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Developing a universal equation to estimate the mass of dewatered wastewater sludge during biological digestion at mesophilic and thermophilic temperatures

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ABSTRACT

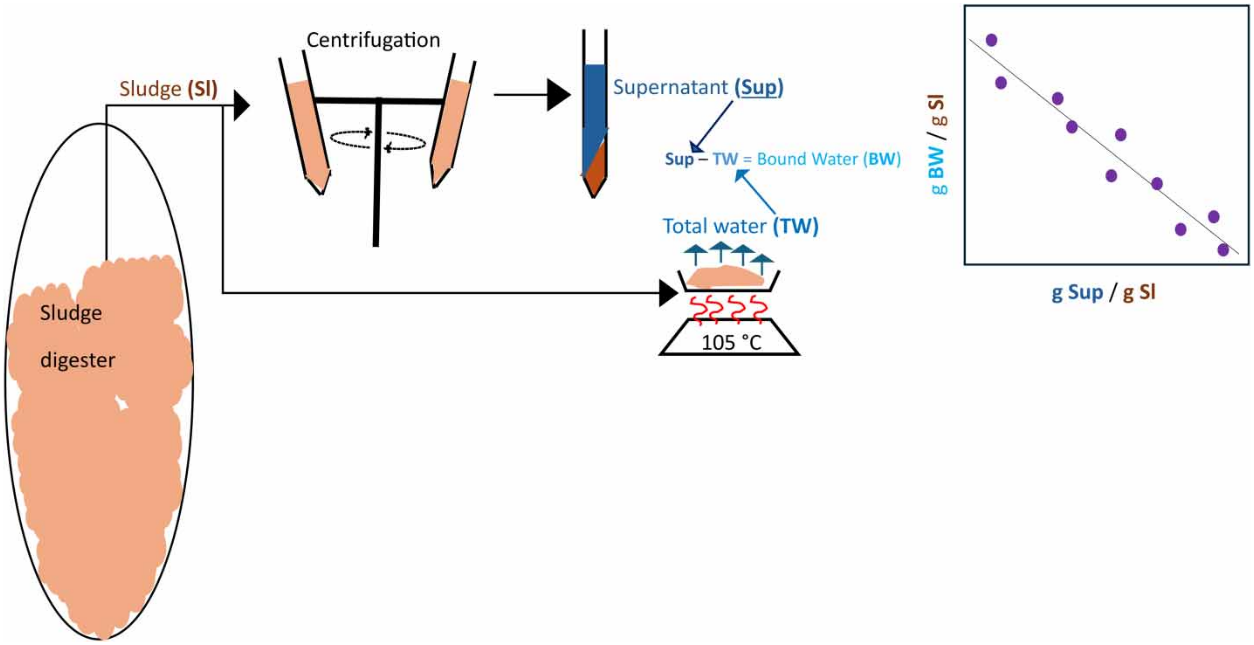
A series of dewaterability tests were conducted on various types of sludges to establish a wholistic relationship between sludge water fractions. Sludge samples were obtained from batch and continuous sludge digesters, which were operated anaerobically and aerobically under mesophilic and thermophilic conditions. Dewaterability of the sludge samples and the distribution of water fractions were studied using centrifugation and thermal drying. Thickened waste activated sludge (T-WAS) contained 10-11 g bound water (BW)/g of total solids (TS), and it was more hydrophilic than primary and digested sludges. During anaerobic digestion, BW content fluctuated between 3.2 and 4.2 g BW/g TS. However, aerobic digestion at 55°C reduced the BW content of the mixed T-WAS + primary sludges from 3.7 to 2.1 g BW/g TS. A linear function was developed to correlate supernatant and BW mass fractions ($R^2 = 0.995$). An equation was derived from the linear function to estimate the mass of dewatered sludge based on the TS concentration of the initial wet sludge. The developed expression is applicable to different kinds of wastewater sludges. Such an expression would be helpful for the designers and operators of sludge thickening and dewatering systems that use centrifugal separation.

Key words: bound water, dewaterability, digestion, sludge, supernatant, temperature

HIGHLIGHTS

- Anaerobic and aerobic digestions affected centrifugal dewatering differently.
- Temperature during aerobic digestion impacted bound water content of sludge.
- Thickened waste activated sludge was more hydrophilic than primary sludge and digested sludge.
- A linear model estimated the supernatant mass fraction of various sludges.
- There was a linear correlation between model parameters.

GRAPHICAL ABSTRACT



1. INTRODUCTION

Wastewater treatment is a necessity for today's societies. In wastewater treatment plants (WWTPs), a combination of physical, chemical, and biological processes are applied to produce an effluent that incurs minimum harm to the environment. A by-product of these processes is wastewater sludge, also known as biosolids. In conventional WWTPs, primary and secondary sludge production rates per capita have been reported to be 0.6–2.2 and 2.5–6 L/d, respectively (Goncalves *et al.* 2007). Handling the produced sludge constitutes a substantial portion of wastewater treatment expenditure in activated sludge systems (Gray 2004). Due to its concentrated nature, sludge has a high contamination potential, necessitating proper treatment before disposal. Biological digestion of sludge is commonly used, and it can be carried out aerobically or anaerobically.

An important consideration in the design and operation of biological digestion processes is the volume of sludge that needs to be treated. Sludge volume determines the volume of digester at a selected operating temperature (i.e., mesophilic, thermophilic). The volume of the sludge digester is important because the heat energy required for operation of the digester depends on its volume. Water is responsible for up to 99% of undigested wastewater sludge (Tchobanoglous *et al.* 2014). Therefore, reducing the water content of undigested sludge (i.e., thickening) lowers the volume of the sludge digester and the required energy for heating. Apart from that, about 95% of digested sludge (DS) is water (Tchobanoglous *et al.* 2014), which makes sludge transport and disposal expensive and difficult. Hence, reducing the DS water content (i.e., dewatering) further reduces the sludge management expenditures. Centrifugation is a method of reducing the water content of sludges (Chu & Lee 2002; Ginisty *et al.* 2021; Novak 2006).

Approximating the quantity of sludge left after the water reduction is essential for a realistic design of sludge treatment reactors and planning a cost-effective sludge transport system. In addition, the water removed from the initial wet sludge during sludge dewatering has a high load of contaminants that need to be removed. The common practice is to return it to the headworks of the treatment plant (Gray 2004). Plant designers and operators should consider the return as it would increase carbon and nutrient loading in the upstream operation and process units and impacts the hydraulics and the capacity of the of the WWTP. Therefore, estimation of the supernatant (i.e., physically separable sludge water) flow rate is of importance as well.

Investigations have been carried out to model centrifugation performance for sludge dewatering. Vesilind & Zhang (1984) developed a first-order model to predict the concentration of suspended solids in the pellet created by centrifugation. Computational and experimental works were conducted focusing on torque calculations of solid-bowl centrifuge (Leung 1998). Chu & Lee (2002) developed equations for the calculation of specific resistances. Stickland *et al.* (2006) performed detailed mathematical modeling of one-dimensional solid-bowl batch centrifugation. In addition, computer modeling by means of artificial neural networks has been performed to predict sludge dewatering efficiency (Kowalczyk & Kamizela 2021). These models are rather complicated and often require parameters that cannot be readily measured or computed by the designers and operators of sludge treatment systems. Furthermore, there are not enough systematic studies based on the variations of the water content of different kinds of wastewater sludge during biological digestion at mesophilic and thermophilic temperatures. The aim of the present study was to develop an easy-to-use yet universal expression for estimating water and thus solids fractions of wastewater sludges. It was also attempted to reveal the implications of the type and temperature of the biological digestion on the changes of sludge water fractions. Findings of this investigation would be useful for the designers and operators of sludge handling facilities that employ biological digestion and centrifugal separation.

2. MATERIALS AND METHODS

2.1. Wastewater treatment plant

Primary sludge (PS), thickened waste activated sludge (T-WAS), and DS were sampled from a WWTP that is located in Ottawa, Canada. The WWTP employs conventional activated sludge with chemical phosphorus removal for municipal wastewater treatment. The chemicals used for phosphorus removal are ferrous chloride, ferric chloride, and alum ferrous blend (Robidoux 2018), which are added to the activated sludge recycled into the aeration tank. Also, the WWTP has a flow-through mesophilic anaerobic digester. The PS was settled in primary clarifiers, and T-WAS was produced by centrifugal thickening of the waste activated sludge created in secondary clarifiers. Both sludges were fed into, mixed, and digested in a full-scale anaerobic digester. The volumetric ratio of PS to T-WAS was roughly 4.5. Solids retention time of the reactor was about 30 days. PS and T-WAS were fed into the reactor via two separate pipes. The average volumetric flowrate through the full-scale digester was 22 L/s. Further details of the treatment process can be found at https://documents.ottawa.ca/sites/documents/files/wastewater_treatment_en.pdf.

The volumes of the sludge samples taken from the WWTP were different depending on the purpose of sampling. Small sample volumes up to 1 L were cooled by ice packs right away after sampling because they were used for measuring sensitive parameters including solids content and pH. Samples of large sample volumes (10–30 L) were considered for laboratory digestion experiments. In less than 3 h, they were transferred to the laboratory and refrigerated. Influent sludge was prepared in the laboratory by mixing PS and T-WAS with the same ratio as they were mixed in the full-scale anaerobic digester of the WWTP. Simultaneous sampling of the influent sludge components and the digestate from the WWTP was carried out twice: first time in March and second time in July. The characteristics of the raw sludge mixture (influent) are shown in Table S1 in the Supplementary material. Apart from that, several more samples of PS, T-WAS, and DS were taken from the WWTP on different occasions.

2.2. Laboratory-scale digesters

Three batch anaerobic reactors were made of 10 L (working volume) Nalgene containers and operated at 35, 45, and 55 °C. Hereafter, the three anaerobic digesters are called AnD.35, AnD.45, and AnD.55 (Figure 1(a)). The required temperatures were provided and maintained using warmer blankets and a temperature control system. With the addition of an aeration system and a condenser, the same reactors were modified to make them suitable for aerobic digestion (Figure 1(b)). Hereafter, the three aerobic digesters are called AD.35, AD.45, and AD.55. The three mentioned temperatures were selected to simulate mesophilic and thermophilic conditions. Further details about building, operating, and sampling of the batch laboratory reactors used in the present research have been described elsewhere (Poorasgari & Örmeci 2022).

2.3. Measurement of TS and FS

Total (TS) and fixed solids (FS) concentrations were determined by thermal method (Poorasgari & Örmeci 2022; Standard Methods 2022), and all the measurements were triplicated. Volatile solids (VS) concentration was calculated from the difference between the two parameters. In this paper, the measured values of all types of solids shown in square brackets denote concentration in g/kg. TS mass fraction ($f_{TS/Sl}$) was calculated by dividing [TS] by 1,000, where f denotes mass fraction and the index Sl denotes sludge, and this applies to all other relevant parameters of this article. The total water (TW) mass fraction ($f_{TW/Sl}$) of sludge was calculated by $1 - f_{TS/Sl}$.

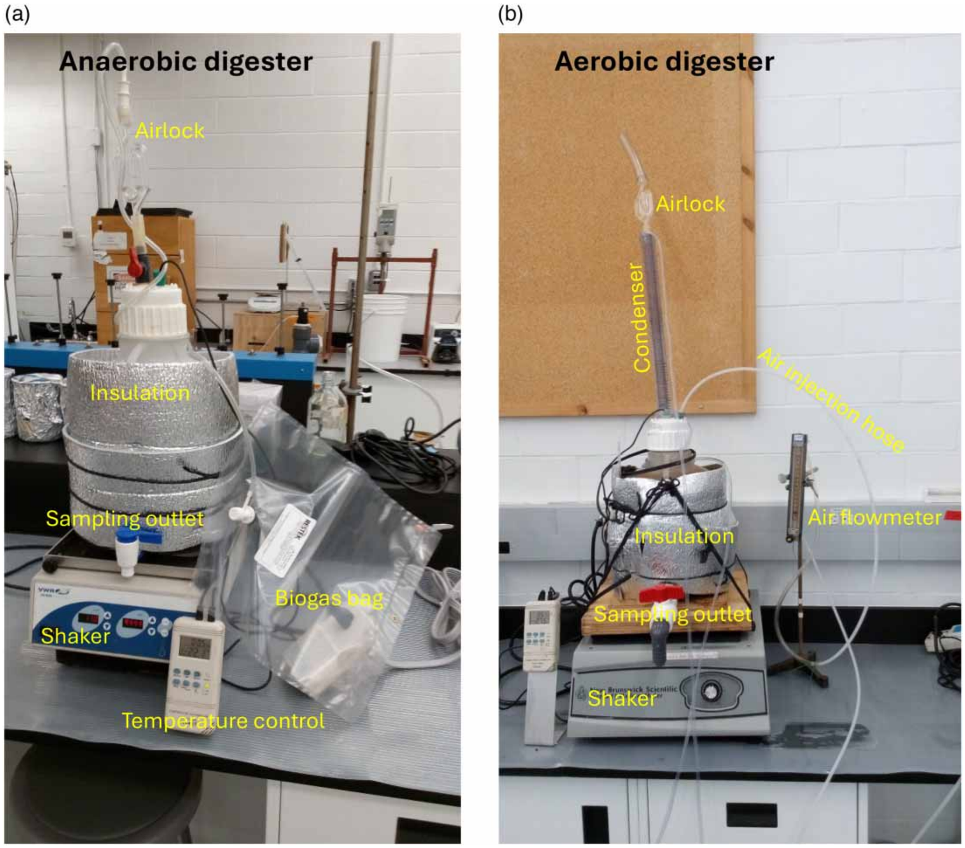


Figure 1 | Laboratory-scale digesters.

2.4. Measurement of sludge supernatant

A fixed-rotor Sorvall Legend RT plus centrifuge was employed to separate the supernatant from the initial wet sludge samples. The rotor was FIBERLite with six wells angled at 67°/23° (Thermo Fisher Scientific). Sludge samples of known masses were centrifuged at several centrifugal accelerations ($C_a =$ [angular velocity$^2 \times$ radius]/9.81) and for several centrifugation times (C_t). The details are given in Table S2.

The temperature during all centrifugations was 4 °C. Note that DS in this text does not include sludge samples obtained from laboratory reactors. Mixed sludges consisted of PS, T-WAS, and DS. Superscript 1 refers to the mixed sludge composition 45% PS + 45% T-WAS + 10% DS and superscript 2 refers to the mixed sludge composition 73% PS + 17% T-WAS + 10% DS (Table S2). The plus sign in the centrifugation time of the means centrifugation was done for two equal durations consecutively. The second centrifugation was carried out on the supernatant taken from the first centrifugation to further remove particles remaining in the first supernatant and reduce the filtration time afterward.

Mass of the supernatant was measured after each centrifugation. Supernatant mass fraction ($f_{\mathrm{Sup/Sl}}$) was calculated by dividing the mass of the supernatant by the initial mass of wet sludge before centrifugation. Measurements were at least duplicated except for the first sample taken from AD.35. The supernatant was then filtered through membranes with a pore diameter of 0.45 μm. The filtrate was used to measure the concentration of the dissolved solids. The work for acquiring the filtrate was carried out on the same day as sludge sampling, and the obtained filtrate was stored at 1–4 °C until further analyses.

2.5. Measurement of dissolved solids

Total dissolved solids (TDS) concentration and fixed dissolved solids (FDS) concentrations of the filtrate were measured by the thermal method (Poorasgari & Örmeci 2022; Standard methods 2022). The measurements were at least duplicated. Volatile dissolved solids (VDS) of the liquid phase was calculated by subtracting [FDS] from [TDS]. $[\mathrm{TDS}]_{\mathrm{Sl}}$, $[\mathrm{VDS}]_{\mathrm{Sl}}$, and $[\mathrm{FDS}]_{\mathrm{Sl}}$

were calculated by multiplying the corresponding filtrate concentrations by $f_{Sup/Sl}$. The sludge concentrations of the dissolved solids were then used to compute concentration of the total, volatile, and fixed suspended solids as follows: [TSS] = [TS] – $[TDS]_{Sl}$, [VSS] = [VS] – $[VDS]_{Sl}$, [FSS] = [FS] – $[FDS]_{Sl}$. Note that average values were used for these calculations.

2.6. Performance parameters of sludge digesters

Oxidation–reduction potential (ORP), dissolved oxygen (DO), and pH were measured using probes Intellical™ ORP-Redox MTC101 (Hach), RDO ORION 087003 (Thermo Scientific), and ORION 9157B (Thermo Scientific), respectively. The probes were calibrated before the start and during the digestion experiments according to manufacturers' instructions. The measurements were carried out immediately after sampling for sludge samples taken from laboratory reactors, and in less than 3 h for samples taken from the WWTP. Some ORP, pH, and DO measurements were repeated to check the precision and accuracy. Biogas production in laboratory anaerobic digesters was monitored by biogas collection bags attached to the exhausts of the batch anaerobic digesters.

2.7. Statistical analysis

To determine the statistical significance between groups of experimental observations, *t*-tests were conducted using Excel. For the statistical analyses, the confidence level was set at 0.95.

2.8. Theory

The supernatant is composed of a fraction of TW plus some solids, consisting of TDS and TSS remaining in the supernatant after the centrifugation. These explanations can be put in mathematical terms as follows:

$$m_{\mathrm{Sup}} = m_{\mathrm{Sup},W} + m_{\mathrm{Sup},S} \tag{1}$$

$$m_{\mathrm{Sup},S} = m_{\mathrm{Sup},TDS} + m_{\mathrm{Sup,TSS}} \tag{2}$$

$$m_{\mathrm{Sup,TDS}} = \frac{m_{\mathrm{Sup,TDS}}}{m_{\mathrm{Sup}}} \times m_{\mathrm{Sup}} = f_{\mathrm{TDS/Sup}} \times m_{\mathrm{Sup}} \tag{3}$$

$$m_{\mathrm{Sup,SS}} = \frac{m_{\mathrm{Sup,SS}}}{m_{\mathrm{Sup}}} \times m_{\mathrm{Sup}} = f_{\mathrm{TSS/Sup}} \times m_{\mathrm{Sup}} \tag{4}$$

$$m_{\mathrm{Sup},S} = m_{\mathrm{Sup}} \times (f_{\mathrm{TDS/Sup}} + f_{\mathrm{TSS/Sup}}) \tag{5}$$

where m_{Sup} is the mass of supernatant, $m_{\mathrm{Sup},W}$ is the mass of supernatant water, $m_{\mathrm{Sup},S}$ is the mass of supernatant solids, $m_{\mathrm{Sup,TDS}}$ is the mass of supernatant TDS, $m_{\mathrm{Sup,TSS}}$ is the mass of supernatant suspended solids, $f_{\mathrm{TDS/Sup}}$ is the mass fraction of TDS in supernatant, and $f_{\mathrm{TSS/Sup}}$ is the mass fraction of suspended solids in the supernatant.

The concentration of suspended solids left in the supernatant was assumed to be negligible. In addition, measurements throughout the current research showed that dissolved solids concentration in the supernatant was below 8 g/kg of supernatant (less than 1%). In other words, summation of $f_{\mathrm{TDS/Sup}}$ and $f_{\mathrm{TSS/Sup}}$ would be a very small value. That means $m_{\mathrm{Sup},W} \gg m_{\mathrm{Sup},S}$. Hence,

$$m_{\mathrm{Sup},W} + m_{\mathrm{Sup},S} \approx m_{\mathrm{Sup},W} \approx m_{\mathrm{Sup}} \tag{6}$$

Based on Equation (6), $m_{\mathrm{Sup}}/m_{\mathrm{Sl}}$ or $f_{\mathrm{Sup/Sl}}$ is almost equal to $m_{\mathrm{Sup},W}/m_{\mathrm{Sl}}$ or $f_{\mathrm{Sup},W/\mathrm{Sl}}$. Accordingly, the difference between TW and supernatant water mass fractions, denoted by $f_{(\mathrm{TW-Sup})/\mathrm{Sl}}$ can be formulated as follows:

$$f_{(\mathrm{TW-Sup})/\mathrm{Sl}} = f_{\mathrm{TW/Sl}} - f_{\mathrm{Sup/Sl}} \tag{7}$$

$f_{(\mathrm{TW-Sup})/\mathrm{Sl}}$ represents part of sludge water that does not come off by centrifugation. $f_{(\mathrm{TW-Sup})/\mathrm{Sl}}$ and $f_{\mathrm{Sup/Sl}}$ can be considered fractions of TW of sludge, as described by Equations (8) and (9).

$$f_{(\mathrm{TW-Sup})/\mathrm{Sl}} = f_{(\mathrm{TW-Sup})/\mathrm{TW}} \cdot f_{\mathrm{TW/Sl}} \tag{8}$$

$$f_{\mathrm{Sup/Sl}} = f_{\mathrm{Sup/TW}} \cdot f_{\mathrm{TW/Sl}} \tag{9}$$

Rearranging Equations (8) and (9) to solve for $f_{TW/Sl}$ and equating the two sub-sequent equations yields Equation (10), which can be rewritten as Equation (11).

$$f_{(TW-Sup)/Sl} = \frac{f_{(TW-Sup)/TW}}{f_{Sup/TW}} \cdot f_{Sup/Sl} \quad (10)$$

$$f_{(TW-Sup)/Sl} = \frac{m_{(TW-Sup)}}{m_{Sup}} \cdot f_{Sup/Sl} \quad (11)$$

$m_{(TW-Sup)}/m_{Sup}$ is mass of the water unseparable by centrifugation divided by the mass of supernatant. The sludge samples taken during digestion are expected to have different water contents and water fraction distributions. Therefore, it is hypothesized that this mass ratio is not the same for various types of sludges, such as those sampled over the course of a biological digestion process. A Y-intercept may be required to fit Equation (11) to experimental data. Accordingly, Equation (12) is proposed to estimate $f_{(TW-Sup)/Sl}$ as a function of $f_{Sup/Sl}$.

$$f_{(TW-Sup)/Sl} = K_{(TW-Sup)/Sup} \cdot f^{n}_{Sup/Sl} + \beta \quad (12)$$

where $K_{(TW-Sup)/Sup}$ is a proportionality factor and β is the Y-intercept (i.e., offset) of the model. In other words, β is $f_{(TW-Sup)/Sl}$ when $K_{(TW-Sup)/Sup} \cdot f_{Sup/Sl}{}^{n}$ equals 0. Units of $K_{(TW-Sup)/Sup}$, $f_{Sup/Sl}$, and β are (g TW − Sup)·(g Sup)$^{-n}$·(g Sl)$^{-1+n}$, (g Sup)^{n}·(g Sl)$^{-n}$, and (g TW − Sup)·(g Sl)$^{-1}$, respectively. The exponent n can be determined by fitting Equation (12) with experimental data. By equating the right-hand sides of Equations (11) and (12), it is found out that $m_{(TW-Sup)}/m_{Sup}$ is related to the proportionality factor via Equation (13).

$$\frac{m_{(TW-Sup)}}{m_{Sup}} = (K_{(TW-Sup)/Sup} \cdot f^{n-1}_{Sup/Sl}) + \frac{\beta}{f_{Sup/Sl}} \quad (13)$$

3. RESULTS AND DISCUSSION

3.1. General sludge characteristics and supernatant mass fractions

Variations in solids concentrations, pH, and DO of batch reactors during anaerobic and aerobic digestions were shown elsewhere (Poorasgari & Örmeci 2022). Variations in solids concentrations during inoculum preparation for anaerobic digestion are shown in Figure 2(a)–2(c). The inoculum for the 35 °C reactor was repeated due to the technical issues that occurred during the first round of inoculum preparation at that temperature. The values shown on the figure are those that were obtained during the second round of inoculum preparation at 35 °C. The reason for relatively large error bars in Figure 2(b) and (c) could be measurement errors. Variations in the ORP levels of the batch digesters are depicted in Figure 2(d) and (e). There was a reduction of ORP with temperature. During anaerobic digestion, there is an electron flow to part of the carbon content of the system, converting it to CH_4, which leaves the system. Other reductive reactions, such as H_2 formation, occur as well. Part of produced hydrogen may also leave the system. Thus, there is a negative electron flow in the system during anaerobic digestion, which explains ORP reduction. The overall reaction rate is dependent on the operating temperature, with faster reaction (faster electron transfer) at higher temperatures. This might explain lower ORP at higher operating temperatures. During aerobic digestion, more oxidized compounds are produced and the species that is reduced (oxygen) typically does not leave the system as much as CH_4 does during anaerobic digestion. However, due to the partial pressure changes and complex chemistry, it is likely that a large part of CO_2 that carries a significant number of electrons moves to the headspace and exhausts. This can explain descending trends observed during aerobic digestion. Such trends may be intensified with increasing temperatures.

A summary of the observations of biogas production by batch anaerobic digesters are tabulated in Table S3. The full-scale anaerobic digester removed about 65% of the incoming VS. pH levels were about 6.3 and 7.5 in the influent and digestate of the full-scale reactor, respectively. The solids concentrations of sludge samples taken from the full-scale reactor are shown in Tables S4 and S5. The supernatant mass fractions of sludge samples taken from the full-scale anaerobic digester are shown in Table S6. The supernatant mass fractions of sludge samples taken from batch anaerobic digesters are shown on Figure 3(a)–3(d). The supernatant mass fractions of sludge samples taken from batch aerobic digesters are shown in Figure 3(e).

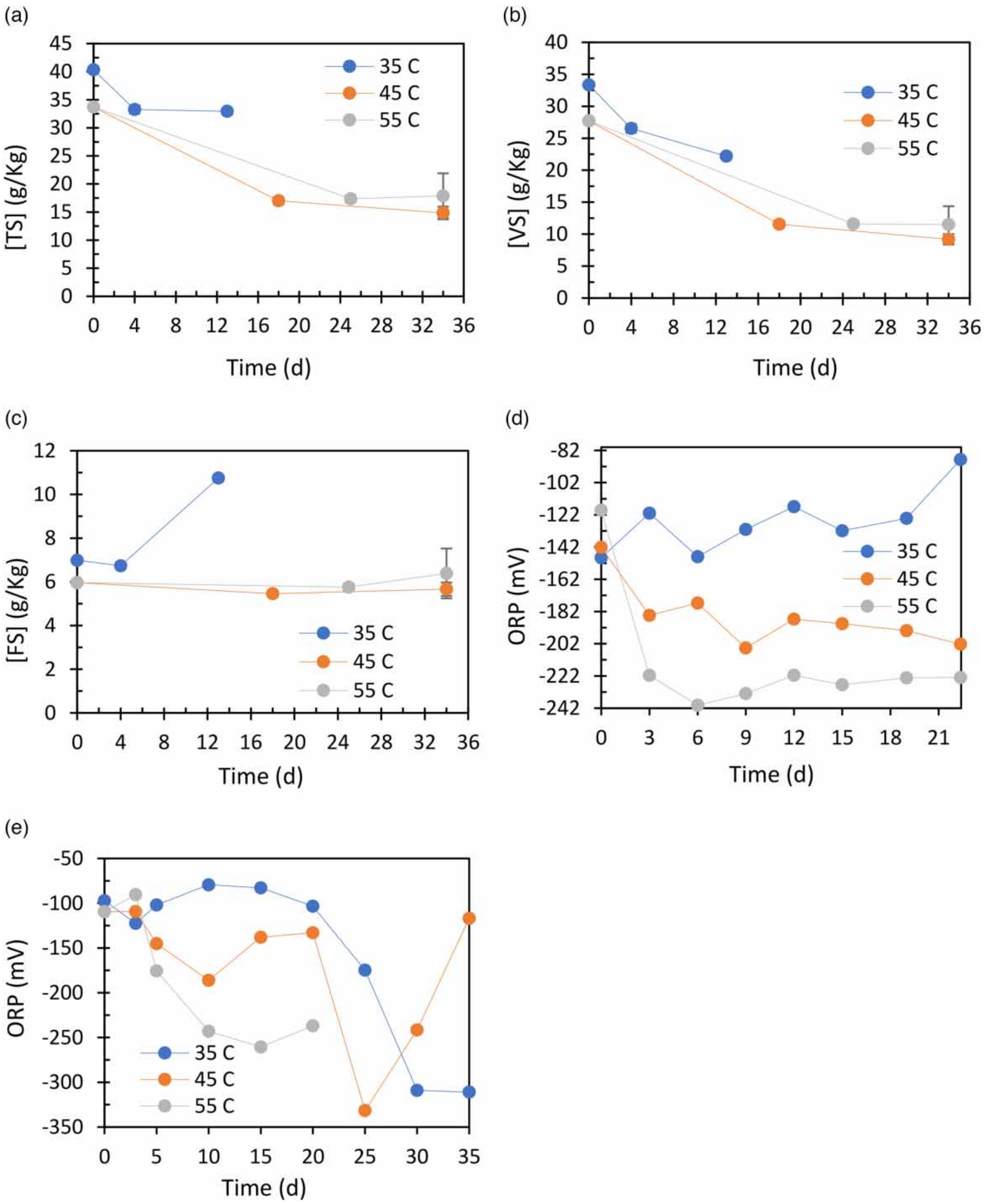


Figure 2 | Solids concentrations of sludge samples taken from batch anaerobic digesters during inoculum preparation: (a) TS concentration; (b) VS concentration; (c) FS concentration, ORP variations in laboratory digesters; (d) anaerobic; (e) aerobic. 35 C, 45 C and 55 C refer to 35, 45 and 55 °C, respectively.

3.2. Correlations between water fractions

To examine the validity of Equation (12), $f_{(TW-Sup)/Sl}$ values were plotted against $f_{Sup/Sl}$ (Figure 4(a)). It was observed that the two parameters matched each other linearly ($R^2 > 0.99$), which means the exponent in Equation (12) is equal to 1. Such a high coefficient of determination indicates the linear model estimates more than 99% of the cases accurately. Table 1 provides a detailed report of the linear regressions in terms of model parameters and coefficients of determination. The data in the table are categorized based on the sludge type, centrifugation intensity, and duration. This table does not include the data obtained from the influent and digestate of the full-scale anaerobic digester where there was no case in which at least three data points were obtained for the same centrifugation intensity and duration, and it would not be informative to establish regression for fewer than three data points.

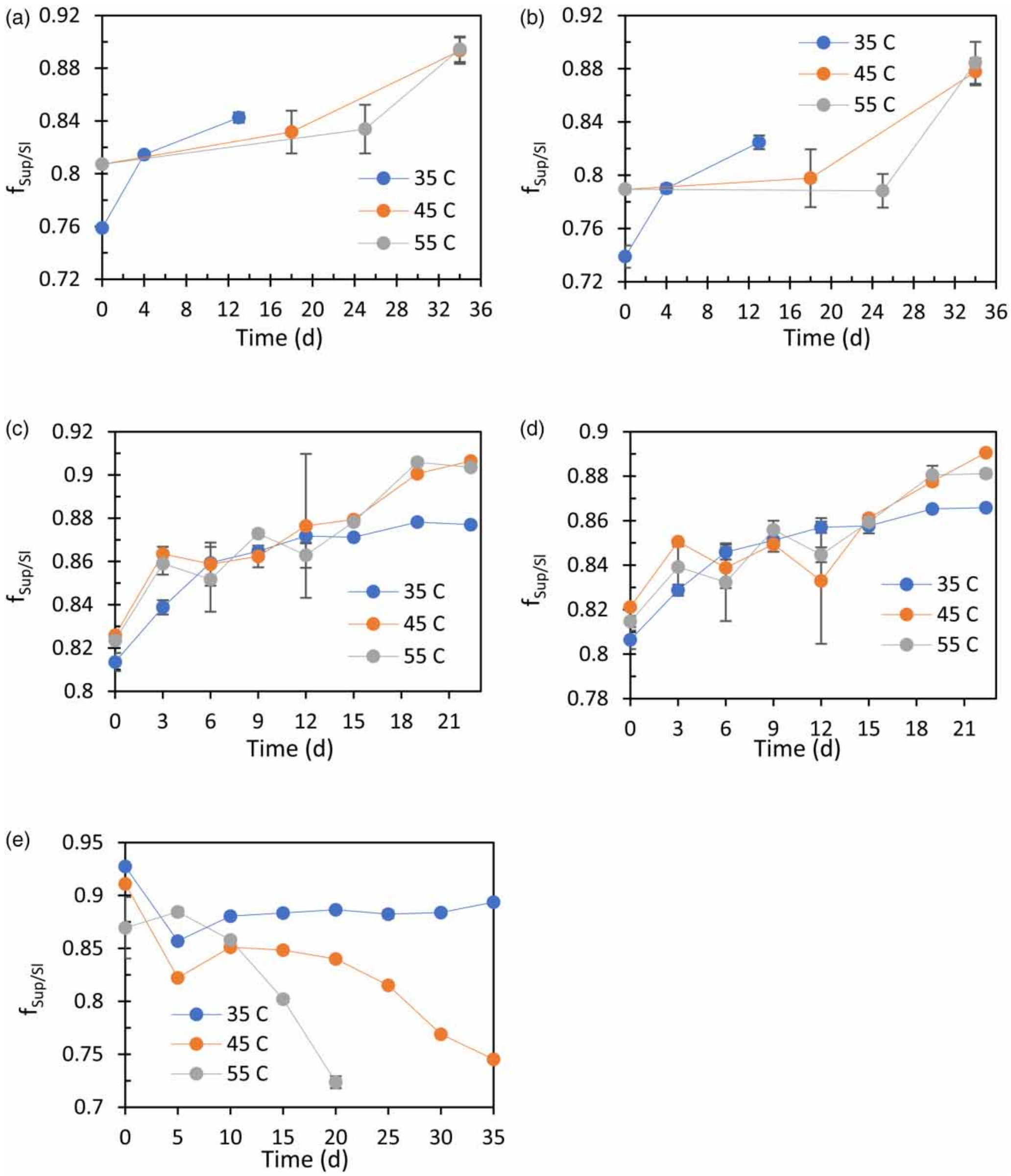


Figure 3 | Variations of $f_{\mathrm{Sup/Sl}}$ during inoculum preparation for batch anaerobic digestion: (a) $C_a = 10{,}000\,g$, $C_t = 10$ min; (b) $C_a = 10{,}000\,g$, $C_t = 10 + 10$ min, variations of $f_{\mathrm{Sup/Sl}}$ during batch anaerobic digestion experiment. (c) $C_a = 14{,}000\,g$, $C_t = 30$ min; (d) $C_a = 14{,}000\,g$, $C_t = 30 + 30$ min. (e) Variations of $f_{\mathrm{Sup/Sl}}$ during batch aerobic digestion ($C_a = 14{,}000\,g$, $C_t = 60$ min). There is only one $f_{\mathrm{Sup/Sl}}$ value for the initial sludge sample taken from AD.35. 35 C, 45 C, and 55 C refer to 35, 45 and 55 °C, respectively.

The first row of Table 1 from top depicts the estimated parameters for the PS fed into the digester. This case had the lowest absolute values for model parameters as well as coefficient of determination. The $f_{(\mathrm{TW-Sup})/\mathrm{Sl}}$ should be the lowest at $f_{\mathrm{Sup/Sl}} = 1$. Also, the PS had the mildest decline of $f_{(\mathrm{TW-Sup})/\mathrm{Sl}}$ non-separable fraction as $f_{\mathrm{Sup/Sl}}$ increased. The lowest R^2 value means that the two fractions were least correlated, as compared to the other types of sludge. The experimental work of the current study showed that the PS could be more easily dewatered than the other types. Nevertheless, the further research is needed to come up with a concrete explanation. Note that the centrifugation intensities and durations were the same for PS and T-WAS cases shown in the table. At the same centrifugation intensity (10,000 g) but two different overall centrifugation times (10 and 10 + 10 min), the closeness of the model estimations was evident for anaerobically digested and mixed sludge samples taken from the WWTP. A similar description would apply to the model fittings of the data obtained

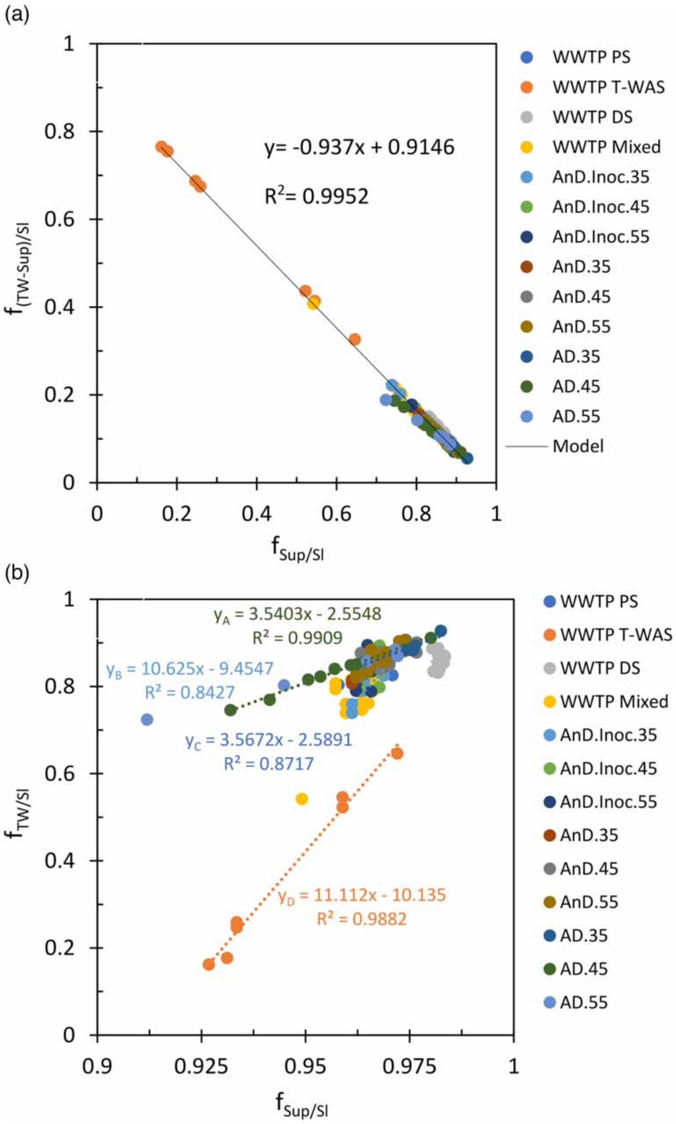


Figure 4 | Correlations between (a) $f_{(\mathrm{TW-Sup})/\mathrm{Sl}}$ and $f_{\mathrm{Sup/Sl}}$ for all the data points and (b) $f_{\mathrm{TW/Sl}}$ and $f_{\mathrm{Sup/Sl}}$ for specific sludge types. In Figure (b), y_A: AD.45, y_B: AnD.Inoc.35, y_C: PS, and y_D: T-WAS.

from the batch anaerobic digesters during inoculum preparation. In the case of the batch anaerobic digestion experiment, the $K_{(\mathrm{TW-Sup})/\mathrm{Sup}}$ and β values slightly decreased after the second centrifugation. Even in this case, the R^2 values of the first and second centrifugations (at 14,000 g for 30 min at each step) were not noticeably different. However, there were clear differences between the values of the model parameters for the data points from the aerobic and the anaerobic digestion experiments, notwithstanding their similar centrifugation conditions.

In spite of all the differences depicted in Table 1, the linear model estimated $f_{(\mathrm{TW-BW})/\mathrm{Sl}}$ very well when it is applied to all the data points obtained (Figure 4(a)). Using the linearity ($n = 1$) and the overall slope and offset value shown on Figure 4(a), Equation (13) is simplified as follows:

$$\frac{m_{(\mathrm{TW-Sup})}}{m_{\mathrm{Sup}}} = \frac{0.9146}{f_{\mathrm{Sup/Sl}}} - 0.937 \tag{14}$$

No universal linear correlation existed between supernatant mass fractions and their corresponding TW mass fractions (Figure 4(b)). The existence and goodness of linear correlation between $f_{\mathrm{TW/Sl}}$ and $f_{\mathrm{Sup/Sl}}$ appeared to be case-specific. The

Table 1 | Estimated model parameters and coefficients of determination for different types of sludges and centrifugation conditions

Sludge type	C_a (g)	C_t (min)	Nu	$K_{(TW-Sup)/Sup}$	β	R^2
PS	8,000	5	3	−0.5759	0.6205	0.8542
T-WAS	8,000	5	3	−0.9095	0.9136	1
DS	8,000	5	3	−0.9207	0.9145	0.9998
DS	10,000	10	3	−0.9355	0.9271	1
DS	10,000	10 + 10	3	−0.9362	0.9287	1
Mixed	10,000	10	3	−0.8929	0.88	0.9943
Mixed	10,000	10 + 10	3	−0.8921	0.8814	0.9959
AnD.Inoc.35	10,000	10	3	−0.9141	0.8955	0.9999
AnD.Inoc.45	10,000	10	3	−0.9538	0.9272	0.9979
AnD.Inoc.55	10,000	10	3	−0.9767	0.9446	0.9987
AnD.Inoc.35	10,000	10 + 10	3	−0.9133	0.8967	0.9994
AnD.Inoc.45	10,000	10 + 10	3	−0.9665	0.9386	0.9976
AnD.Inoc.55	10,000	10 + 10	3	−0.9908	0.9567	0.9989
AnD.35	14,000	30	8	−0.852	0.8407	0.9983
AnD.45	14,000	30	8	−0.8545	0.842	0.9811
AnD.55	14,000	30	8	−0.856	0.8426	0.999
AnD.35	14,000	30 + 30	8	−0.8373	0.83	0.9978
AnD.45	14,000	30 + 30	8	−0.812	0.8086	0.9885
AnD.55	14,000	30 + 30	8	−0.8299	0.8231	0.9989
AD.35	14,000	60	8	−0.7556	0.7577	0.9848
AD.45	14,000	60	8	−0.7201	0.7238	0.9986
AD.55	14,000	60	8	−0.6149	0.6344	0.9964

Notes: Inoc refers to sludge samples taken during inoculums preparations. Nu, number of sludge samples.

left-hand sides of the regression equations AD.45, AnD.Inoc.35, PS, and T-WAS are denoted by y_A, y_B, y_C, and y_D, respectively. R^2 values of the regression equations obtained for AD.45, AnD.Inoc.35, PS, and T-WAS were 0.9909, 0.8427, 0.8717, and 0.9882, respectively. Yet, there was no correlation for the data points obtained from DS sludge samples. Therefore, the application of the correlation between $f_{TW/Sl}$ and $f_{Sup/Sl}$ would be limited to just some of the sludge types specified on Figure 4(b).

As fractions of sludge, both $f_{Sup/Sl}$ and $f_{(TW-Sup)/Sl}$ lie between 0 and 1. $f_{Sup/Sl}$ being 1 means mass of supernatant is equal to mass of sludge from which supernatant is obtained. In other words, such sludge consists of a liquid phase only, with $f_{Sup/Sl}$ and $f_{TW/Sl}$ being the same, and $f_{(TW-Sup)/Sl}$ equaling 0. Although such an extreme case is unrealistic, Equation (12) and the data in Table 1 do indicate that the two fractions have a negative linear relationship. Therefore, as $f_{Sup/Sl}$ approaches 1, $f_{(TW-Sup)/Sl}$ approaches 0. Examination of Equation (12) under such condition reveals β equals $-K_{(TW-Sup)/Sup}$. From this perspective, a negative linear correlation has to exist between the model parameters for a set of equations. This was confirmed when β and $K_{(TW-Sup)/Sup}$ (Table 1) values were plotted against each other, as shown in Figure 5.

Rearrangement of Equation (12), with the exponent being 1, and solving it for supernatant mass fraction yields Equation (15). This equation shows how the TW/TS and supernatant mass fractions are linked through the proportionality factor. Based on this expression, as TS concentration increases, $f_{Sup/Sl}$ goes down. In other words, an increase in the concentration of total solids results in lowered centrifugal dewaterability. This relationship would apply to the raw sludge and DS. This is consistent with the increased capillary suction time (CST), a measure of dewaterability, with solids concentration sludge observed by Wang *et al.* (2020).

$$f_{Sup/Sl} = \frac{1 - \left(\frac{[TS]}{1,000} - \beta\right)}{1 + K_{(TW-Sup)/Sup}} \tag{15}$$

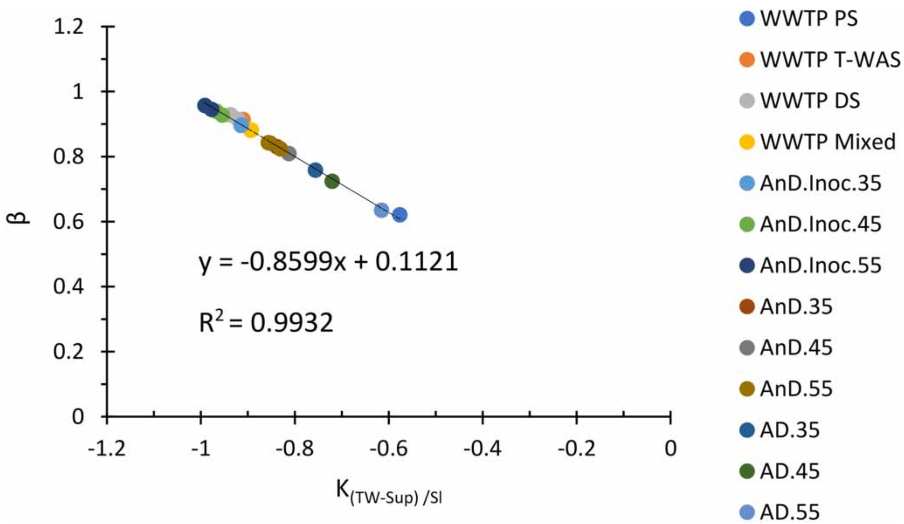


Figure 5 | Correlation between model parameters.

Equation (16) is a modified version of Equation (15) to estimate the mass flowrate ($\dot{m}$) of dewatered sludge (DeW-Sl) leaving a continuous flow centrifuge (see Section S7). ρ_{Sl} is the density and Q_{Sl} is the volumetric flowrate of sludge before entering centrifuge. ρ_{Sl} depends on solids content and composition, and it is very close to water density (Goncalves *et al.* 2007). Equation (16) may be considered a tool for the design and operation of centrifugal sludge dewatering systems, as it is based on a model with very high R^2 value (Equation (12)). Using the values of the relevant parameters for the digestate of the full-scale digester, provided Table 1 and Table S5, the mass flowrate of the dewatered digestate is estimated to be 3.62 kg/s.

$$\dot{m}_{\mathrm{DeW-Sl}} = \rho_{\mathrm{Sl}} \cdot Q_{\mathrm{Sl}} \cdot \left[\frac{K_{(\mathrm{TW-Sup})/\mathrm{Sup}} + \frac{[\mathrm{TS}]}{1,000} + \beta}{1 + K_{(\mathrm{TW-Sup})/\mathrm{Sup}}} \right] \tag{16}$$

It is realized that the C_a levels applied in the present work were high in comparison with the levels reported in the literature (Table 2). In full-scale plants, where the use of solid-bowl centrifuges is common, rotational speeds and bowl radii could be up to 3,200 rpm and 0.9 m, respectively (Goncalves *et al.* 2007). This means the machine can exert C_a values as high as 10,300 *g*. This value is still lower than 14,000 *g* applied to many sludge samples of the current work. Due to the geometry and practical differences, the type of laboratory centrifuge used in the current study would not be appropriate for sludge dewatering at those

Table 2 | Centrifugation accelerations and times used in other investigations

Sludge type	Hydraulic regime	C_a (g)	C_t (min)	Reference
Not specified	Batch	46–1,949	5–20	Vesilind & Zhang (1984)
WAS	Batch	Up to 2,414 (4,000 rpm)[a]	60	Lee (1994)
PS + WAS	Continuous flow rate = 6.8–16 m^3/h	2,500 and 3,000		Leung (1998)
WAS	Batch	400	10	Chen *et al.* (2001)
WAS	Batch	32–200	Up to about 300	Chu & Lee (2002)
Anaerobically digested WAS	Batch	2,000	10	Wang *et al.* (2016)
Fecal sludge	Batch	3,345	10	Ward *et al.* (2019)

[a]The number provided in the paper was 4,000 rpm, and C_a was approximated using the equation presented in Section 2.4.

low accelerations applied in the field. If such low but realistic centrifugation accelerations were applied, a significant amount of the pelletized solids would be resuspended into the supernatant (because of pulling the centrifugation vials out of the rotor, among other reasons). That would affect the accuracy of the experimental work. Nevertheless, one has to bear in mind that the actual shear imparted on sludge in a continuous solid-bowl centrifuge can be higher than what can be done by centrifugation at 10,300 *g*. This is due to the shear force that appears as a result of the difference in the velocities of the spinning bowl and screw conveyor located in the proximity of each other inside the centrifuge.

3.3. Bound water content

Sludge water can be divided into free water and bound water (BW). The latter consists of adsorbed, capillary, and cellular waters (Gray 2004; Goncalves *et al.* 2007). According to the procedures used for BW quantification in other studies (Masihi & Gholikandi 2020, 2021; Wang *et al.* 2020), BW fraction should be the fraction remaining in the pellet after centrifugation. Therefore, $f_{(\mathrm{TW-Sup})/\mathrm{Sl}}$ can be considered BW fraction or a representative of it. As BW is associated with suspended solids, there should be a correlation between the two. Wu *et al.* (1998) demonstrated that BW content of kaolin sludge exponentially decreased with decreasing solids content. A correlation was reported between VSS concentration and dewaterability of various kinds of sludge (Skinner *et al.* 2015). A multiple regression model with an R^2 value of 0.938 was developed by Zhang *et al.* (2019a), in which BW as a fraction of TW was linearly associated with the VS content expressed in percent. An increase of bound-to-TW from 7 to 13% was observed by Wang *et al.* (2020) when VS-to-TS ratio was increased from 35.72 to 61.11%. For all sludge samples of the present work, TS, VS, and FS were determined. Also, respective suspended fractions were calculated through subtraction of the dissolved counterparts in some cases. Even for those sludge samples where the suspended solids fractions were not determined, the total solids fractions (including volatile and fixed) would represent the corresponding suspended fractions. Numerous measurements from the sludges of anaerobic and aerobic digesters showed that suspended solids fractions are 88% $\pm$ 0.03% of their respective TS fractions (see Table S8). With this perspective, correlations between $f_{(\mathrm{TW-Sup})/\mathrm{Sl}}$ values and solids concentrations were established (Table 3). For cases in which centrifugation was done in two steps and water fractions were determined after each step, only correlations obtained in the second step are shown because there was no difference between the first and second correlations.

Overall, the difference between TW and supernatant mass fractions correlated with volatile and/or FS concentrations. The VS concentration positively correlated with $f_{(\mathrm{TW-Sup})/\mathrm{Sl}}$ for all cases except sludge samples taken during inoculum preparation for anaerobic digestion at 55 °C. This could be an outlier since the number of data points was at a minimum of 3 and the coefficient of determination was only 0.6874. Excluding the inoculum correlations, the slope values ranged from 0.0021 to 0.0134 for the VS-based correlations. Comparison between the [VS]- and [VSS]-based correlations of the sludge samples taken from the aerobic digesters shows that the slopes were very similar.

The correlations in Table 3 confirm that the difference between TW and supernatant mass fractions measured in the current investigation represents the BW water fraction. Accordingly, the $f_{(\mathrm{TW-Sup})/\mathrm{Sl}}$ trends versus time would in fact depict the variations of BW fractions over time. One should notice that those variations occurred while the solids concentrations were changing. To make the varying $f_{(\mathrm{TW-Sup})/\mathrm{Sl}}$ values comparable, normalization should be done. Since the values of the aforementioned parameter correlated with [VS] and/or [FS], normalization can be done by dividing $f_{(\mathrm{TW-Sup})/\mathrm{Sl}}$ values by TS mass fraction. The division of $f_{(\mathrm{TW-Sup})/\mathrm{Sl}}$ by $f_{\mathrm{TS/Sl}}$ is equal to the difference between TW and supernatant masses divided by the corresponding mass of TS, which is denoted by $m_{(\mathrm{TW-Sup})/\mathrm{TS}}$. This ratio may be considered a measure of the hydrophilicity of the sludge solids. The calculated values are plotted in Figure 6(a)–6(e). The trends obtained during the inoculum preparation period for batch anaerobic digestion were not meaningful (Figure 6(a)). Nevertheless, the parameter slightly decreased for sludge samples taken from batch anaerobic digesters during the main digestion experiment (Figure 6(b)). The trends were relatively smooth (i.e., with smaller fluctuations of $m_{(\mathrm{TW-Sup})/\mathrm{TS}}$) for AnD.35 and AnD.55, and somewhat erratic for AnD.45. This might be related to the fluctuations in the VS removal by the reactor, which was discussed in a previous study (Poorasgari & Örmeci 2022). The final $m_{(\mathrm{TW-Sup})/\mathrm{TS}}$ values were about 3.5, which is similar to the BW fraction of an anaerobic DS reported by Robinson & Knocke (1992). The overall reduction of $m_{(\mathrm{TW-Sup})/\mathrm{TS}}$ was indeed expected, as sludge stabilization resulting in VS destruction improved centrifugal dewatering (Goncalves *et al.* 2007). This can be elucidated by the fact that supernatant mass fraction increased with the progression of anaerobic digestion (see Figure 3(b) and 3(c)). In line with this perspective, BW and supernatant mass were investigated using a variety of techniques, and it was demonstrated that anaerobic digestion of wastewater sludge at 35 °C improved dewaterability (Zhang *et al.* 2019b). $m_{(\mathrm{TW-Sup})/\mathrm{TS}}$ levels determined for the influent and digestate of the full-scale anaerobic digester of the WWTP indicated

Table 3 | Correlations between $f_{(TW-Sup)/SI}$ values and solids concentrations

Sludge type	C_a (g)	C_t (min)	Number of data points	$f_{(TW-Sup)/SI}$ =	R^2
Various[a]	8,000	5	11	0.0134[VS] − 0.077 0.041[FS] − 0.0974	0.6749 0.9005
Various[a]	10,000 10,000	10 + 10 10 + 10	9	0.0106[VS] − 0.0359 0.0515[FS] − 0.1864	0.5923 0.9119
AnD.Inoc.35	10,000	10 + 10	3	1.4941[VS] − 19.898 −0.0133[FS] − 2.884	0.9383 0.5718
AnD.Inoc.45	10,000	10 + 10	3	0.4687[VS] − 13.827 −0.1643[FS] − 1.1351	0.4361 0.9192
AnD.Inoc.55	10,000	10 + 10	3	−0.3714[VS] − 27.276 −0.0262[FS] − 0.005	0.6874 0.0203
AnD.35	14,000	30 + 30	8	0.005[VS] − 0.0005 0.0132[FS] + 0.014	0.9081 0.1475
AnD.45	14,000	30 + 30	8	0.0033[VS] − 0.0403 −0.0094[FS] + 0.1914	0.8667 0.4482
AnD.55	14,000	30 + 30	8	0.0042[VS] − 0.0137 −0.0181[FS] + 0.2597	0.9752 0.4718
Full-scale influent and digestate	14,000	30 + 30 and 60	4	0.0024[VS] − 0.0756 0.03[FS] − 0.1036	0.9132 0.091
AD.35	14,000	60	8	0.0028[VS] + 0.0352 0.0028[VSS] + 0.0404 0.0266$[VDS]_{SI}$ + 0.0274 0.0115[FS] + 0.011 0.0038[FSS] + 0.0676 0.065$[FDS]_{SI}$ + 0.0786	0.629 0.5857 0.5423 0.8716 0.1318 0.2637
AD.45	14,000	60	8	0.0041[VS] − 0.0056 0.0048[VSS] − 0.0105 0.0216$[VDS]_{SI}$ + 0.0488 0.006[FS] + 0.054 0.0063[FSS] + 0.0567 0.0763$[FDS]_{SI}$ + 0.0464	0.9445 0.9122 0.8618 0.9473 0.958 0.5071
AD.55	14,000	60	8	0.0021[VS] − 0.0521 0.0023[VSS] − 0.0515 0.0179$[VDS]_{SI}$ + 0.0602 0.0067[FS] + 0.0508 0.0007[FSS] + 0.0541 0.1006$[FDS]_{SI}$ + 0.0288	0.9682 0.9762 0.8584 0.9926 0.9921 0.657

Note: Inoc refers to sludge samples taken during inoculums preparations.
[a]PS, T-WAS, DS, and mixed.

that the BW fraction increased after digestion (Figure 6(c)). However, statistical analysis conducted on March and July samples combined showed that the difference between $m_{(TW-Sup)/TS}$ values of the influent and digestate was insignificant (p-value = 0.25).

However, and in contrast to the overall $m_{(TW-Sup)/TS}$ trajectories during the anaerobic digestion of the present study, it was reported anaerobic digestion increased BW/TS mass ratio from 1.6–2.1 to 1.7–2.7 (García-Bernet *et al.* 2011). Furthermore, anaerobic sludge digestion at 20 and 55 °C increased CST (Novak *et al.* 2003; Wang *et al.* 2016), indicating the digestion process worsened sludge dewaterability. This was in agreement with the increasing CST observed for sludge digestion in completely stirred tank reactors operated at 35 and 55 °C during the startup phase (Gebreeyessus 2020). Moreover, increasing the operating temperature of the digester from 15 to 35 °C lowered CST during anaerobic digestion (Hidaka *et al.* 2022). Therefore, the effect of biological digestion on sludge dewaterability could be dependent on the dewatering method and how dewaterability is quantified.

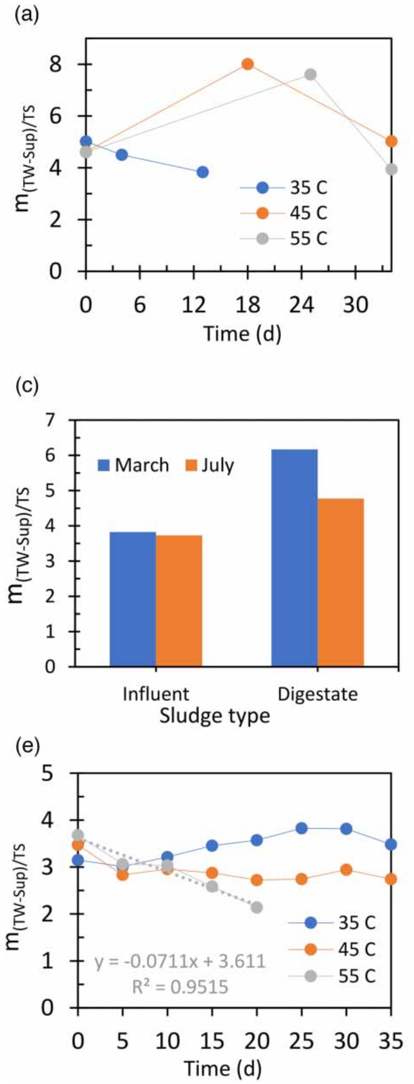


Figure 6 | $m_{(TW-Sup)/TS}$ values for (a) batch anaerobic digesters during inoculum preparation, (b) batch anaerobic digesters during anaerobic digestion, (c) influent and digestate of full-scale digester, (d) different types of sludge sampled from WWTP, and (e) batch aerobic digesters during aerobic digestion. Nu refers to the number of sludge samples. 35 C, 45 C, and 55 C refer to 35, 45, and 55 °C.

Further examination of the sludge samples from the plant showed that $m_{(TW-Sup)/TS}$ values had the following order with either centrifugation conditions: T-WAS > DS > PS (Figure 6(d)). The mentioned order means the most hydrophilic solids existed in T-WAS. In this case, as well, the respective $f_{Sup/Sl}$ values (see Table S6) were in line with $m_{(TW-Sup)/TS}$ levels, meaning that T-WAS was the most difficult sludge of the current research to dewater by centrifugation. This is despite the positive effects of the phosphorus removal chemicals (see Section 2.1) added to the recirculated activated sludge (Ranjbar *et al.* 2021). Further processing of the recirculated sludge in the aeration tank might have negated those positive effects. Using a combination of centrifugation and vacuum filtration, the BW content of waste activated sludge was found to be 3–5 kg/kg of dry solids (Lee 1994). The relatively high $m_{(TW-Sup)/TS}$ value for the T-WAS in the current research is consistent with the findings of Yin *et al.* (2006), using the dilatometric method to determine BW fraction and reported 16.7 g BW/g dry solids. Conversely, 1 g BW/g dry solids for the same type of sludge was determined when differential thermal analysis was used for bound water measurement (He *et al.* 2017). Using the same method, BW content of activated sludge was measured to be about 2.2 g/g of dry solids (Chen *et al.* 2021). Having employed a centrifugation method, 3.8 g BW/g of dry solids was measured for activated sludge (Masihi & Gholikandi 2021). Jin *et al.* (2004) showed that the value of the BW fraction depends on the centrifugal force applied. Important links between extracellular polymeric substances, floc structures, and BW content have been demonstrated (He *et al.* 2017). Thus, the discrepancies could be due to not only the employed methods but also different sludge characteristics.

For the sludge samples taken from laboratory aerobic digesters, there were three different trends of $m_{(TW-Sup)/TS}$ over digestion time (Figure 6(e)). There was an overall increase in the level for AD.35 and the final value was 10% higher than the initial. For AD.45 and AD.55, however, the trends showed a decline. The final values were 22 and 42% lower than the initial for the reactors operated at 45 and 55 °C, respectively.

Particularly in the case of AD.55, the $m_{(TW-Sup)/TS}$ profile followed a strong linear function. A clear incongruity between the three reactors was the intensity of the water evaporation rate. The difference in the trends could be caused by the difference in evaporation rates and/or the temperature of the digestive process both directly and indirectly. About 70% of sludge water is free water and about 20% is floc water (Gray 2004). These two fractions are understandably subject to evaporation. The bound water fraction consists of capillary water and intracellular water. The latter is four times greater than the former (Gray 2004). Hence, the release and evaporation of intercellular water that could have ensured a probable heat/evaporation-induced cell lysis could be the main underlying mechanism. Supernatant mass fraction of sludge went down as aerobic digestion proceeded at 45 and 55 °C, while it was not very clear in case of AD.35 (see Figure 3(e)). The $f_{Sup/Sl}$ reduction observed for AD.45 and AD.55 should be scrutinized. Such reductions can be generally regarded as an indication of worsening dewaterability. Nevertheless, the simultaneous decrease in $f_{Sup/Sl}$ and $m_{(TW-Sup)/Sl}$ points to the evaporation of both free and bound water fractions, supporting the notion that the intercellular water released due to cell lysis was evaporating during digestion. In line with this conception, the mildest overall change in both $f_{Sup/Sl}$ and $m_{(TW-Sup)/Sl}$ were experienced by AD.35, the reactor with the lowest operating temperature.

3.4. Important implications and future perspectives

Outcomes of this study suggest that the water content of different kinds of sludge can be estimated from the total and volatile solids fractions. This may be of significance to the design and operation of future sludge dewatering facilities. Although the dewatering process applied in the current study was centrifugation, it is likely that similar patterns could apply to other dewatering processes. Also, the presented results indicate that the bound water content, which is not possible to remove by dewatering processes, can be reduced by biological digestion of the wastewater sludge.

4. CONCLUSION

A linear model was developed to link total, supernatant, and bound water of wastewater sludge, which enables us to estimate the mass fractions of supernatant and sludge cake solids after centrifugal dewatering. The model was successfully applied to a wide range of sludges including PS, T-WAS, and aerobic and anaerobic DS under mesophilic and thermophilic conditions. The model developed to estimate the amount of thickened/DeW-Sl in the current study may be used as a helpful tool for the design and operation of centrifugal sludge thickening and dewatering systems. Among the sludges tested, T-WAS had the most hydrophilic solids and thus the highest bound water content. Aerobic and anaerobic digestion affected centrifugal dewaterability of the sludges differently, and digestion temperature played a key role in determining the distribution of water fractions. Higher temperatures (45 and 55 °C) during anaerobic digestion increased centrifugally separated water, while the opposite was observed during aerobic digestion. However, aerobic digestion at those temperatures co-occurred with a high level of water evaporation. This resulted in DSs with far lower centrifugally separable water contents, as compared to anaerobic DSs.

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DATA AVAILABILITY STATEMENT

All relevant data are included in the paper or its Supplementary Information.

CONFLICT OF INTEREST

The authors declare there is no conflict.

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