standard infiltrometers at -3 cm hydraulic head, this time for 5 min.

3.2.3. Calculation of Soil Water Repellency

Early time sorptivity of both water and 95% (v/v) ethanol as a function of cumulative infiltration and time is described by Philip (1969):

$$S = \frac{i}{\sqrt{t}} \quad [3.1]$$

where S is sorptivity ($\text{cm hr}^{-1/2}$), i is the early cumulative infiltration (cm) and t is time (hr).

The RI is defined by Tillman et al. (1989) as the ratio of the soil-ethanol sorptivity (S_E ; $\text{cm hr}^{-1/2}$) to the soil-water sorptivity (S_W ; $\text{cm hr}^{-1/2}$)

$$RI = 1.95 \frac{S_E}{S_W} \quad [3.2]$$

Tillman et al. (1989) stated that $RI = 1.95$ is the threshold between wettable and water repellent soils.

3.2.4. Soil Physiochemical Properties

Soil water content was determined gravimetrically. Particle size distribution was measured using a Laser Scattering Particle Size Distribution Analyzer (Horiba LA – 950, Horiba Instruments Inc., Irvine, CA) after air-drying, sieving to 2 mm, removing organic matter using peroxide, and dispersing samples using sodium hexametaphosphate (Eshel et al., 2004). Bulk density was determined on undisturbed samples collected in cores (5 cm deep and 7.5 cm in diameter) from each sampling point. Total C and N contents were determined using a LECO CNS – 2000 analyzer (LECO Corp., St. Joseph, MI). These results are reported in Table 3.1.

1.1.1. Statistical Analysis

Statistical analysis was conducted using Sigma Plot v. 11. The assumptions of normality and homoscedasticity could not be satisfied by logarithmic, square root or exponential transformations. Therefore, the non parametric Mann-Whitney rank sum test was used to test our hypothesis. A significance level of $\alpha = 0.05$ was used.

3.3. Results and Discussion

There was strong variability in RI values between the different infiltrometer methods and sites. The site means and coefficients of variation (CV) of RI varied from 1.23 to 24.6 and 39% to 164%, respectively (Table 3.2). The mean and CV of all RI values from the mini infiltrometers were higher than from the standard infiltrometer (9.61 and 3.46 and 180% and 110%, respectively). The variability within sites dominated the variability in RI for the two methods. Despite these obvious trends, RI values between infiltrometer sizes were statistically different for only two individual sites using the Mann-Whitney rank sum test (Table 3.2).

Table 3.2. Mean and coefficient of variation (CV) of water repellency index (RI) values for all sites for mini and standard tension infiltrometers in 2008 and 2009 with the *P* values generated from Mann-Whitney rank sum tests.

Site (year)	Sample Size n	-- 4.5 cm diameter disc --		-- 20 cm diameter disc --		<i>P</i> value
		Mean RI	CV (%)	Mean RI	CV (%)	
AE1 (08)	5	24.6	164	7.9	105	0.69
SV10 (08)	5	7.0	91	1.2	108	0.03**
SV26 (09)	10	5.4	64	1.6	135	0.07
SV27 (08)	5	2.3	65	2.8	81	>0.99
SS (08)	5	6.3	74	1.2	108	0.03**
SS (09)	10	15.6	153	4.1	75	0.43
ALFH (09)	10	15.0	114	6.0	65	0.35
CC (08)	5	1.3	39	2.0	70	0.55
SD (08)	5	1.9	90	3.0	83	0.55

** significant at the $P \leq 0.05$ level of probability

At each of the sites, five points for 2008 and 10 points for 2009 were selected for measurements of RI. Because of the inherent spatial variability in soil water repellency, there was strong variability in measured RI values (Table 3.2). Remarkably, an increase in the number of sampling points from 5 to 10 did not substantially decrease the CV values (Table 3.2). Therefore, a prohibitively large number of RI measurements are required to increase the power of the statistical test.

The mean RI and CV at each site were positively correlated ($r^2 = 0.45$), especially for $RI > 4$ ($r^2=0.86$) (Figure 3.1). Consistent with the observation of higher values with greater variability under smaller disc sizes, the top two RI and CV values were from the mini tension infiltrometer. The higher variability and mean values under smaller disc sizes was expected because of a smaller zone of influence for each sampling point (Sisson and Wierenga, 1981).

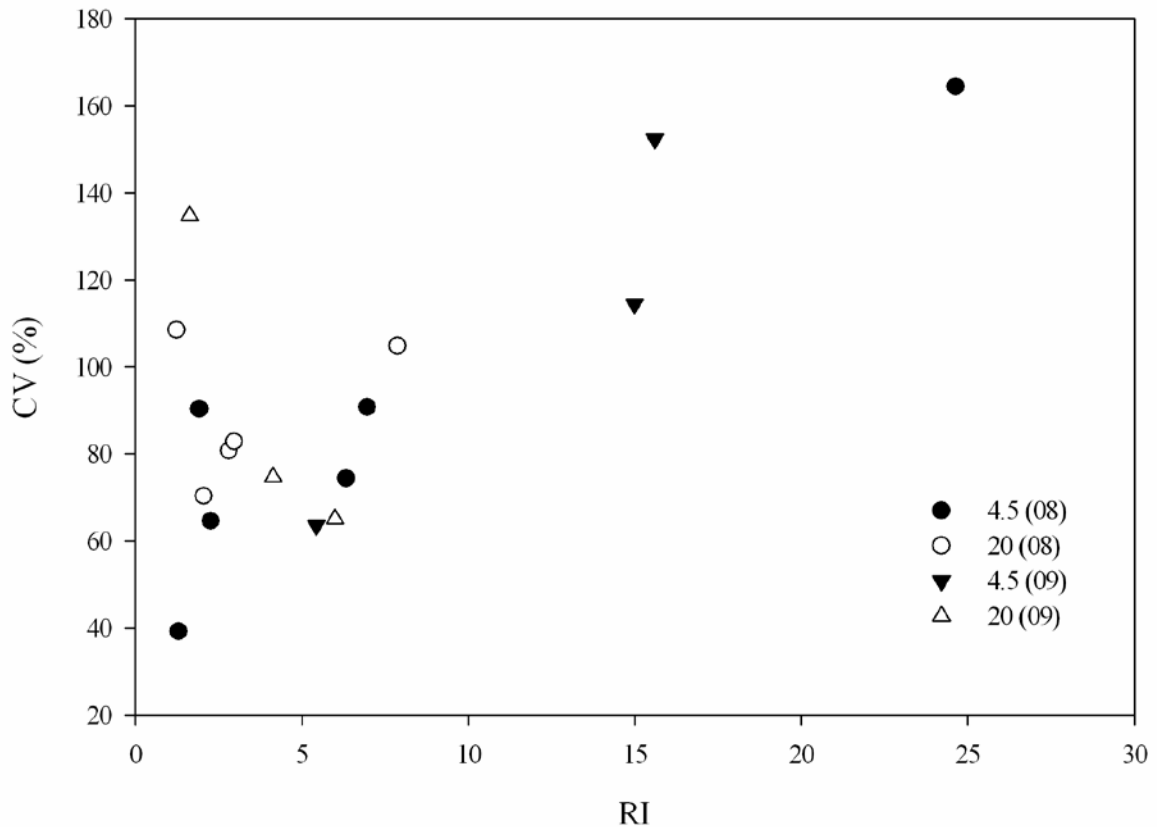


Figure 3.1. Comparison between water repellency index (RI) and coefficient of variation (CV) for each disc size in 2008 and 2009.

These results are consistent with those by Hallett et al. (2004) and Sepaskah et al. (2005). Hallett et al. (2004) compared RI measurements from the ponded ring infiltrometer (7.4 and 11 cm diameter) to the tension infiltrometer (8 and 0.014 cm diameter disc) and also found RI values were inversely related to infiltrometer size. Sepaskah et al. (2005) compared sorptivity values determined using a 10 cm diameter ponded ring infiltrometer to that from a ring 4 to 5 times larger. They observed higher sorptivity values and variability using the smaller ring sizes but concluded that the methods were comparable despite the minor variances. In the Sepaskah et

al. (2005) study, macropore flow may have contributed to the variability in measured sorptivity from smaller ring infiltrometers due to the strong spatial variability of macropores. In this study, macropore flow was eliminated by infiltration under a matric potential of -3 cm. However, heterogeneity of water repellent soils could have also contributed to the high variability in measured RI using the smaller ring size. Furthermore, coarse textured soils tend to be more water repellent than finer-textured soils. In the range of soil textures considered in this study (sand to loam), there was no significant trend between soil texture and RI.

Though there are obvious trends between RI measured from the mini and standard infiltrometers, the mini infiltrometer is an appropriate tool for site assessment of water repellency in reclamation. To this end, Figure 3.2 compares the RI from both the standard and mini infiltrometers at each site. The standard infiltrometer RI values were used as the control against which the mini infiltrometer values were compared. The RI values from both methods are similar ($r^2 = 0.73$).

The statistical terms ‘type I error’ and ‘type II error’ are used to describe cases in which the results from the different methods do not yield the same classification. When both methods indicate that a site is water repellent, the result is called a ‘true positive’. A type I error (false positive) occurs when the mini infiltrometer incorrectly indicates that the RI exceeds the threshold. A type II error (false negative) results when the mini infiltrometer under-represents the RI. If error occurs, type I error is desirably more cautious. This will flag water repellency as a concern when it is not, rather than potentially allowing water repellency to exist undetected.

When $RI > 1.95$ was used as the threshold between wettable and water repellent soils, there was a rate of 33% type I error (false positive) where the standard infiltrometer $RI \leq 1.95$ while mini infiltrometer $RI > 1.95$. Type II error (false negatives), where the standard infiltrometer indicated that the site was water repellent and the mini infiltrometer indicated that the site was not, occurred in 22% of cases. For this study, there was 44% consistency between the standard and mini tension infiltrometers.

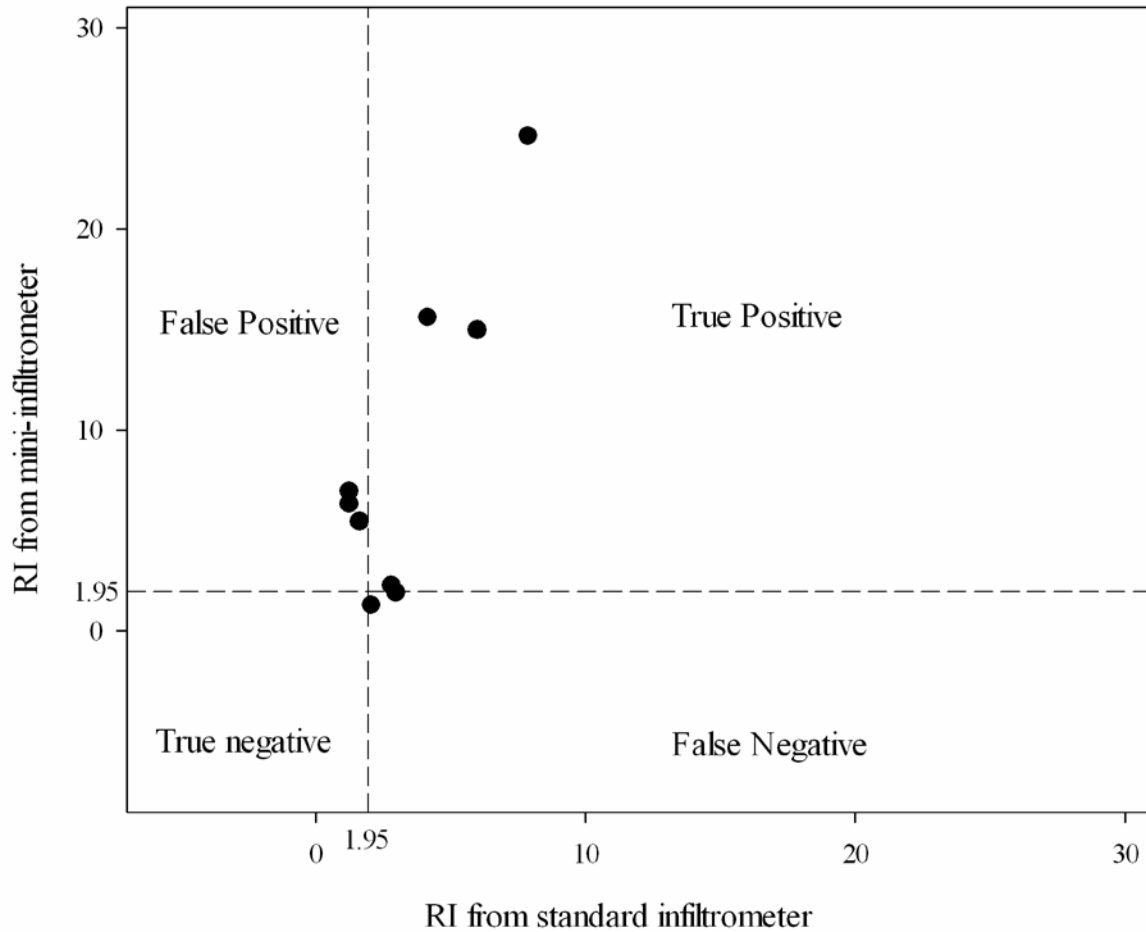


Figure 3.2. Scatter plot of water repellency indices (RI) from the standard and mini tension infiltrmeters. Soil with $RI > 1.95$ is considered water repellent and soils with $RI \leq 1.95$ are considered wettable.

For reclamation purposes, water repellent materials should be placed on flat landscapes to prevent potential runoff and erosion, while wettable materials may be placed on sloping landscapes. False negatives could result in reclamation failure due to runoff if the tested materials were improperly placed on the landscape. The mini tension infiltrrometer method will seldom result in a false negative RI, and thus may be used for identifying wettable cover materials for reclamation of sloping landscapes.

3.4. Conclusion

There was strong variability in measured soil water repellency indices within and among sites. Although there were differences in measured RI, they were seldom statistically significant

at a site. If $RI = 1.95$ is used as the threshold between wettable and water repellent soil, the mini infiltrometer yielded a type I error response for 3 out of the 9 sites studied with a 22% incidence of type II error (false negative). The result of type I error is preferred to type II error, as it will lead to conservative use of reclamation materials. This suggests that the mini infiltrometer would be well suited for *in situ* analysis of soil water repellency at the site level. The mini infiltrometer allows for more efficient and extensive monitoring of water repellency.

4. SOIL WATER REPELLENCY AND CRITICAL WATER CONTENT OF UNDISTURBED AND RECLAIMED OIL SANDS SITES IN NORTHERN ALBERTA, CANADA

Preface

The previous chapter compared methods for measuring soil water repellency index *in situ*. This method, along with the WDPT and MED tests were used to investigate the water repellency at undisturbed and reclaimed oil sands sites in order to establish the naturally occurring level of water repellency and to determine whether current reclaimed sites differ. If water repellency is more prevalent in current reclaimed sites, are there ways to minimize water repellency in future reclamation projects? The CWC of reclamation materials was investigated as a means to provide information that may be used to prevent or mitigate challenges with severe water repellency.

4.1. Introduction

The Athabasca Oil Sands Region (AOSR) in northern Alberta, Canada is home to the largest oil sands deposits in the world. The Athabasca, Peace River and Cold Lake oil sands region in Alberta, covers 140 000 km² (Johnson and Miyanishi, 2008). This area is rich in bitumen deposits, a mix of heavy oil and sand, from which sweet crude oil is produced. A large proportion of bitumen is excavated from up to 100 m below the soil surface in open-pit mines, removing whole ecosystems. After mining, sites are to be restored to their original land capacity.

Naturally occurring surface soils are carefully salvaged and stored in stockpiles for use in reclamation. Some surface soils in the AOSR contain chunks of naturally occurring bitumen deposits called tarballs (Fleming et al., 2011) Organic materials are used in mixtures with mineral materials in order to increase organic content, and provide a propagule bank for newly reclaimed sites (Mackenzie and Naeth, 2007; Mackenzie and Naeth, 2010). These resources are referred to as reclamation materials. Physical disturbance and changes in air flow patterns, water regimes, vegetative and microbial communities during storage may alter the properties of the stockpiled reclamation materials.

Though there are several reclamation successes, observations of erosion and low water infiltration rates have lead researchers to consider soil water repellency (or hydrophobicity) as a possible hindrance in reclamation (personal communication, C. Dubyk, May, 2008). Subcritical soil water repellency, when water infiltration is slowed, is naturally occurring and is often an asset to soil (Tisdall and Oades, 1982; Ellies et al., 2005). In extreme cases, water repellency may completely prohibit infiltration, or restrict it detrimentally (Dekker and Ritsema, 1994), leading to preferential flow (Buczko and Bens, 2006), runoff and erosion (Ellies et al., 2005). This may limit the productivity of reclaimed sites, as plant and microbial communities depend on soil water to thrive and survive.

Soil water repellency is caused by amphiphilic organic or hydrocarbon coatings on soil particles. The interaction between these organics and soil particles is largely governed by water content (Doerr et al., 2000; Ellies et al., 2005). As soils dry, organic particles bind to soil particles, when soil is being wetted, these particles are liberated into solution. Drying may intensify soil water repellency as mainly hydrophobic portions of organic matter remain exposed, while wetting can mitigate soil water repellency by exposing hydrophilic portions (Doerr, 1998; Lichner et al., 2007). Dry soil becomes wettable above a threshold water content. Likewise moist, wettable soil may become water repellent when dried. This threshold is called the critical water content (CWC). This is most commonly reported as a range rather than a single water content (Dekker and Ritsema, 1994; Ritesema and Dekker, 1994; de Jonge et al., 1999; Dekker et al., 2001).

The low surface area per unit volume of coarse textured soils means that less organic matter is required to coat particle surfaces (Doerr et al., 2000). As such, coarse textured soil is more susceptible to challenges with soil water repellency (Tschapek, 1984; Harper and Glikes, 1994; Karunarathna et al., 2010), though water repellency has also been observed in fine textured soils (Dekker et al., 2005). Because sandy and organic soils dominate the AOSR, water repellency is likely to be naturally occurring in the region. As such, reclamation materials (mineral soil, peat and LFH) may be vulnerable to soil water repellency; however, physical disturbance that happens during handling and transport of reclamation material may abrade the organic coatings and reduce or eliminate water repellency.

Though there have been studies on soil water repellency in crude oil contaminated sites in Alberta, Canada (Roy and McGill, 1998; Roy and McGill, 2000; Roy et al., 2003), there is a lack of information about the incidence of naturally occurring soil water repellency and its role in reclamation in the AOSR. As such, the objectives of this study were to investigate 1) the range of soil water repellency *in situ* for undisturbed and 2) reclaimed sites and CWC of reclamation materials.

4.2. Materials and Methods

4.2.1. Site Description

The AOSR of Northeastern Alberta, Canada is located in the Boreal Mixedwood ecological area, dominated by the Central Mixedwood Natural Subregion (Beckingham and Archibald 1996). Coarse textured soils prevail in the uplands with organic bogs and fens dominating the lowlands. Many uplands are open canopied jack pine (*pinus banksiana*) stands with a lichen layer covering coarse textured, nutrient poor, acidic soils that are classified as A ecosites (Beckingham and Archibald 1996).

4.2.2. Measurement of Water Repellency of Undisturbed and Reclaimed Sites

This study was conducted on nine sites in the AOSR in the summers of 2008 and 2009. A range of undisturbed and reclaimed sites were examined. Five undisturbed A ecosites were studied, including A ecosite 1 and 2 (AE1 and AE2) and soil vegetation plots 10, 26 and 27 (SV10, SV26 and SV27) (Table 4.1). Four reclaimed sites were studied, including the shallow stripping trial (SS), coke cover capping study (CC), Aurora LFH capping study (ALFH) and south west 30 (SW30) (Table 4.2). Mackenzie and Naeth (2007 and 2010) studied and reported on the ALFH site in detail. Reclaimed sites varied in composition, but were generally a mix of peat and mineral soil over tailings sand (Figure 4.1).

RI was used to determine the severity of water repellency. In 2008 five undisturbed A ecosites and four reclaimed sites were studied. Five surface and subsurface points were sampled at each site. Surface refers to 0 cm depth, while subsurface refers to 20 cm depth for undisturbed sites and the top of the second layer of materials for reclaimed sites. In 2009 the sampling intensity was increased to 40 surface points per site at only one undisturbed and two reclaimed sites. The number of sampling points were limited to a number that could be completed in one

Table 4.1. Location, gravimetric water content, soil texture, bulk density, total carbon and total nitrogen of undisturbed A ecosites.

Site	Location	Year of Analysis	Gravimetric Water Content †		----- Soil Texture -----		Bulk Density (g cm ⁻³)	---- Total Carbon ---- (g g ⁻¹)		--- Total Nitrogen --- (g g ⁻¹)	
			Surface	Subsurface	Surface	Subsurface		Surface	Subsurface	Surface	Subsurface
A Ecosite 1 (AE1)	N 57° 16' 01" W 111° 33' 29"	2008	2.7 % (0.9)	3.0 % (1.5)	Loamy Sand	Sandy Clay Loam	1.26	0.62	0.49	0.04	0.04
A Ecosite 2 (AE2)	N 57° 12' 33" W 111° 31' 17"	2008	5.7 % (1.8)	4.2 % (0.5)	Loamy Sand	Sand	--	0.97	0.51	0.06	0.04
Soil Vegetation Plot (SV10)	N 57° 04' 31" W 111° 35' 40"	2008	5.4 % (2.6)	4.7 % (1.4)	Sand	Loamy Sand	1.24	1.11	0.73	0.06	0.05
Soil Vegetation Plot (SV26)	N 57° 30' 39" W 111° 25' 48"	2008	4.6 % (1.3)	3.5 % (0.4)	Sand	Sand	1.21	0.65	0.25	0.04	0.03
		2009	8.0 % (2.4)	6.9 % (4.4)	Sand	Sand	1.21	1.10	0.38	0.05	0.03
Soil Vegetation Plot (SV27)	N 57° 30' 21" W 111° 26' 12"	2008	7.7 % (2.4)	--	Sand	---	1.31	0.76	---	0.06	---

† Values are reported as mean and coefficient of variation

Table 4.2. Location, gravimetric water content, soil texture, bulk density, total carbon and total nitrogen of of reclaimed sites.

Site	Location	Year of Analysis	Gravimetric Water Content †		----- Soil Texture -----		Bulk Density (g cm ⁻³)	---- Total Carbon ---- (g g ⁻¹)		--- Total Nitrogen --- (g g ⁻¹)	
			Surface	Subsurface	Surface	Subsurface		Surface	Subsurface	Surface	Subsurface
Shallow-Stripping Study (SS)	N 57° 15' 33" W 111° 31' 21"	2008	4.2 % (4.4)	5.7 % (3.4)	Sandy Loam	Loamy Sand	1.21	2.69	5.51	0.13	0.35
		2009	8.8 % (8.6)	13.0 % (10.0)	Sandy Loam	Sandy Loam	0.96	6.12	10.35	0.33	0.58
Coke Cover Capping Study (CC)	N 57° 00' 34" W 111° 30' 10"	2008	25.0 % (3.4)	7.3 % (2.0)	Sandy Clay Loam	Sandy Loam	0.91	8.41	4.41	0.35	0.23
Aurora LFH Capping Study (ALFH)	N 57° 04' 31" W 111° 30' 40"	2008	1.2 % (0.4)	1.1 % (0.3)	Sand	Sand	1.23	0.96	0.33	0.05	0.03
		2009	7.5 % (3.2)	5.6 % (1.7)	Sand	Sand	---	1.28	0.41	0.07	0.02
South West 30 (SW30)	N 56° 59' 48" W 111° 37' 11"	2008	37.2 % (2.2)	40.5 % (4.0)	Heavy Clay	Clay Loam	---	11.85	4.30	0.47	0.25

† Values are reported as mean and coefficient of variation

Depth (cm)	SS	CC	ALFH	SW30
0 – 10	LFH and Ae horizon mix	peat and	LFH	peat and
10 – 20	peat and	mineral soil mix	peat and	mineral soil mix
20 – 30	mineral sand			
30 – 40	mix			
40 – 50	tailings sand	tailings sand	mineral soil mix	Sodic overburden
50 – 60				
60 – 70				
70 – 80				
80 – 90				
90 – 100				

Figure 4.1. Reclamation prescriptions of the shallow stripping study (SS), the coke cover capping study (CC), Aurora LFH capping study (ALFH) and the south west 30 (SW30) sites.

day. At each point, approximately 1 m² was cleared of vegetation and leveled with a hand shovel and straight edge. Care was taken to minimize compaction and disturbance.

The mini tension infiltrometer (Perroux and White, 1988; White and Perroux, 1992; Hunter et al., 2011) was used to determine the water repellence index (RI) *in situ* (Tillman et al., 1989). For this, the water-soil and ethanol-soil sorptivities were determined by infiltrating tap water and 95% ethanol at a matric potential of -3 cm for 5 min using a mini tension infiltrometer with a 4.5 cm disc. Early time sorptivity was calculated through the relationship described by Philip (1969):

$$S = \frac{i_2 - i_1}{\sqrt{t_2} - \sqrt{t_1}} \quad [4.1]$$

where S is sorptivity (cm hr^{-1/2}), i_1 and i_2 is infiltration (cm) at time t_1 and t_2 (hr), respectively.

The soil-water and soil-ethanol sorptivities were compared using the water repellency index (RI) (Tillman et al., 1989):

$$RI = 1.95 \frac{S_E}{S_W} \quad [4.2]$$

where S_E and S_W are the soil-ethanol and soil-water sorptivities ($\text{cm hr}^{-0.5}$) respectively.

The WDPT test was used to determine the persistence of water repellency at all sampling points used for RI in 2008. In 2009, the WDPT test was used at the surface of 10 points only. The time for infiltration of 10, 10 μL droplets were averaged for each sampling point. Five seconds was used as the cutoff between water repellent and wettable as has been done by Watson and Letey (1970), King (1981), Dekker and Ritsema (1994), Bachmann et al. (2003). If the droplet penetration time was $\geq 300\text{s}$, 300 s was used in the calculation of the mean. Blanks occur in the data set where measurement was forgotten in error.

The MED test was used to determine small scale severity of water repellency in 2008 at the same 5 sampling points as RI and the WDPT test. This test was not repeated in 2009 because of the similar trends between the measures being used. For this analysis, 10 μL droplets of ethanol dilutions starting at 0% and increasing by 8% by volume were applied to the soil surface until the droplets infiltrated within three seconds as was done by Doerr (1998). The lowest ethanol concentration to infiltrate in under three seconds was reported in the results. Again, some holes in data occur where measures were forgotten in error.

4.2.3. Measurement of Critical Water Content of Reclamation Materials

The CWC of mineral and organic reclamation materials was examined. Samples were collected alongside the *in situ* study from the surface of five points at each site with the exception of peat. All samples were collected in 2008, with the exception of LFH which was collected in 2009. Reclamation materials examined in this study are summarized in Table 4.3.

Mineral soil from two reclaimed sites (SS and CC) were used. Tarball affected sites, labeled with tarballs (WTB) and without tarballs (WOTB) were also examined. These samples were very coarse textured (97% sand) and nearly devoid of organic matter and nutrients. The tarball affected soils were also studied by Fleming et al. (2011).

Peat samples were excavated from a 120 cm pit and were categorized by their level of decomposition. Samples were accordingly named Fibric Peat (FP), Mesic Peat (MP) and Humic Peat (HP). The von Post decomposition level (Soil Classification Working Group, 1998) of FP, MP, and HP were 3, 6 and 8, respectively. Lichen and leaf litter on the forest floor (or LFH) samples from AE1, AE2, SV10, SV26 and SV27.

The CWC of reclamation materials was investigated by determining the relationship between water repellency and water content. Water contents were altered by placing approximately 1 g of air dried sample at ambient room temperature and humidity into a plastic bag, adding water and allowing the soil to equilibrate for 5-10 days as was done by de Jonge et al. (1999). Samples were brought to gravimetric water contents ranging from 0-25 % in increments of approximately 2.5 %.

Table 4.3. Reclamation materials used in the critical water content study.

----- Mineral Soil -----		----- Organic material -----	
Reclaimed Soil	Tarball Affected Soil	Peat	LFH
shallow stripping trial (SS)	with tarballs (WTB)	fibric peat (FP)	A ecosite 1 (AE1)
coke cover capping study (CC)	without tarballs (WOTB)	mesic peat (MP)	A ecosite 2 (AE2)
		humic peat (HP)	soil vegetation plot 10 (SV10)
			soil vegetation plot 26 (SV26)
			soil vegetation plot 27 (SV27)

Water contents were calculated gravimetrically using the weight of the dry sample and the weight of water that was initially added. Ideally, the final water content would be calculated before the CA and WDPT were measured. Erroneous results were produced when water contents were calculated by subtracting the initial dry weight from the final wet weight, with many values equaling large negative water contents. Water contents reported here were calculated using the weight of water added at the time of the addition. As such, water contents are to be considered as relative values only. Figures with water contents calculated using the final weight of the sample just before measurement are reported in Appendix A. This issue resulted from the high margin of error created by using a small amount of sample (approximately 1 g) and the instability of the

plastic bags on the balance. The bags were continually moving and touching the sides of the balance. This problem may be mitigated in the future by 1) using a larger amount of sample; 2) using a more rigid container (e.g. a dram vial); and 3) taking a subsample of the wet material and determining the water content by oven drying it, rather than relying on subtracting original values.

A PG-X goniometer (FIBRO System AB, 2006) was used to capture digital images of a 4 μ L droplet on a flattened sample surface over time. From these images, the CA and WDPT were determined. The CA was calculated using the multi-platform java image processing program, Image J (available at: <http://rsbweb.nih.gov/ij/>) using the Low Bond Axisymmetric Drop Shape Analysis Model of Drop Shape Analysis plug in (Stalder et al., 2010) (available at <http://bigwww.epfl.ch/demo/dropanalysis/>). The WDPT was determined by observing the last recorded time that the droplet remained on the soil surface.

4.2.4. Soil Physiochemical Properties

Standard methods were used to determine physical characteristics of sites and samples (Dane and Topp, 2002). Water content was determined gravimetrically. The bulk density was determined on undisturbed samples collected in cores of 5 cm depth and 7.5 cm in diameter. Bulk density measures are missing for SV27 (2008), ALFH (2009) and SW30 because of an error made in sample collection. Bulk density Total carbon and nitrogen content were determined on ball milled samples using a LECO CNS – 2000 analyzer. These results are reported in Table 4.1 and Table 4.2.

Particle size distribution analysis was carried out on organic matter-free, dispersed samples. Samples were air dried and sieved to 2 mm then organic matter was removed using 30% hydrogen peroxide (Gee and Or, 2002) for 2008 surface samples and 6% sodium hypochlorite (common household bleach) (Mikutta et al., 2005) for 2008 subsoil and all 2009 samples. Samples prepared using the peroxide were dispersed using 150% calgon solution, while those treated with bleach required no further dispersion (Mikutta et al., 2005). Different methods were used for particle size analysis because information about the efficacy of bleach in removing organic matter was located after the initial analysis was completed. The bleach method was chosen for economic reasons. In both cases, samples were dried down to a thick paste and analyzed using a Horiba LA-950 Laser Scattering Particle Size Distribution Analyzer as per the

laser diffraction method outlined by Eshel et al. (2004). The instrument measures particles 0.011 – 3000 μm in diameter. Particle size fractions and texture classes were assigned according to the Canadian System of Soil Classification (Soil Classification Working Group, 1998). Sand, silt and clay particles range from <0.002 mm, 0.002 – 0.05 mm and 0.05 - 2 mm respectively. Texture classes are reported in Table 4.1 and Table 4.2. A detailed report of the particle size distribution is presented in Appendix B. Hydrocarbon analysis was also measured, but was not correlated to results here. Results from hydrocarbon analysis are reported in Appendix C.

4.2.5. Statistical Comparison of Reclaimed and Undisturbed Sites

Statistical analysis was conducted using Sigma Plot v. 11. The assumptions of normality and homoscedasticity could not be satisfied using a logarithmic, square root or exponential transformation. Therefore, the non parametric Mann-Whitney ranked sum test was used at a significance level of $\alpha = 0.05$. The coefficient of variation (CV) of each measure are provided to illustrate the spatial variability and range of each measurement. The overall mean and CV of reclaimed and undisturbed sites are calculated using the measures taken at individual points to minimize errors of weighting the results from each sites.

4.2.6. Statistical Analysis for Critical Water Content Analysis

For the analysis of CWC, scatter plots are used to illustrate the relationship between water content and water repellency. Each point represents the average of the five replicates at each water content. Error bars are used in figures 4.2 and 4.3, but not in 4.4 and 4.5. The variability was so high for the peat and LFH samples that the inclusion of error bars rendered the figures illegible.

The relationship between water content and water repellency was quantified using r^2 of a linear relationship. This is a flawed model, as the relationship is not expected to be linear; however, no better relationship was established. The WDPT of peats of differing decomposition level were compared using ANOVA with Tukey's multiple comparison test in Sigma Plot v. 11.

4.3. Results and Discussion

1.1.2. *In Situ* Comparison Between Reclaimed and Undisturbed Sites

Surface RI from the reclaimed and undisturbed sites ranged from 1.3 to 34.0 with the CV ranging from 39 to 241 %, while the subsurface RI ranged from 0.6 to 16.3 with the CV ranging from 55 to 224 % (Table 4.4). These values are within the range of RI values presented in Tillman et al. (1989) and Wallis et al. (1991). The high CV illustrated the naturally high spatial variability of soil water repellency as discussed by Leighton-Boyce et al. (2005) and Sepaskhah et al. (2005).

At undisturbed sites, surface RI ranged from 2.3 to 24.6 with an average of 8.8, while the subsurface RI averaged 5.8 and ranged from 1.9 to 16.3. The surface of reclaimed sites had an average RI of 12.2 and ranged from 1.3 to 34.0, while RI from the subsurface of reclaimed sites ranged from 0.6 to 6.5 and averaged 3.7. The average RI value for the surface of reclaimed sites was higher than that of subsurface at reclaimed sites; however, there were no statistical differences between RI values of surface reclaimed and undisturbed sites ($P=0.213$), or subsurface of reclaimed and undisturbed sites ($P=0.717$).

Overall, surface WDPT ranged from 0.0 to 121.5 s with the CV ranging from 0 to 307 % (Table 4.4). The overall subsurface WDPT ranged from 0 to 187.3 s with the CV ranging from 0 to 224 %. These values are within the range of WDPT values presented in Wallis et al. (1991). The high CV illustrated the naturally high spatial variability of soil water repellency as discussed in Regalado and Ritter (2005).

At undisturbed sites, surface WDPT ranged from 0.1 to 84.1 s and averaged 20.9 s, while the subsurface WDPT ranged from 0.0 to 60.0 s and averaged 20.0 s. The WDPT of reclaimed sites ranged from 0.0 to 121.5 s and averaged 33.2 s at the surface. The WDPT at the subsurface of reclaimed sites ranged from 0.0 to 187.3 s and averaged 78.1 s. There was no statistical difference between WDPT of reclaimed and undisturbed surface soils ($P=0.810$); however, reclaimed subsurface soils were statistically higher than undisturbed subsurface soils ($P=0.046$).

The overall surface MED ranged from 0.0 to 12.0 % ethanol with the CV ranging from 0 to 200 % (Table 4.4). The subsurface MED ranged from 0.0 to 18.4 % ethanol with CV ranging

from 0 to 224 %. These values are also within the range of MED values presented in Wallis et al. (1991). At the surface of undisturbed sites, MED ranged from 0.0 to 12.0 % ethanol and averaged 4.2. Subsurface MED ranged from 0.0 to 4.8 % ethanol and averaged 1.7 %. The MED of reclaimed sites ranged from 0.0 to 10.0 % ethanol and averaged 3.3 %. Subsurface MED at reclaimed sites ranged from 0.0 to 18.4 % ethanol and averaged 8.0 %. Again, there was no statistical difference between MED of reclaimed and undisturbed surface soils ($P = 0.982$) or subsurface soil ($P=0.085$).

The measured RI, WDPT and MED values were most commonly greater at the surface than the subsurface. This is consistent with many other studies on soil water repellency and depth (Dekker et al., 2001) and corresponds to the often higher water content at the soil surface (Tables 4.1 and 4.2). Exceptions to this norm occurred for all three measures at SV10 in 2008, WDPT and MED at CC in 2008 and RI at SV26 and SS in 2008. It is suspected that surface soil at SV10, an undisturbed A ecosite, has a higher hydrocarbon content than the other undisturbed sites, based on hydrocarbon analysis on LFH at this site (Appendix C). This comparison cannot be made directly with the current data set, but further investigation is warranted. The higher water content at the subsurface at the reclaimed CC site is likely caused by heat from the coke underlying this site. Coke is a byproduct of the upgrading process, and was originally placed hot, leaving coke piles to smolder, sometime for years. Heat from the coke pile is implicated in many unusual hydrological behaviors of this site (Dr. S. Lee Barbour, 2009, Personal Communication).

Though RI, WDPT and MED were often higher for reclaimed than undisturbed sites, the differences were seldom statistically significant. There was a similar range and trend between reclaimed and undisturbed sites and the different methods used. This suggests that current reclamation practices do not exacerbate soil water repellency.

4.3.1. Critical Water Content of Reclamation Materials

The clearest relationship between soil water repellency and water content was present for the mineral soil samples (CC and SS) (Figure 4.2). Generally, CA was inversely related to water content. Variability was high and the relationship between water content and water repellency was weak. At SS, $r^2=0.92$ for the linear relationship between CA and water content and $r^2=0.15$ between WDPT and water content. At CC, $r^2=0.53$ for the linear relationship between CA and

Table 4.4. Mean and coefficient of variation (CV) of water repellency index (RI), water droplet penetration time (WDPT) and molarity of an ethanol droplet (MED) test values for undisturbed and reclaimed sites in 2008 and 2009.

Site (year)	Number of samples for RI measures (n)		RI†		WDPT (s)†		MED (eth. conc. %)†	
	Surface	Subsurface	Surface	Subsurface	Surface	Subsurface	Surface	Subsurface
<i>Undisturbed Sites</i>								
AE1 (2008)	5	5	24.6 (164)	5.4 (61)	84.1 (156)	0.2 (224)	12.0 (82)	0.0 (0)
AE2 (2008)	5	5	3.1 (54)	3.0 (55)	0.1 (224)	0.0 (0)	0.0 (0)	0.0 (0)
SV10 (2008)	5	5	7.0 (91)	16.3 (173)	1.5 (114)	60.0 (224)	1.6 (200)	4.8 (224)
SV26 (2008)	5	5	4.4 (82)	6.3 (112)	12.2 (224)	---	0.0 (0)	---
SV26 (2009)	40	10	7.2 (130)	1.9 (59)	3.1 (307)	---	---	---
SV27 (2008)	5	5	2.3 (65)	---	42.3 (222)	---	6.4 (200)	---
Mean (CV)			8.8 (175)	5.8 (207)	20.9 (305)	20.0 (386)	4.2 (216)	1.7 (374)
<i>Reclaimed Sites</i>								
SS (2008)	5	5	6.3 (74)	6.5 (80)	2.0 (91)	0.0 (0)	0.0 (0)	0.0 (0)
SS (2009)	40	10	11.1 (171)	4.3 (63)	57.0 (135)	---	---	---
CC (2008)	5	5	1.3 (39)	0.6 (224)	0.0 (0)	187.3 (83)	0.0 (0)	18.4 (54)
ALFH (2008)	5	5	34.0 (109)	6.0 (81)	0.0 (0)	---	10.0 (77)	0.0 (0)
ALFH (2009)	40	10	12.1 (241)	3.4 (79)	121.5 (106)	0.3 (200)	---	---
SW30 (2008)	5	5	1.5 (76)	1.0 (76)	---	---	---	---
Mean (CV)			12.2 (221)	3.7 (97)	33.2 (216)	78.1 (172)	3.3 (190)	8.0 (159)

† Values are reported as mean and coefficient of variation

water content and $r^2 = 0.80$ between WDPT and water content (Table 4.4). Though CWC was not clearly identifiable due to the high variability, soil was generally wettable above gravimetric water contents of 5-10% for both CA and WDPT. Similar trends were seen between CA and WDPT, though SS was proportionally less water repellent at low water contents as measured by the WDPT test.

Coarse textured tarball affected soils (WTB and WOTB) were less water repellent than the reclaimed mineral soils studied. This is suspected to result more from the difference in organic content than the hydrocarbon content of these two reclamation materials. There was a strong relationship between water content and water repellency for these samples. There was no difference in the behavior of WTB and WOTB, suggesting that direct contact with tarballs does not increase the risk of severe water repellency. This result supports the recommendation by leming et al. (2011) that tarball affected materials may be appropriately used in reclamation covers.

There was no strong relationship between water content and soil water repellency for peats (Figure 4.4). The r^2 was 0.07, 0.00 and 0.08 between soil water content and CA of fibric, mesic and humic peat, respectively (Table 4.5). A similar lack of relationship between water repellency and water content was noted for LFH (Figure 4.5), where AE1, AE2, SV10, SV26 and SV27 has r^2 values of 0.01, 0.02, 0.00, 0.00, and 0.00, respectively between CA and water content (Table 4.5).

Though there are many studies on CWC of mineral soils, the CWC of organic materials is not well understood. The existing information shows a strong relationship between soil water repellency and water content for mineral materials and implicates organic materials as the leading cause. This implies that organic materials themselves are likely to follow a similar trend. This study found no relationship between water content and water repellency of fibric, mesic or humic peat.

Though there was no strong relationship between water content and water repellency, both peat and LFH hovered around the mid-range of wettability and straddled the threshold

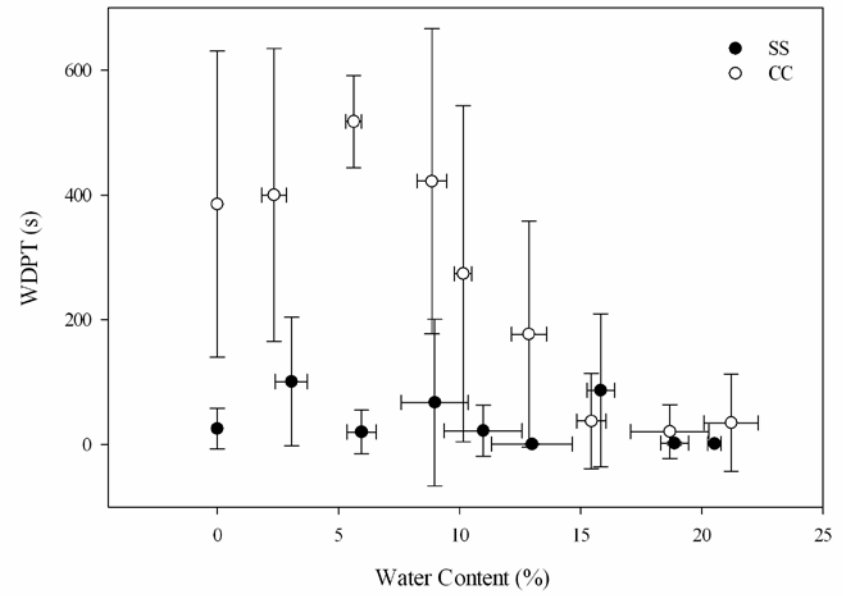
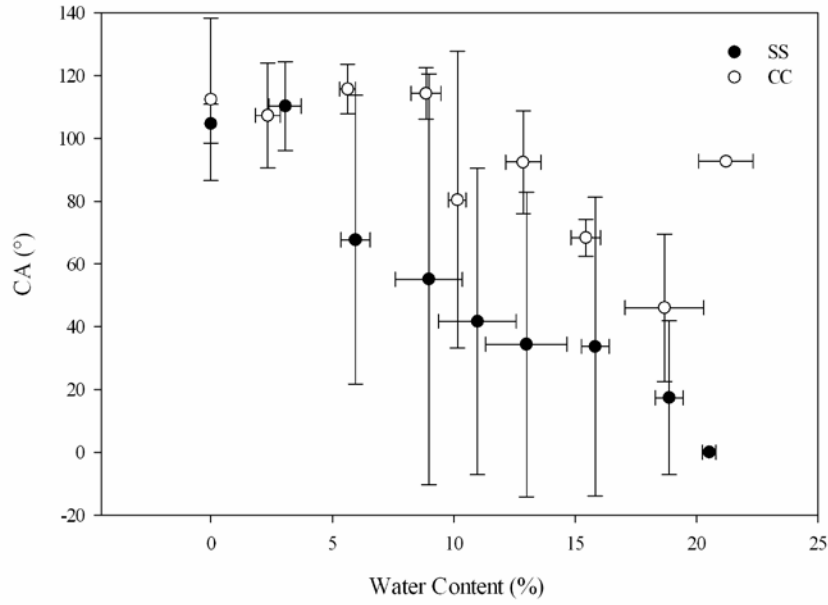


Figure 4.2. Relationship between soil water repellency and gravimetric water content based on contact angle (CA) and water droplet penetration time (WDPT) for reclaimed mineral soil at the shallow stripping (SS) trial and the coke cover capping (CC) study.

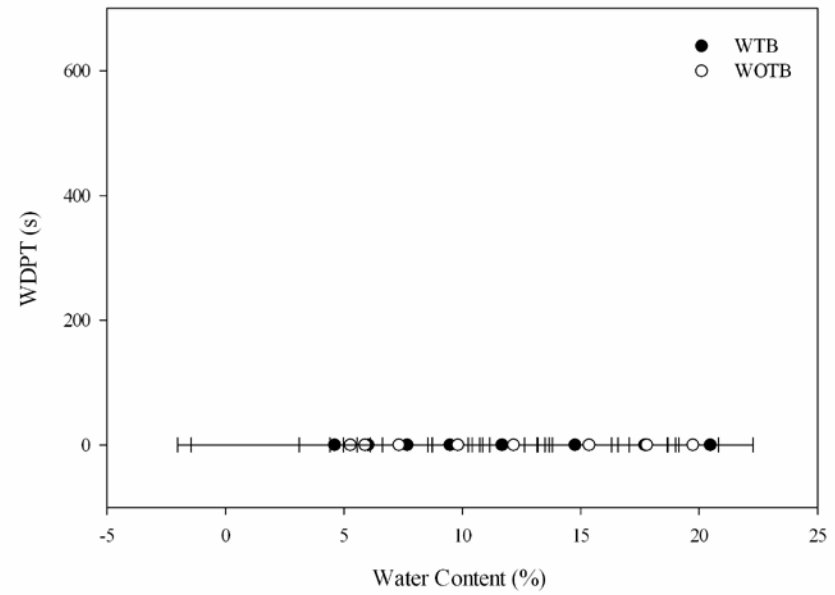
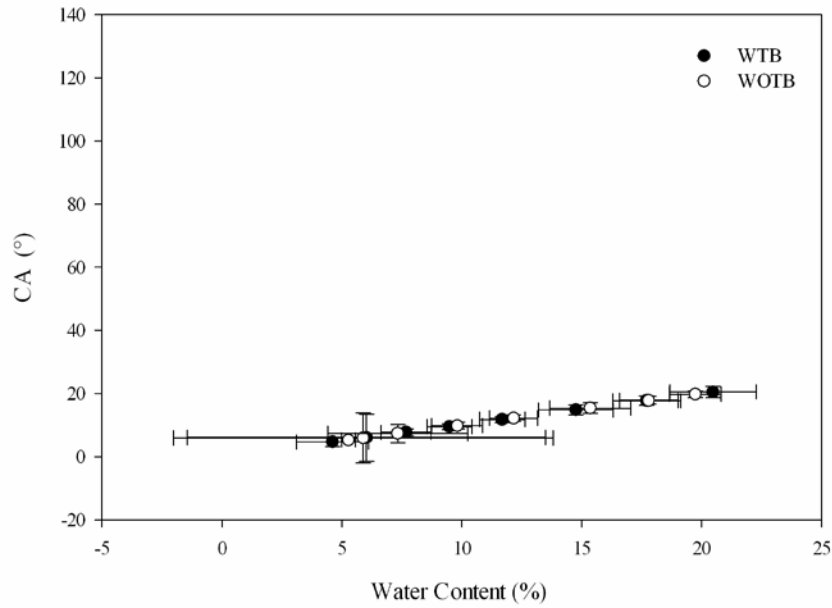


Figure 4.3. Relationship between soil water repellency and gravimetric water content based on contact angle (CA) and water droplet penetration time (WDPT) for coarse textured tarball affected reclamation materials with tarballs (WTB) and without tarballs (WOTB).

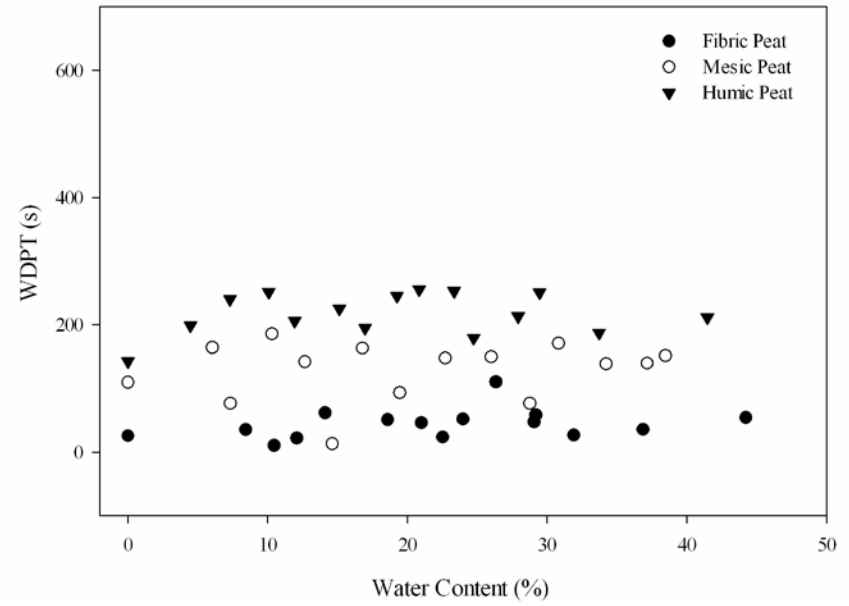
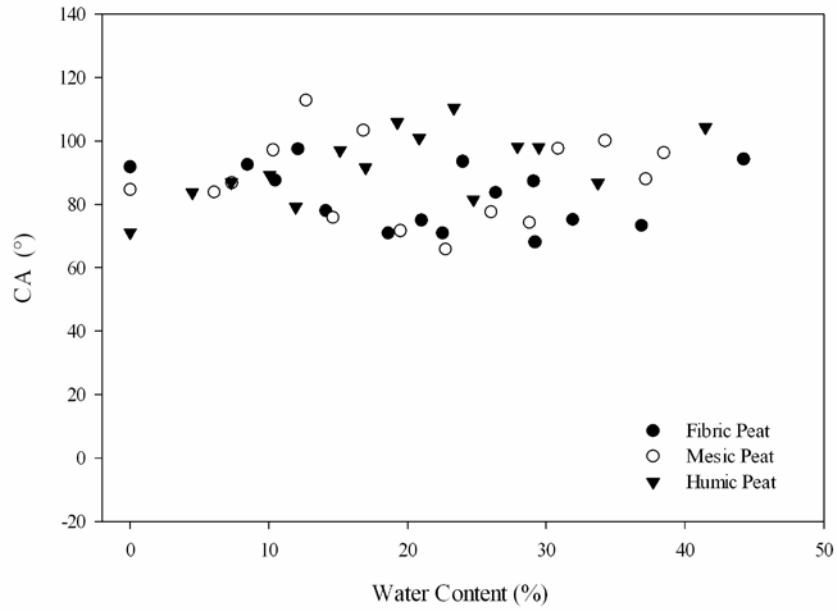


Figure 4.4. Relationship between soil water repellency and gravimetric water content based on contact angle (CA) and water droplet penetration time (WDPT) for fibric, mesic and humic peat.

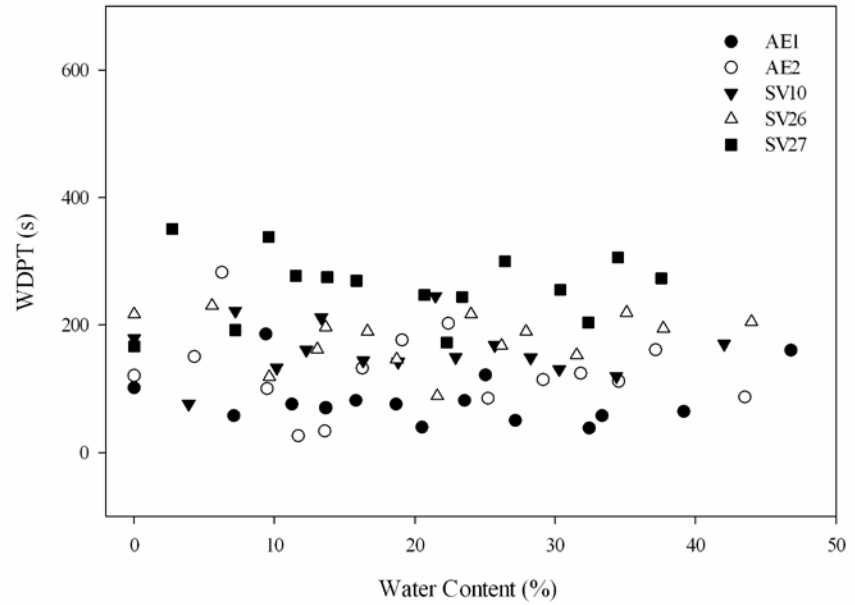
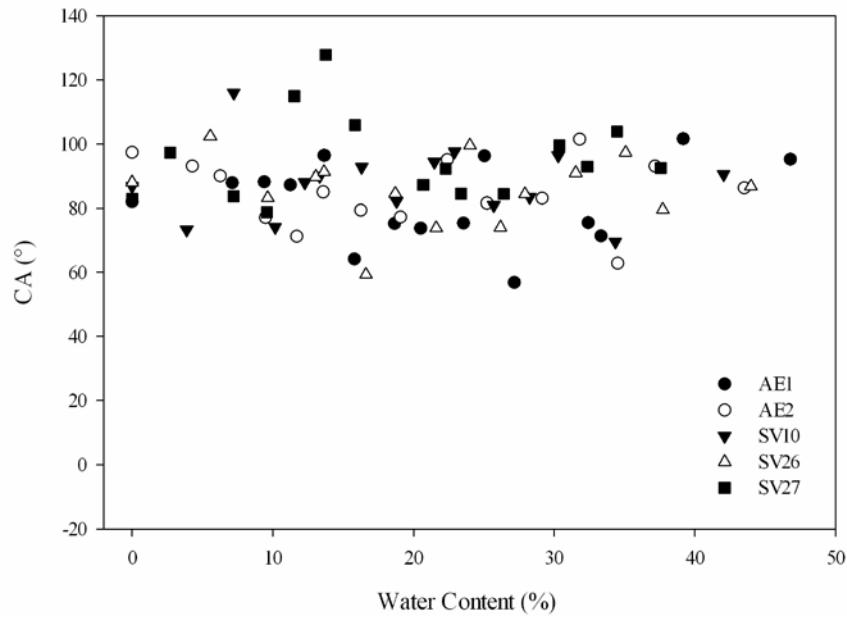


Figure 4.5. Relationship between soil water repellency and gravimetric water content based on contact angle (CA) and water droplet penetration time (WDPT) for LFH from A ecosite 1 (AE1), A ecosite 2 (AE2), soil vegetation plots 10, 26 and 27 (SV10, SV26 and SV27).

Table 4.5. Relationship between water content and soil water repellency for reclaimed soil, coarse textured tarball affected soil, peat and LFH as illustrated by average and r^2 contact angle (CA) and water droplet penetration time (WDPT) values.

Site	CA		WDPT	
	Average (°)	r^2	Average (s)	r^2
----- <i>Surface Soil From Reclaimed Sites</i> -----				
SS	52	0.92	36	0.15
CC	92	0.53	252	0.80
----- <i>Coarse Textured Tarball Affected Soil</i> -----				
WTB	12	1.00	0	N/A
WOTB	12	1.00	0	N/A
----- <i>Peat</i> -----				
Fibric	83	0.08	44	0.11
Mesic	88	0.00	128	0.03
Humic	92	0.08	217	0.11
----- <i>LFH</i> -----				
AE1	82	0.01	84	0.00
AE2	85	0.02	127	0.01
SV10	88	0.00	160	0.01
SV26	86	0.00	179	0.00
SV27	95	0.00	258	0.00

between wettable and water repellency (90° and 100-300 s). This suggests that these materials are not severely water repellency, but may inhibit water infiltration under the right circumstances. The combination of materials, structure and initial moisture content should be carefully considered for placement in reclamation.

Because humic organic matter has been clearly identified as an organic fraction that greatly contributes to soil water repellency (Roberts and Carbon, 1972; Chen and Schnitzer, 1978), an increase in water repellency with decomposition level was expected. The average WDPT was 44, 128 and 217 s for fibric, mesic, and humic peat, respectively (Table 4.5). The WDPT of peats of differing decomposition level are statistically different ($P < 0.001$).

4.4. Conclusion

Successful reclamation is dependent on careful consideration of many factors, including soil water repellency. Good water infiltration is essential to the development of new ecosystems from reclamation materials. The effects of soil water repellency in undisturbed and reclaimed sites were investigated. Furthermore, the critical water content of reclamation materials was examined.

These results showed that the mean and range of soil water repellency of reclaimed soils are similar to that of undisturbed sites in the AOSR. Therefore, current practices do not dramatically increase water repellency. Exposed soil surfaces are vulnerable to soil water repellency, leading to erosion and reduced infiltration. The situation is particularly hazardous when water repellent material is placed on a sloping surface. The conditions of soil water repellency should be continually monitored, and placement of cover materials should avoid use of water repellent materials on slopes.

Further investigation into the increased water repellency at the subsurface of SV10 and CC are required. A detailed analysis of the hydrocarbon content at depth for SV10 would provide some insight to whether the change in water repellency is due to the higher hydrocarbon content. The increased repellency at the subsurface of CC suggests that the placement of hot coke is detrimental to the success of the reclamation cover.

Overall, the relationship between water content and water repellency was weak. Mineral materials were generally wettable above 5-10 % gravimetric water content. Coarse textured tarball affected soil showed only subcritical water repellency. Peat and LFH showed a complete lack of relationship between water content and water repellency and both showed moderate water repellency at all water contents tested. Generally, mineral soils were the most water repellent materials tested, followed by peat and LFH, with coarse textured tarball affected materials being almost completely wettable.

Water repellency increased significantly with decomposition level of peat, supporting previous claims that humic organic fractions contribute to water repellency. Determination of CWC is quite challenging due to the strong variability in CA and WDPT. There is a need for

further investigation into the relationship of water content and water repellency of organic materials.

These results further confirm the data showing that water repellency occurs on a continuum. The concept of CWC is flawed and should be abandoned in preference of language such as water content dependent repellency.

5. SUMMARY AND CONCLUSIONS

The AOSR of northern Alberta, Canada contains the world's largest bitumen deposits, which are excavated from up to 100 m below the surface in open-pit mining operations. These sites are reclaimed using surface materials that are salvaged and stockpiled during mining. Soil water repellency is naturally occurring in the AOSR because of the dominance of organic materials and coarse textured soils. Exacerbating water repellency in reclamation materials could reduce the availability of water to developing plant communities and increase erosion.

A main cause of soil water repellency is organic matter that coats soil particles. These organics reorient themselves on soil particles with changes in soil water content. Generally, drier soil is more water repellent and wet soil is more wettable. The water content at which soil changes from being wettable to behaving as a water repellent soil is called the critical water content (CWC).

Water repellency has most commonly been studied as a result of forest fire or fungal colonization (as in the case of fairy rings in golf turfs). Studies have been conducted on soil water repellency associated with crude oil spills in Alberta, Canada indicating that organic carbon of petroleum origin may lead to soil water repellency. No similar studies have been conducted on undisturbed or reclaimed sites in the AOSR. Soil water repellency in the AOSR was studied in order to improve the methods available for monitoring soil water repellency, determine the current level of water repellency in reclaimed and undisturbed sites, examine the CWC of reclamation materials in order to minimize complications with severe water repellency and ultimately contribute to reclamation practices.

The mini tension infiltrometer was compared to the standard tension infiltrometer as a means of measuring RI. The mini infiltrometer is smaller, simpler, less expensive and less time consuming. As such, there are benefits to using it over the cumbersome, time consuming and expensive standard infiltrometer. There was strong variability in measured soil water repellency indices within and among sites. Although there were differences in measured RI, they were seldom statistically significant. When $RI \geq 1.95$ is used as the threshold between wettable and water repellent soil, the mini infiltrometer yielded results that were the same as, or more

cautionary than the standard infiltrometer. This suggests that the mini infiltrometer is well suited for *in situ* analysis of soil water repellency at the site level.

A field study compared of soil water repellency in reclaimed and undisturbed sites using RI, WDPT test and MED test. These studies showed no statistical difference between the soil water repellency of reclaimed and undisturbed sites. The variability of soil water repellency was high at all sites. These trends were consistent between RI, WDPT and MED results. Soil water repellency was most often higher at the soil surface than at the subsurface.

The relationship between water content and water repellency of mineral and organic reclamation materials was examined using the WDPT test and CA from images captured using a PG-X goniometer. There were few strong trends between water content and soil water repellency for the reclamation materials studied. Coarse textured tarball affected materials were only subcritically water repellent. Though there was not a strong relationship between soil water repellency and water content for peat and LFH, overall soil water repellency increased with decomposition level. Accurate analysis of CWC was further complicated by challenges with accurately quantifying the water content of test materials.

From these studies, the following conclusions and recommendations are made:

1. The mini tension infiltrometer is a suitable method for monitoring soil water repellency
2. Current reclamation practices do not dramatically increase soil water repellency, though it should continue to be monitored.
3. Soil water repellency is highly spatially variable. A large number of sampling points should be used in order to effectively monitor soil water repellency.
4. Wettable reclamation materials are suitable for use on highly exposed surfaces and steep slopes.
5. Water repellent materials are best used on protected, north facing slopes, on flat surfaces, at the subsurface or in mixes with highly wettable materials.
6. Mineral soils are generally wappable above gravimetric water content 5-10%.

7. Coarse textured tarball affected mineral materials are subcritically water repellent.
8. Peat and LFH showed no strong relationship between water repellency and water content and both straddled the threshold between wettable and water repellent.
9. Severity of water repellency of materials examined followed the following trend:

Mineral Soil > Peat \approx LFH > Coarse textured tarball affected materials
10. Organic content seems to have a greater effect on wettability than hydrocarbon content.
11. Water repellency increases with decomposition level of peat. More decomposed peat should be placed at the subsurface or on flat surfaces where it is less susceptible to drying and erosion. Less decomposed peat may be used for mixes on slopes.
12. Organic reclamation materials may be wettable, but are at risk of becoming water repellent in the right circumstances. Slope and structure are important considerations for these materials.
13. Further investigation is required to establish the relationship between water content and water repellency of organic materials.
14. The concept and even the term critical water content should be discarded in favor of terminology such as water content dependent repellency.
15. Heat from the coke under the coke cover capping study has increased the water repellency at its subsurface.

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APPENDIX A

**CRITICAL WATER CONTENT OF RECLAMATION MATERIALS WITH
WATER CONTENT CALCULATED BY SUBTRACTING THE BAG AND
DRY SAMPLE WEIGHT FROM THE TOTAL WEIGHT BEFORE
MEASURING SOIL WATER REPELLENCY**

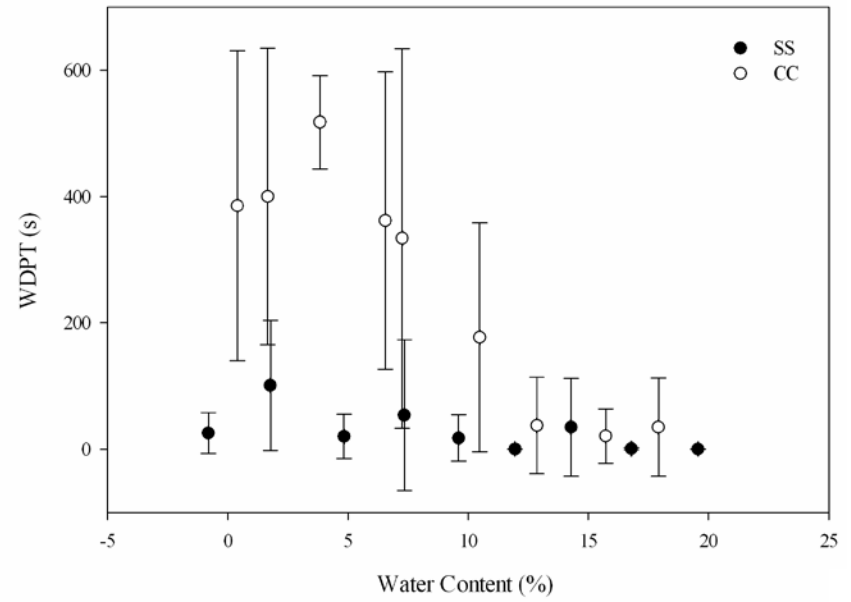
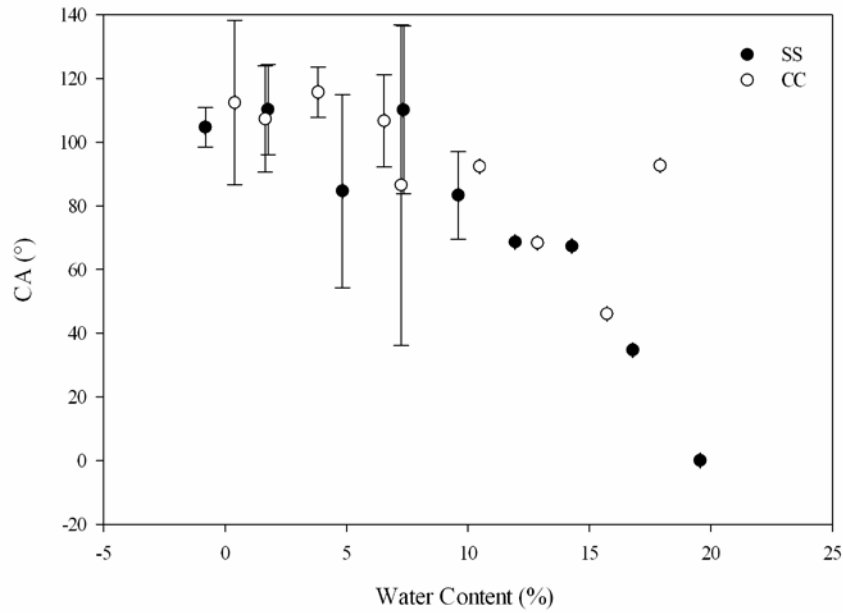


Figure A.0.1. Relationship between soil water repellency and gravimetric water content based on contact angle (CA) and water droplet penetration time (WDPT) for reclaimed mineral soil at the Shallow-Stripping trial (SS) and the Coke Cover Capping Study (CC) as calculated using the final sample weight before measuring soil water repellency.

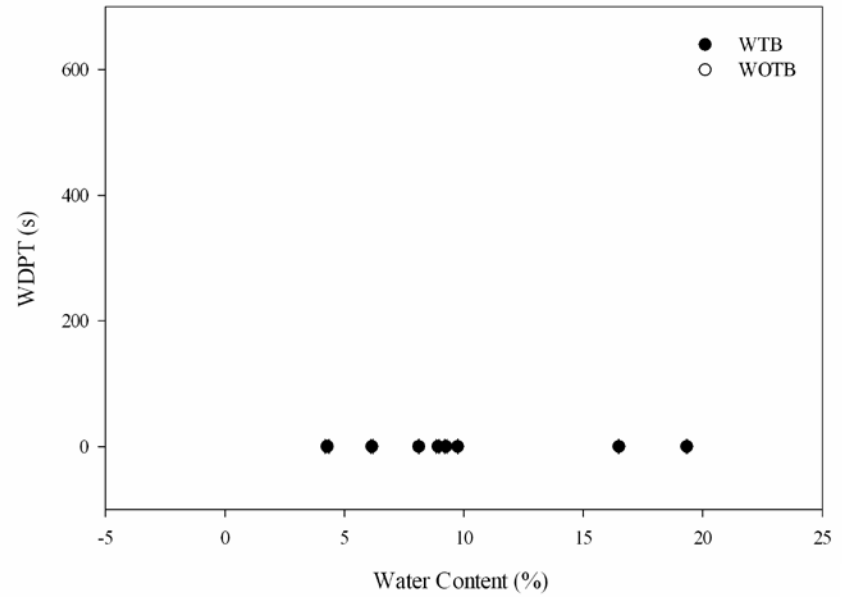
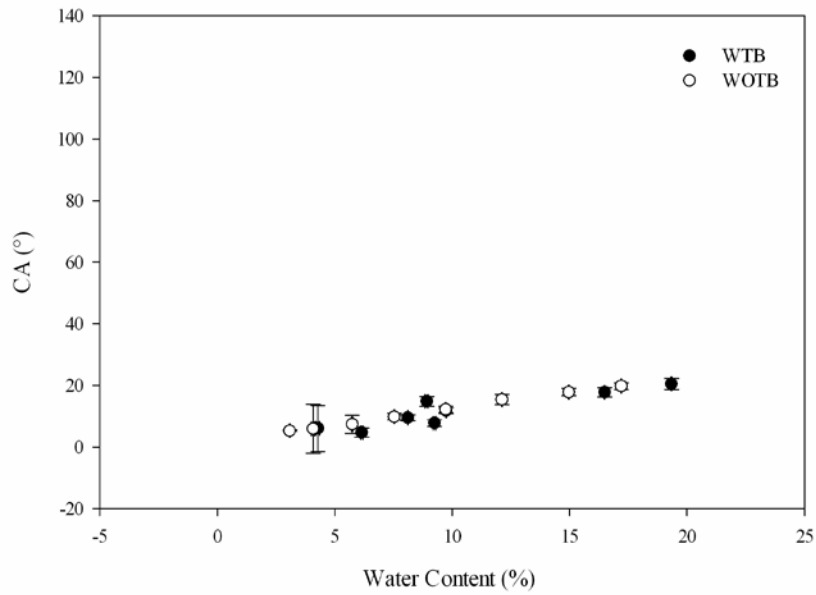
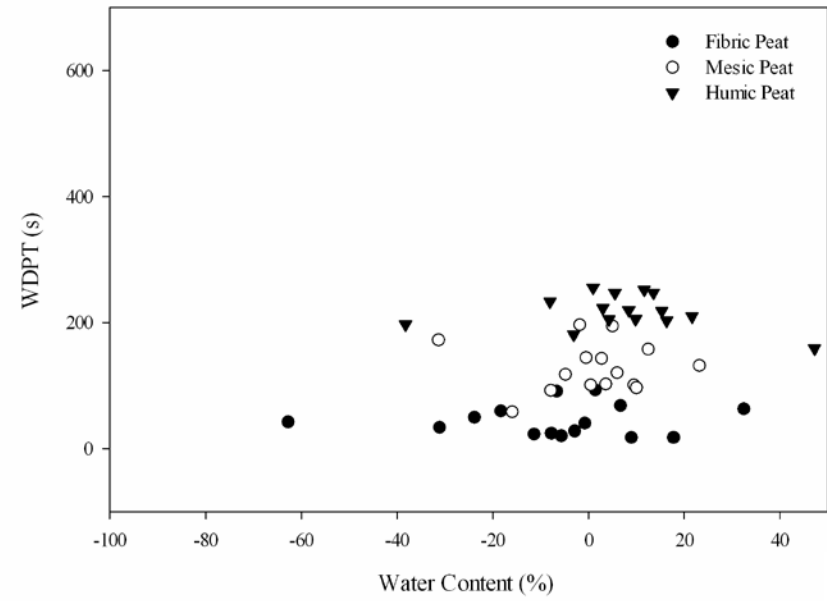
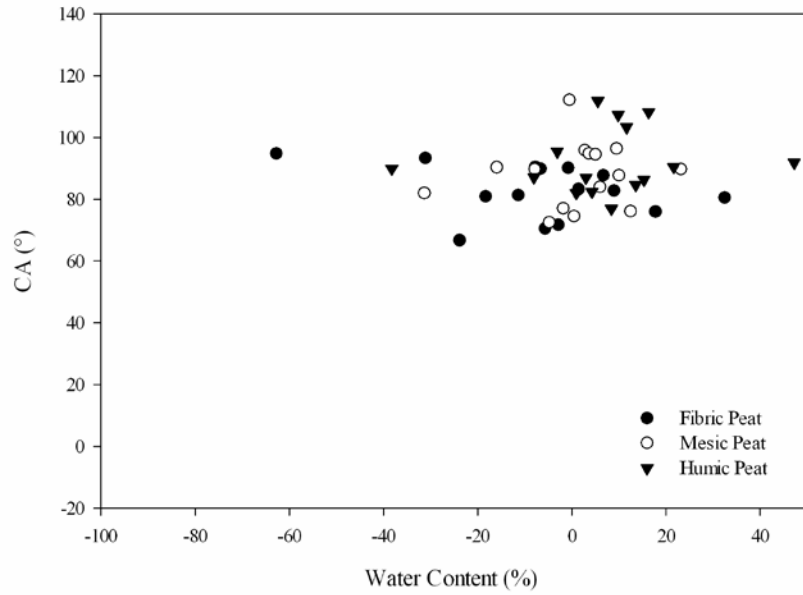
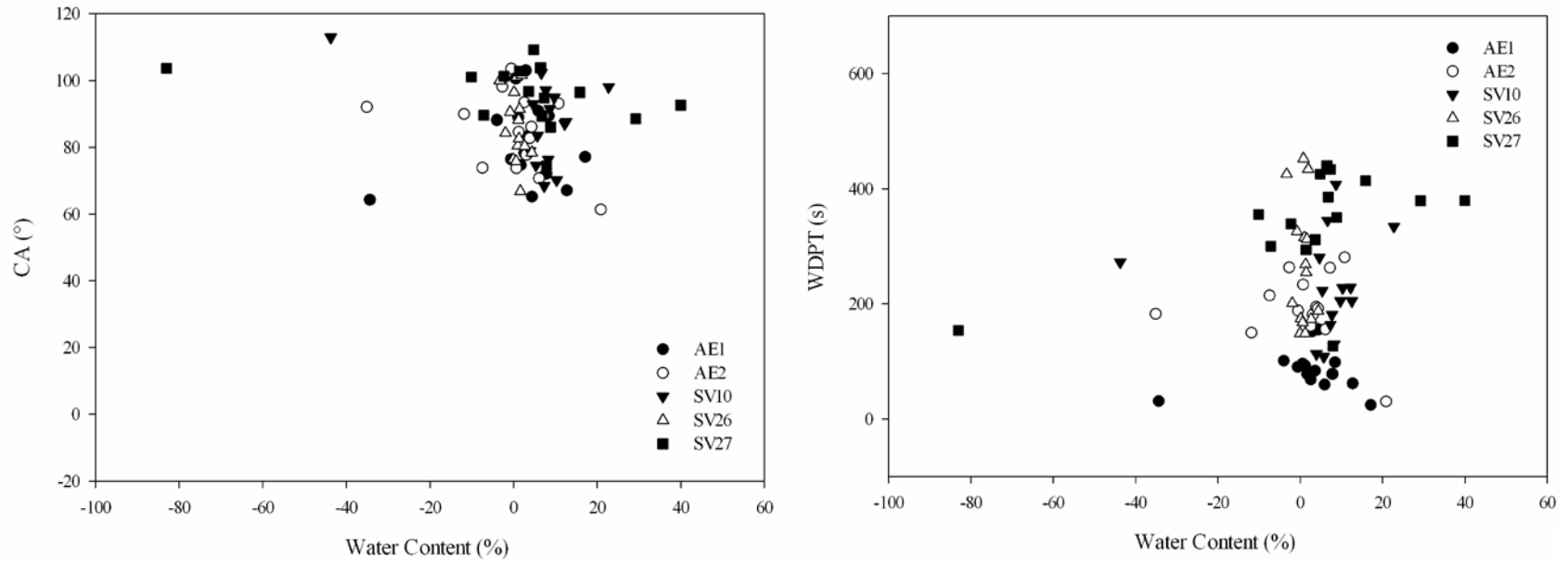


Figure A.0.2. Relationship between soil water repellency and gravimetric water content based on contact angle (CA) and water droplet penetration time (WDPT) for coarse textured tarball affected reclamation materials With Tarballs (WTB) and Without Tarballs (WOTB) as calculated using the final sample weight before measuring soil water repellency.



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Figure A.0.3. Relationship between soil water repellency and gravimetric water content based on contact angle (CA) and water droplet penetration time (WDPT) for peat as calculated using the final sample weight before measuring soil water repellency.



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Figure A.0.4. Relationship between soil water repellency and gravimetric water content based on contact angle (CA) and water droplet penetration time (WDPT) for LFH from A ecosite 1 (AE1), A ecosite 2 (AE2), Soil Vegetation Plot 10 (SV10), Soil Vegetation Plot 26 (SV26) and Soil Vegetation Plot 27 (SV27) as calculated using the final sample weight before measuring soil water repellency.

APPENDIX B

PARTICLE SIZE DISTRIBUTION OF MINERAL SOIL SAMPLES ANALYZED

Particle size distribution analysis was carried out on organic matter-free, dispersed samples. Samples were air dried and sieved to 2 mm then organic matter was removed using 30% hydrogen peroxide (Gee and Or, 2002) for 2008 surface samples and 6% sodium hypochlorite (common household bleach) (Mikutta et al., 2005) for 2008 subsoil and all 2009 samples. Samples prepared using the peroxide were dispersed using 150% calgon solution, while those treated with bleach required no further dispersion (Mikutta et al., 2005). Different methods were used for particle size analysis because information about the efficacy of bleach in removing organic matter was located after the initial analysis was completed. The bleach method was chosen for economic reasons. In both cases, samples were dried down to a thick paste and analyzed using a Horiba LA-950 Laser Scattering Particle Size Distribution Analyzer as per the laser diffraction method outlined by Eshel et al. (2004). The instrument measures particles 0.011 – 3000 μm in diameter. Particle size factions and texture classes were assigned according to the Canadian System of Soil Classification (Soil Classification Working Group, 1998).

Table B.0.1. Particle size distribution of soil samples.

Site		sand	silt	clay	soil texture
<i>undisturbed sites</i>					
AE1	surface	87.8	11.6	0.6	Loamy Sand
	subsurface	76.9	23.1	0.0	Sandy Clay Loam
AE2	surface	86.9	9.0	4.1	Loamy Sand
	subsurface	89.5	9.3	1.2	Sand
SV10	surface	95.9	3.4	0.8	Sand
	subsurface	83.4	14.2	2.4	Loamy Sand
SV26	surface	93.5	4.4	2.1	Sand
	subsurface	91.3	7.6	1.0	Sand
SV27	surface	90.3	6.7	3.0	Sand
<i>reclaimed sites</i>					
SS	surface	60.2	32.8	7.0	Sandy Loam
	subsurface	59.9	29.9	10.1	Sandy Loam
CC	surface	56.4	21.8	21.7	Sandy Clay Loam
	subsurface	55.1	31.9	13.0	Sandy Loam
ALFH	surface	88.0	10.1	1.9	Sand
	subsurface	94.8	4.9	0.3	Sand
SW30	surface	5.5	29.2	65.4	Heavy Clay
	subsurface	23.6	40.2	36.2	Clay Loam
WTB	surface	97.1	2.9	0.0	Sand
WOTB	surface	97.9	1.8	0.2	Sand
<i>grassland site in Central Saskatchewan</i>					
SD	surface	40.8	34.6	24.6	Loam

APPENDIX C

TOTAL PETROLEUM HYDROCARBON CONCENTRATION OF RECLAIMED MINERAL SOIL, LFH AND PEAT

Samples were tested for their hydrocarbon content by Bodycote Testing Group (now Exova) (Calgary, AB, CAN) according to the Canadian Council of Ministers of the Environment (CCME) guidelines for petroleum hydrocarbons in soil (CCME, 2001). F2 through F4+ fractions were identified using High Temperature Gas Chromatography with Flame Ionization Detection, while the F4G fraction was determined gravimetrically. Values reported are an average of the measures from each of the five points. For values reported as under the detection limit, the detection limit was used in the calculation of the mean.

Table C.0.1. Hydrocarbon analysis of reclaimed mineral soil, LFH and peat.

Sample	F2 C10-C16 (mg kg ⁻¹)	F3 C16-C34 (mg kg ⁻¹)	F4 C34-C50 (mg kg ⁻¹)	F4HTGC C34-C50+ (mg kg ⁻¹)	%C50+ (%)	F4G (mg kg ⁻¹)
----- <i>Reclaimed Mineral Soil</i> -----						
SS	20	187.4	144.4	375.6	35.9	2216
CC	20	424.4	344.6	1013.6	44.2	4454
ALFH	20	20.8	30	30	5	966.4
----- <i>LFH</i> -----						
AE1	28	1732	1235	2562	28.02	25220
AE2	21.6	911.2	1213	2920	43.56	36960
SV10	40.6	1559.4	1062.4	3576	49.04	29880
SV26	30.4	645.8	834.4	1534	30.8	32560
SV27	37.6	688.6	538.4	1202.6	35.62	38580
----- <i>Peat</i> -----						
Fibric	26.4	597.4	230.8	1226.4	55	20680
Mesic	27.8	482.0	188	1173.6	57.5	22260
Humic	37.2	425.4	216	912.8	53.82	9973.8