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Impact of volatile solids destruction on the shear and solid-liquid separation behaviour of anaerobic digested sludge

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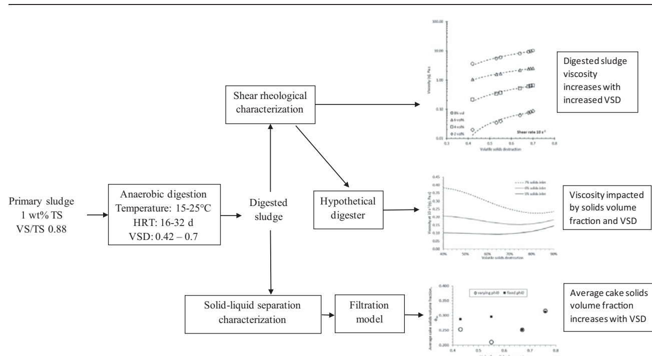
HIGHLIGHTS

- Digestate viscosity increases 1.3 to 3.3 times when VSD changes from 43 % to 70 %.
- Optimum digestate viscosity is achieved in the VSD range 65 %–80 %.
- Dewatering behaviour (final cake solids) improves by increasing VSD.
- Final cake solids increases from 21 % to 31 % when VSD changes from 55 % to 76 %.
- VSD has no impact on solids flux and throughput of thickener and filter press.

GRAPHICAL ABSTRACT



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ABSTRACT

Systematic and comprehensive characterisation of shear and solid-liquid separation properties of sludge across a wide range of solids concentration and volatile solids destruction (VSD) is critical for design and optimization of the anaerobic digestion process. In addition, there is a need for studies at the psychrophilic temperature range as many unheated anaerobic digestion processes are operated under ambient conditions with minimal self-heating. In this study, two digesters were operated at different combinations of operating temperature (15–25 °C) and hydraulic retention time (16–32 d) to ensure a wide range of VSD in the range of 0.42–0.7 was obtained. For shear rheology, the viscosity increased 1.3 to 3.3 times with the increase of VSD from 43 % to 70 %, while other parameters (temperature, VS fraction) having a negligible impact. Analysis of a hypothetical digester indicated that there is an optimum VSD range 65–80 % where increase in viscosity due to the higher VSD is balanced by the decrease in solids concentration. For solid-liquid separation, a thickener model and a filtration model were used. No significant impact of VSD on the solids flux, underflow solids concentrations or specific solids throughput was observed in the thickener and filtration model. However, there was an increase in average cake solids concentration from 21 % to 31 % with increase of VSD from 55 % to 76 %, indicating better dewatering behaviour.

1. Introduction

Anaerobic digestion of sewage sludge is widely used in wastewater treatment facilities (Mata-Alvarez et al., 2000). The most common

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application is a well-mixed digester configuration, but technologies such as covered anaerobic lagoons, biofilm or plug flow reactors are also used (Mao et al., 2015). Effective mixing of the sludge during digestion is necessary to maintain design HRT, and consistent temperatures, microbe and solids distribution (McLeod et al., 2019). Mixing is energy intensive and is dependent on fluid viscosity, which in digesters, is non-Newtonian (Sadino-Riquelme et al., 2018; Wei et al., 2018). Hence, the rheological behaviour of the digester sludge plays a key role in the heat and mass transfer processes during anaerobic digestion. In addition, the solid-liquid separation or dewatering behaviour of digested sludge is important to minimise sludge volume, facilitate transportation, and reduce leachate production during transport, storage, and final use (Hong et al., 2018). 50–70 % of the overall wastewater treatment plant operation cost is associated with sludge management and disposal, so understanding the evolution of shear rheological and solid-liquid separation properties of anaerobic digested sludge is critical for optimizing the digestion process performance and digestate management (Dieudé-Fauvel et al., 2014; Miryahyaei et al., 2020a; Miryahyaei et al., 2019).

Digested sludge has been widely characterised as a shear-thinning (viscosity decreases with the increase in shear rate), non-Newtonian suspension with a shear yield stress at total suspended solids (TSS) concentrations above the gel point due to a continuous particulate network (Baudez et al., 2011; Stickland, 2015). The Herschel-Bulkley model is the most applicable model to describe these characteristics (Markis et al., 2016; Stickland et al., 2013). This model has three fitting parameters (consistency, k, flow index, n, and shear yield stress, τ_y), which can be obtained from the best fit of the experimental rheograms (shear stress and shear rate data). These model fitting parameters can be measured across the range of suspended solids volume fractions (ϕ) relevant to the process (Ratkovich et al., 2013). k, n and τ_y are dependent on the solids concentration, VSD and digestion temperature (Baroutian et al., 2013; Cao et al., 2016; Eshtiaghi et al., 2013; Miryahyaei et al., 2019).

The compressional rheology approach of Buscall and White (1987) is a rigorous approach that can be used to characterise solid-liquid separation properties of the digested sludge across a broad range of solid volume fractions. Gravity batch settling tests are used for characterisation at low solids concentrations and slightly beyond the gel point (Lester et al., 2005), whereas single pressure filtration tests are conducted for characterisation at high solids concentration to obtain the compressibility and permeability (Stickland, 2015; Stickland et al., 2008a). The solid-liquid separation parameters, compressive yield stress, $P_y(\phi)$, and hindered settling function, $R(\phi)$, can be used to predict the compressive settling behaviour of the sludge at different solids concentrations.

The evolution of sludge rheology and solid-liquid separation has been studied widely, and it has been observed that shear yield stress, viscosity, and dewaterability decrease (improve) through the anaerobic digestion process (Forster, 2002; Guibaud et al., 2004; Miryahyaei et al., 2019; Miryahyaei et al., 2020b; Moeller and Torres, 1997; Monteiro, 1997). Dai et al. (2014); Zhang et al. (2016) observed a decrease in shear rheological parameters with anaerobic digestion at higher solids retention time (SRT) due to reduction in solids content and concluded that SRT has higher impact on sludge rheology than AD temperature. Feng et al. (2016) had similar observations where sludge viscosity decreased significantly due to anaerobic digestion and attributed this to the changes in VSS and sludge composition and structure. In general, the reduction in shear and improvement in dewaterability properties can be attributed to both the reduction in solids concentration due to the degradation of organic matter as well as changes to microstructure due to digestion and the destruction of volatile solids. The way in which these two interact, and hence, an optimum VSD for rheology and dewaterability has not been previously assessed quantitatively, and is critical operationally. Therefore, comprehensive and simultaneous characterisation of shear and solid-liquid separation properties across a wide range of solid volume fractions and VSD is needed to quantitatively differentiate the impacts of changes to microstructure and solids concentration, which is currently unavailable in existing literature.

It is to be noted that most of the dewatering studies used capillary suction time (CST) or specific resistance to filtration (SRF) tests to measure the dewatering parameters. One of the major shortcomings of these test methods is that the results are not related to material properties (Christensen et al., 2015; Skinner et al., 2015). In a compressional rheology approach, the dewatering behaviour of sludges are characterised using sedimentation and filtration tests to produce a description of dewatering material property including compressive yield stress, $P_y(\phi)$, hindered settling function, $R(\phi)$, and solids diffusivity, $D(\phi)$. These material properties provide a more comprehensive description of sludge dewatering behaviour across a wider range of solids concentrations and can be used as input for appropriate modelling of separation processes such as thickening and filtration to inform equipment design and optimization of operation (Stickland, 2015; Stickland and Buscall, 2009).

The evolution of rheological and solid-liquid separation behaviour in anaerobic digestion reflects the complex changes in the sludge microstructure as organic matter is degraded and microbial colonies grow and exude extracellular polymeric substances (EPS). These complex polymeric networks have been reported as responsible for stronger water binding capability and higher viscosity of the digested sludge (Houghton and Stephenson, 2002; Houghton et al., 2001; Li and Yang, 2007; Neyens et al., 2004; Raynaud et al., 2012; Shin et al., 2001). The increase in the EPS with digestion, indicates the presence of a strong correlation for the shear rheological and solid-liquid separation properties of digested sludge as a function of VSD, which has not yet been rigorously developed.

In addition, most of the existing studies on the rheology and solid-liquid separation behaviour of anaerobic digested sludge have been conducted in the mesophilic temperature range (37 °C) (Cao et al., 2016; Dieudé-Fauvel et al., 2014; Forster, 2002; Miryahyaei et al., 2019; Miryahyaei et al., 2020b). In contrast, anaerobic digestion processes such as anaerobic lagoons are unheated systems where there is a significant seasonal variation in operating temperature. This is typically in the psychrophilic range (15 °C to 25 °C), which has lower VSD than mesophilic digestion (Bowen et al., 2013; Lettinga et al., 2001; Lin et al., 2016; Massé et al., 2003). Operating temperature is a critical factor in efficient digestion of organic matter (Batstone et al., 2002; De Vrieze et al., 2015; Vanwonterghem et al., 2015) and will influence the shear and solid-liquid separation properties. Hence, understanding the shear and solid-liquid separation behaviour in these psychrophilic conditions can have potential application in modelling and optimization of unheated anaerobic digestion systems.

In this study, two continuously fed digesters were operated at different combinations of operating temperature (15 °C and 25 °C) and HRT (16, 24 and 32-d) to ensure a wide range of VSD (0.42 to 0.7) is obtained. Shear rheology and solid-liquids separation properties were determined and linked to observed VSD. For shear rheology, a hypothetical digester was modelled to investigate the combined impact of solids volume fraction and VSD on the digested sludge viscosity. A thickener model and a filtration model were used to investigate the impact of VSD on solid-liquid separation properties.

This is the first study where the simultaneous impact of VSD and solids concentration on the shear and solid-liquid separation behaviour of anaerobic digested sludge was investigated in the psychrophilic temperature range, especially using the compressional rheology approach to characterize the dewatering behaviour.

2. Materials and methods

2.1. Continuous digesters

Two Lobster Max-i digesters (Anaero Technologies LTD, Cambridge, United Kingdom) were used (Fig. 1). The digesters had an operating volume of 5.2 L. They were fabricated of stainless steel with a horizontal discharge port of 32 mm diameter for the collection of digestate. The operating temperatures of the digesters were monitored using fitted thermocouples (temperature data were logged in the PLC system). Insulated heater jackets (110 V) were used to operate the digesters in the range of 22 °C–32 °C

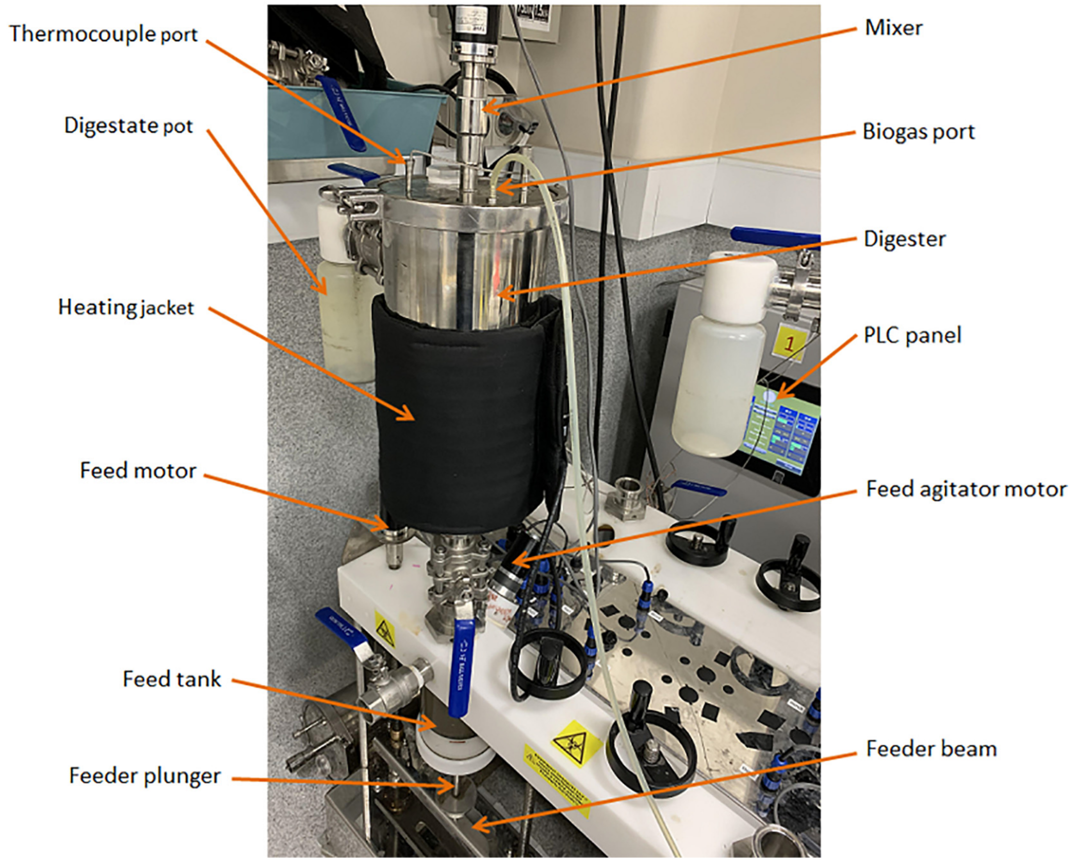

Fig. 1. Continuous digester with PLC controlled heating, feeding and mixing systems.

(temperature controlled by the PLC). A combination of refrigerated bath and heater circulator system was used to operate at 15 °C. The digester contents were continuously mixed using gas-tight top mounted paddle mixers rotating at 40 rpm. The biogas production from the digesters was measured using a calibrated liquid displacement gas flow meter.

The digesters were automatically fed by a syringe pump system programmed via a PLC controller from 1 L feeder syringes located at the bottom of each digester. The feeding interval was 1 h and the feed volume and duration depended on the respective HRT. The feed was pre-mixed for 90 s to ensure homogeneous feeding. A non-return valve was fitted between the digester and the feeder syringes to prevent the movement of digester content into the feeder syringe.

2.2. Feed and operating conditions of continuous digesters

The digesters (D1 and D2) were inoculated with digested sludge samples obtained from anaerobic lagoons of Western Treatment Plant, Werribee, Victoria, Australia. The total solids (TS) and volatile solids (VS) content of the inoculum was 8.34 wt% and 5.25 wt% with a volatile to total solids contents (VS/TS) ratio of 0.63. The pH of the inoculum was 7.2. Primary sludge obtained from Eastern Treatment Plant, Carrum Downs, Victoria, Australia was fed to digesters. The TS content of the primary sludge varied between 3.9 wt% to 4.5 wt% and the VS content was between 3.5 wt% and 3.96 wt%. The VS/TS was approximately 0.88. The primary sludge feed was diluted to 1.12 ± 0.06 wt% TS (0.92 ± 0.05 wt% VS) using its own supernatant obtained from centrifugation at 3500 rpm for 1 h in an Eppendorf 5804 centrifuge (Eppendorf AG, Hamburg, Australia).

Both D1 and D2 were initially operated at 32 °C and 32-d HRT. After this initial start-up period, the operating temperatures of both digesters were decreased to 25 °C and maintained at these conditions (25 °C and 32-d HRT) until steady state was achieved, based on VSD. The operating temperature of digester D2 was then decreased to 15 °C, requiring the refrigerated bath and heater temperature control described above. The two digesters were operated at these combinations of operating temperature and HRT (D1 at 25 °C and 32-d HRT, D2 at 15 °C and 32-d HRT) until sufficient samples were collected for characterisation of shear rheological and solid-liquid separation properties.

After this, the HRTs of both digesters were lowered to 16-d. However, the digester operating at 15 °C (D2) became unstable due to low pH (as low as 5.7) and high soluble COD. The HRT of digester D2 was increased to 24-d to prevent souring. Both digesters were operated at these conditions (D1 at 25 °C and 16-d HRT, D2 at 15 °C and 24-d HRT) until sufficient samples were collected for material characterisation.

2.3. Volatile solids destruction (VSD)

The VSD was calculated from the mass balance (Eq. 1) (Switzenbaum et al., 2003) using the VS concentrations in the inlet (feed) and outlet (effluent).

$$\mathrm{VSD}\ (\%) = \frac{\mathrm{VS}_{\mathrm{conci}} - \mathrm{VS}_{\mathrm{conco}}}{\mathrm{VS}_{\mathrm{conci}}} \times 100 \tag{1}$$

where $\mathrm{VS}_{\mathrm{conci}}$ and $\mathrm{VS}_{\mathrm{conco}}$ are the VS concentrations (g/kg) in the inlet and outlet, respectively.

2.4. Physicochemical properties

The physicochemical properties such as TS, VS, dissolved and suspended solids contents (TDS and TSS), solids and liquid densities and the suspended solids volume fractions were measured and calculated using the methods and equations described in Das et al. (2022b).

The TS and VS contents were measured for feed and digested sludge samples collected from the digesters in two to three-day intervals. The

TSS, solids density and solids volume fractions were measured for the digested sludge samples used for shear rheology and solid-liquid separation characterisation as described in Section 2.5 and Section 2.7.

2.5. Measurement of shear rheology

All shear rheology measurements were performed using the geometries (Table S.2.5) and methods described in Das et al. (2022b). Steady-state viscosities were measured at constant rate using a stepped logarithmic distribution of decreasing angular velocities from 50 to 0.01 rad/s for the vane geometry and 30 to 0.001 rad/s for the bob geometry. The steady-state torque values were recorded using the selection criteria of 1 % tolerance between three consecutive readings.

The methods and equations for calculation of shear stress and shear rate for the narrow gap bob and wide gap vane geometry are presented in Das et al. (2022b).

Shear rheological characterisation of digested sludge samples collected under each operating condition was conducted (Table 1). Each set of digested sludge samples was obtained from combining the sludge samples collected from the digesters in two to three days interval over the days of operation at the same condition. The sludge for each set of samples was then centrifuged in a Beckman Coulter Allegra X-12 centrifuge at 3500 rpm for 2 h to concentrate in the range of 10 wt% to 14 wt%. The centrate collected from centrifugation of the same sample were used to progressively dilute concentrated sludge samples to lower solids concentrations.

2.6. Measurement and curve fitting of solid-liquid separation properties

Solid-liquid separation properties for four sets of digested sludge samples collected at different operating conditions (temperature and HRT) were characterised using batch sedimentation and pressure filtration tests. The operating conditions and corresponding VSD at the time of sample collection is presented in Table S2.7.

Dewaterability data were extracted from gravitational batch settling tests at low TSS (0.5 to 1.1 wt%), whereas dewaterability data at high TSS were determined from several single pressure filtration tests.

The methodology for the extraction and curve fitting of solid-liquid separation properties from the analysis of gravity batch settling and constant pressure filtrations tests is provided in Das et al. (2022b).

2.6.1. Steady-state thickener model

To compare the solid-liquid separation behaviour of the digested sludge samples at lower solids volume fraction range, a steady-state thickener model (Usher and Scales, 2005) was used. Modelling is necessary to quantify the performance in dewatering since performance depends on compressibility and permeability. The modelling here accounts for both the effect of digestion on microstructure and the reduction in TSS due to digestion. For the material properties, the correlations for compressive yield stress and hindered settling function as continuous functions of solids volume fraction were used. The thickener model predicted the solids flux (q) as a function of underflow solids volume fraction (ϕ_{uf}) at different heights. The solids flux (q) is defined as solids volume per unit time per thickener area. However, to be consistent with industry conventions, all q values are reported in tonnes h^{-1} m^{-2} in this study.

2.6.2. Fill and squeeze diaphragm filtration model

To compare the solid-liquid separation behaviour of the digested sludge samples at the higher solids volume fractions, a fill and squeeze model for diaphragm filtration (Stickland et al., 2008b) was used. For the material properties, the correlations for $P_y(\phi)$ and $R(\phi)$ as continuous functions of solids volume fraction were provided as input. The fill pressure (Δp_f) and squeeze pressure (Δp_s), the applied pressures during the fill and squeeze stages, respectively, were constant at 100 kPa. A half-cavity width h_0 of 0.01 m were used. The fill time (t_f) at the beginning and handling time (t_h) after the end of squeeze were 0.01 s and 1200 s, respectively. The fill time (t_f) is the time needed to load the filter cavity with sludge suspension, and the handling time (t_h) consists of the time needed for pipe flushing, plate pack opening, cake discharge, membrane cleaning and pack closing depending on the industrial practice. The fill time was chosen as 0.01 s to start the numerical solution, but step straight into the squeeze phase. The squeeze time (t_s) for the sludge samples varied in the range of approximately 25,000 s to 130,000 s. The filter press model was used to generate specific filtrate volume (V_f) as a function of time (t) for the four digested sludge samples. The $V_f(t)$ results were then used to calculate the specific throughput (Q) as a function of cake solids volume fraction (ϕ) using Eq. 3.

$$Q = \frac{V_f + h_0}{t_f + t_s + t_h} \tag{3}$$

All solid-liquid separation data is stored in Figshare (Das et al., 2022a).

2.7. Estimation of Herschel-Bulkley parameters and hydrolysis coefficient

The secant method (lsqcurvefit) in Matlab R2018b was used to fit the experimental rheograms by non-linear parameter estimation to extract the Herschel-Bulkley model parameters. 95 % confidence intervals were determined based on a two-tailed t-test from apparent parameter error from the last step Jacobian. Parameter correlations were determined from the parameter-residual Jacobian (linear approximation). To identify that the linear approximation was valid, the true parameter confidence region was also evaluated by an F-test (non-linear ANOVA, Batstone et al., 2009).

Apparent hydrolysis coefficient at each temperature was determined by fitting the apparent first order k_T value in the following solution to a steady state 1st order process (Eq. 2) (Batstone et al., 2009).

$$\mathrm{VSD} = f_d\left(1 - \frac{1}{1 + k_T \cdot \mathrm{HRT}}\right) \tag{2}$$

Table 1
Operating conditions, corresponding VSD values and period of sample collection for shear rheological characterisation for the continuous digesters.*

Digester	Temperature, °C	HRT, d	g VS fed/d	Duration of sample collection for rheological characterisation		VS/TS	VSD
				Start, day	End, day		
D1	32	32	1.5	–	–	–	–
	25	32	1.5	233	271	0.582 ± 0.007	0.69 ± 0.03
	25	16	3	309	330	0.631 ± 0.007	0.70 ± 0.02
	25	16	3	377	389	0.603 ± 0.009	0.68 ± 0.01
D2	32	32	1.5	–	–	–	–
	25	32	1.5	–	–	0.584 ± 0.011	0.69 ± 0.04
	15	32	1.5	226	250	0.627 ± 0.018	0.65 ± 0.03
	15	32	1.5	254	271	0.678 ± 0.003	0.55 ± 0.01
	15	16	3	–	–	0.704 ± 0.006	0.54 ± 0.02
	15	24	2	307	323	0.708 ± 0.003	0.42 ± 0.01
	15	24	2	360	377	0.682 ± 0.009	0.54 ± 0.01

* Confidence intervals are 95 % based on a two tailed t-test.

where VSD is the average volatile solids destruction, f_d *is* the degradability (fixed across temperatures), HRT is the hydraulic retention time, and k_T is the hydrolysis coefficient at temperature *T*.

3. Results and discussion

3.1. Impact of operating temperature and HRT on VSD

The variation of VSD for the two digesters (D1 and D2) at different operating conditions (temperature and HRT) is presented in Fig. 2. The averaged VSD data for each set of operating conditions over the days when samples were collected for material characterisation are presented in Table 1. Both the digesters converged to an approximately constant VSD of 0.75 after 120 days of operation at 25 °C and 32-d HRT (220 days of operation from the start-up). After lowering the operating temperature to 15 °C, there was a gradual decrease in the VSD of D2 until it stabilized to around 0.55 after 270 days. When the HRTs of both D1 and D2 were decreased to 16-d, there was a sharp decrease in the VSD of both digesters; however, D1 recovered from this and gradually stabilized to approximately 0.7 VSD after 320 days. In contrast, D2, which was being operated at 15 °C and 16-d HRT, became unstable due to low pH and high soluble COD. So, the HRT of D2 was increased to 24-d HRT after 294 days and maintained at this HRT for the remaining period of operation. At 15 °C and 24-d HRT, the VSD of D2 gradually increased and stabilized to approximately 0.4 after 305 days. There were fluctuations in the trend for both the digesters; with the VSD fraction of D1 averaging approximately 0.7, while that for D2 is approximately 0.55 up to 390 days.

Paired *t*-testing for D1 vs D2 VSD from the data in Fig. 2 during the test period identifies an obvious and significant difference ($p \sim 0$) due to temperature. Analysing unpaired average values in Table 1 by ANOVA (continuous factor on temperature and HRT) identifies a significant impact due to temperature ($p = 0.01$), but a non-significant impact due to HRT, likely because of the limited impact at 25 °C. Fitting f_d, k_{25} and k_{15} across all VSD data in Table 1 identified a degradability (f_d) of 75 % ± 14 %, a k_{15} of 0.09 d^{-1} (upper confidence limit of 0.33 d^{-1}) and a k_{25} of 0.65 d^{-1} (lower confidence limit of 0.13 d^{-1}). Overall R^2 value for the model was 0.77. Confidence intervals for the *k* values were highly asymmetric. Therefore, while the process appears to be significantly faster at 25 °C than 15 °C, this cannot be identified by the parameter analysis due to the limited number of average (steady state) observations.

3.2. Impact of VSD on shear rheology of digested sludge

For each sample described in Table 1, experimental rheograms were generated at six to eight different solids volume fractions using the method outlined in Section 2.5. The experimental rheograms and their Herschel-Bulkley model curve fits for one sample (collected from D1 at 25 °C and 32-d HRT) are presented in Fig. 3(a).

The shear yield stress (τ_y) and consistency (k) were predicted for all the experimental rheograms using an average value of flow behaviour index (n) for each sample. The shear yield stress (τ_y) and consistency (k) were fitted across the entire range of solids volume fractions (ϕ) using both power law and exponential functions. For all samples, the shear yield stress (τ_y) followed a power law relation with ϕ. For consistency (k) as functions of ϕ, an exponential function was most consistent across all samples ($R^2 > 0.95$), although some samples achieved a better fit using a power law function. The correlation coefficients for the power-law fits varied from 0.93 to 0.99. The details of the optimised fits and fitting parameters for each sample is provided in Table 2. The variation of the fitting parameters across the solids volume fractions and their power law and exponential curve fits for one sample (collected from D1 at 25 °C and 32-d HRT) are provided in Fig. 3(b) and (c).

The impact of key physicochemical and operational factors (VS/TS, temperature, HRT, and VSD) was analysed on each parameter via linear modelling. In general, VSD had a strong impact ($p < 0.01$) on all parameters (τ_1, k_1, k_2, and n) except τ_2 ($p = 0.32$). For all other factors (VS/TS, temperature, HRT) tested, none of them were significant, except organic fraction (VS/TS) for k_2, which was poorer ($p = 0.01$) compared with using VSD ($p = 0.008$). This implies that it is the proportion of VS that have been consumed rather than the total amount of VS that has most effect on shear

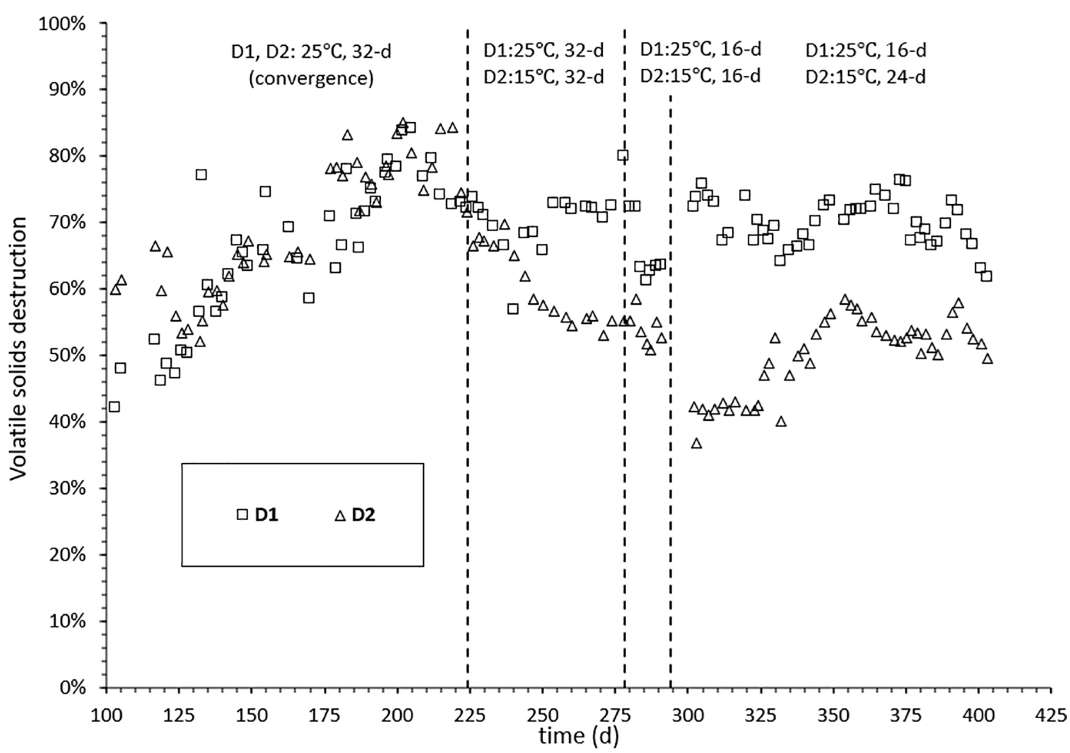

Fig. 2. VSD variation with time for the two digesters at different operating temperatures and HRTs. (a) Both D1 and D2 at 25 °C and 32-d HRT; (b) D1 at 25 °C and 32-d HRT; D2 at 15 °C and 32-d HRT; (c) D1 at 15 °C and 16-d HRT; D2 at 15 °C and 16-d HRT; (d) D1 at 25 °C and 16-d HRT; D2 at 15 °C and 24-d HRT.

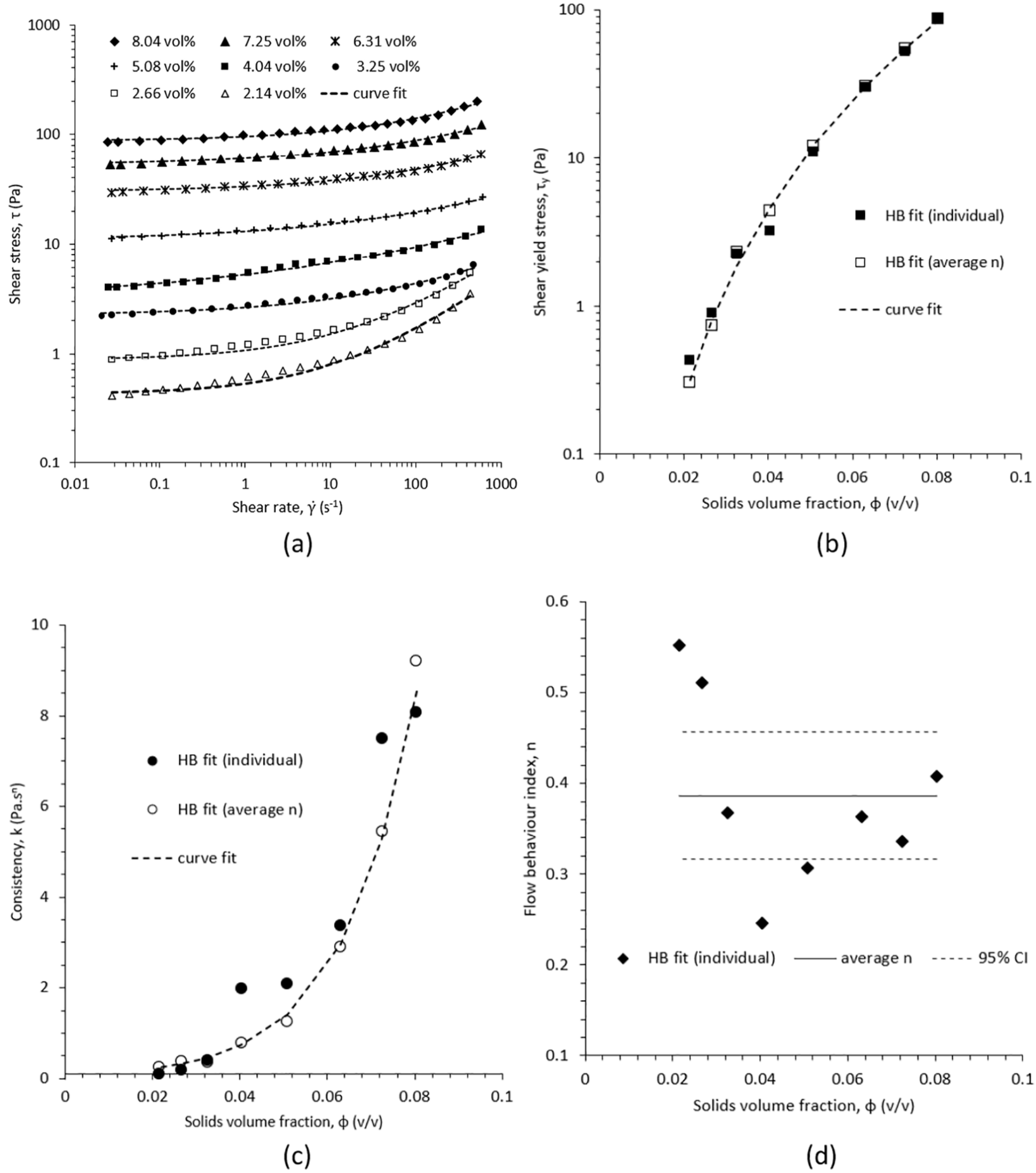

Fig. 3. (a) Experimental rheograms and their Herschel-Bulkley model curve fits, (b) power law fit for shear yield stress (τ_y), (c) exponential fit for consistency (k) and (d) individual and average flow behaviour index for the sample collected from D1 at 25 °C and 32-d HRT.

rheology, which might indicate the concept that the microstructural changes due to digestion drive rheological change.

The power law and exponential fitting parameters for shear yield stress and consistency (τ_1, k_1 and k_2) and flow behaviour index (n) were correlated as continuous functions of VSD. The VSD-parameter relationships with the highest correlation coefficients were τ_1 as a power-law, τ_2 as a constant (fixed at the average of 4.14), k_1 as an exponential function, k_2 linear, and n linear. Parameters for all 53 rheological runs were used to fit this model, and had overall correlation coefficients of $R^2 = 0.97$ for τ_y, $R^2 = 0.85$ for k, and $R^2 = 0.76$ for n. Note that there were only 7 observations for n. However, using average value of Herschel-Bulkley predicted flow behaviour indices from individual fits across solids volume fraction for each sample is justified by the reasonably low (varying ± 0.04 to ± 0.08) 95 % confidence intervals presented in Table 2. This also improves the correlations for predicted shear yield stress (τ_y) and more significantly consistency (k), which is evident in Fig. 3(b) and (c).

The correlations with VSD for the fitting parameters are summarised in Table 3. The fitting parameters provided in Table 3 in combination with those presented in Table 2 can be used to predict the shear rheological behaviour, most importantly viscosity, for anaerobic digested sludge samples across a wide range of solids volume fractions and VSD, which can be subsequently used for mixing system design. Mixing is dependent on the viscosity of the digester content and its design requires a non-Newtonian viscosity prediction model as input (Battista et al., 2016; Singh et al., 2019; Wu, 2010; Wu and Chen, 2008).

For the purpose of comparison, the viscosities of digested sludge samples were calculated at four different solids volume fractions using the optimised models for fitting parameters presented in Table 3 as continuous functions of VSD at a shear rate of 10 s^{-1}, which is an approximation of the

Table 2
Optimised parameters for volume fraction dependence of Herschel-Bulkley model fits for sludge samples collected from the digesters at different operating conditions and range of VSD.*

Digester	Temperature (°C)	HRT (d)	VSD (−)	Shear yield stress, τ_y (Pa) $\tau_y = \tau_1 \phi^{\tau_2}$ $\tau_1 = 10^{\tau_{11}}$		Consistency, k (Pa.s^n) $k = k_1 e^{k_2 \phi}$ $k_1 = e^{k_{11}}$		Flow index, n
				τ_{11}	τ_2	k_{11}	k_2	(−)
D1	25	32	0.69	6.6 ± 0.3	4.2 ± 0.2	−2.8 ± 0.4	61 ± 7	0.39 ± 0.07
	25	16	0.7	6.4 ± 0.4	4.0 ± 0.3	−2.6 ± 0.5	55 ± 10	0.42 ± 0.04
	25	16	0.68	6.5 ± 0.4	4.1 ± 0.3	−2.5 ± 0.4	55 ± 8	0.41 ± 0.08
D2	15	32	0.64	6.2 ± 0.4	3.9 ± 0.3	−3.6 ± 0.4	54 ± 7	0.62 ± 0.06
	15	32	0.55	6.3 ± 0.3	4.2 ± 0.2	−3.5 ± 0.5	54 ± 11	0.54 ± 0.06
	15	24	0.42	6.1 ± 0.2	4.2 ± 0.1	−5.0 ± 0.3	44 ± 6	0.77 ± 0.05
	15	24	0.53	6.3 ± 0.4	4.3 ± 0.3	−4.9 ± 0.3	48 ± 5	0.78 ± 0.07

* Confidence intervals.

average shear rate of 6.8 s^{-1} in industrial digesters reported in literature (Singh et al., 2020). The results are presented in Fig. 4(a) across the experimental VSD range. Comparison of the predicted viscosities (η) for shear rate of 10 s^{-1} at four different solids concentration across a range of VSD (0.4 to 0.7) demonstrated the impact of VSD on the shear behaviour independent of the impact of solids concentration. This illustrates the increase in viscosity for digested sludge at the same solids concentration with the increase of VSD, which may reflect the microstructural changes in the EPS network.

The results as plotted in Fig. 4(a) do not account for the decrease in TSS as a sludge is digested. The models presented above in Tables 2 and 3 can provide the combined impact of VSD and solids volume fraction and can be used to project viscosity at varying effluent solids and VSD. A hypothetical digester which was fed with a feed at VS fraction of 80 % and different feed TS concentrations of 5 %, 6 %, and 7 % were considered. Outlet concentrations were determined for these feeds at different VSD values. The viscosity of sludge inside this hypothetical digester at shear rate of 10 s^{-1} is plotted in Fig. 4(b) to determine the optimum viscosity level where the increase in viscosity caused by VSD is balanced by the decrease in viscosity caused by lower solids concentration due to digestion. There is a shallow optimum generally in the 65 %–80 % VSD range, depending on feed concentration, but this is asymmetric, and in general, it is better to achieve a higher VSD to achieve optimal viscosities, with the increase in viscosity caused by EPS than compensated for by the decreased effluent solids except at very high VSDs. The operational significance of the findings in this paper are that while a maximum VSD should always be targeted, due to both rheology, but also dewaterability (Section 3.3), energy yield, and solids destruction, when designing and operating systems, VSD should also be

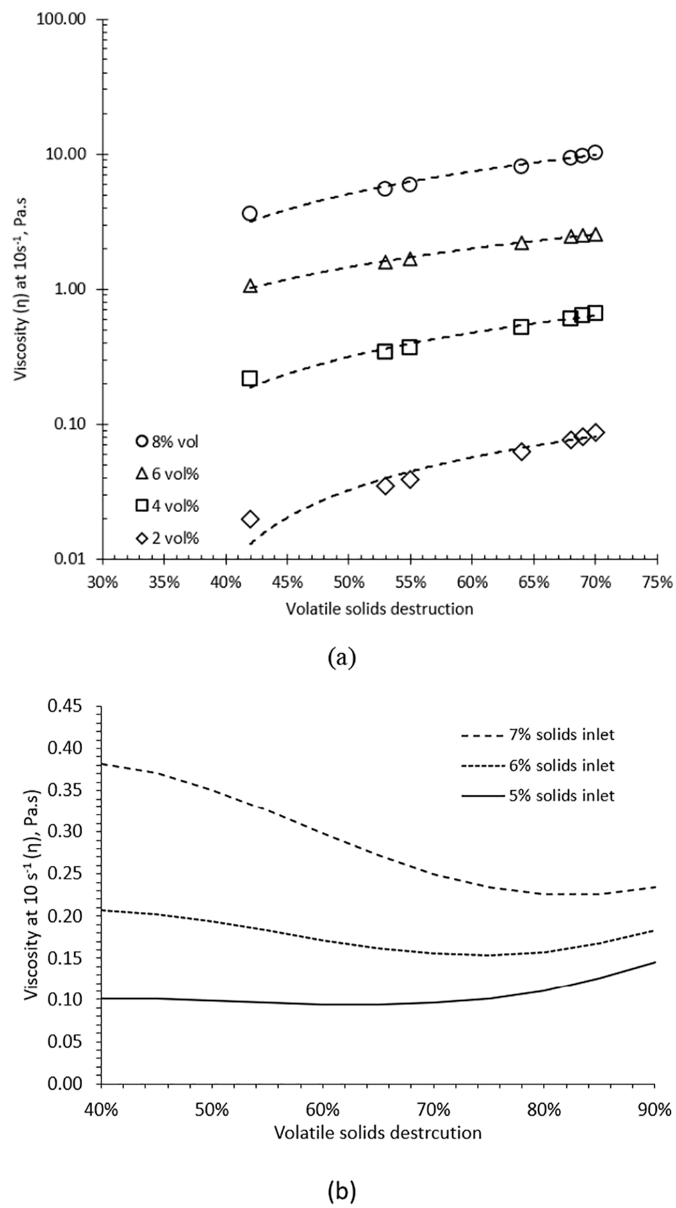

Fig. 4. Variation of predicted viscosities (η): (a) as linear functions of VSD at four different solids volume fractions (ϕ) for 10 s^{-1} shear rate and (b) in a hypothetical digester with three different feed concentration (80 % VS content) showing a shallow optimum viscosity in the range of 65–80 % VSD.

Table 3
Correlations for fitting parameters of shear yield stress, consistency and flow behaviour index with VSD.

Shear yield stress, τ_y (Pa) $\tau_y = \tau_1 \phi^{\tau_2}$ $\tau_1 = 10^{\tau_{11}}$	Consistency, k (Pa·s^n) $k = k_1 e^{k_2 \phi}$ $k_1 = e^{k_{11}}$	Flow index, n
Fitting parameter	Correlation with VSD	Constitutive equation
τ_1	Power	$\tau_1 = 10^{\tau_{21}}\,\mathrm{VSD}^{\tau_{22}}$
		$\tau_{21} = 6.7 \pm 0.4$
		$\tau_{22} = 1.7 \pm 1.5$
τ_2	Constant	4.14 ± 0.10
k_1	Exponential	$k_1 = e^{k_{21}} e^{k_{22}\cdot \mathrm{VSD}}$
		$k_{21} = -9.1 \pm 2.9$
		$k_{22} = 9.2 \pm 4.7$
k_2	Linear	$k_2 = k_{31}\cdot\mathrm{VSD} + k_{32}$
		$k_{31} = 47 \pm 29$
		$k_{32} = 25 \pm 17$
n	Linear	$n = n_1\cdot\mathrm{VSD} + n_2$
		$n_1 = -1.4 \pm 0.9$
		$n_2 = 1.4 \pm 0.6$

Table 4
Fitting parameters for the correlation between hindered settling function and VSD at different operating conditions.
$R(\phi) = r_1\phi^{r_2}$.

Fitting parameter	D1 25 °C 32 HRT	D1 25 °C 16 HRT	D2 15 °C 32 HRT	D2 15 °C 24 HRT
r_1 ($\times 10^{18}$)	2.6	3.49	3.64	4.60
r_2	4.65	4.72	4.69	5.10

considered in determining final sludge rheology, potentially using the model presented in this paper.

3.3. Impact of VSD on solid-liquid separation behaviour of digested sludge

The compressive yield stress, $P_y(\phi)$, from the gravity batch settling test and constant pressure filtration tests were curve fit using a composite power law function (Das et al., 2022b; Usher et al., 2013) presented in Eq. 4 for the four digested sludge samples described in Table S2.7.

$$P_y(\phi) = \begin{cases} P_{y,1}(\phi) = \left(\dfrac{a_1\left(\phi_{cp,1} - \phi\right)\left(b_1 + \phi - \phi_{g,1}\right)}{\left(\phi - \phi_{g,1}\right)}\right)^{-k_1} & : \phi_g \le \phi \le \phi_p \\ P_{y,2}(\phi) = \left(\dfrac{a_2\left(\phi_{cp,2} - \phi\right)\left(b_2 + \phi - \phi_{g,2}\right)}{\left(\phi - \phi_{g,2}\right)}\right)^{-k_2} & : \phi_p \le \phi \le \phi_{cp} \end{cases} \tag{4}$$

The fitting parameters for the curve fit of compressive yield stress as continuous function of solids volume fraction in the range of approximately 2 vol% to 40 vol% ($\phi = 0.02$ to 0.4) are presented in Table S3.3.1. The predicted gel points ($\phi_{g,1}$), which is the minimum solids volume fraction (ϕ) at which the solids form a continuous network to resist applied stress, are also presented in Table S3.3.1. For all four samples, $b_1 = b_2 = 0.01$ and $\phi_{cp,1} = \phi_{cp,2} = 0.63$ were kept constant.

The gel points ($\phi_{g,1}$) were correlated with VSD, which is a power law with a correlation coefficient of $R^2 = 0.9$. Gel point (ϕ_g) indicates the minimum solids volume fraction at which the solids form a network to resist applied stress, and generally a higher gel point corresponds to better solid-liquid separation behaviour. However, except for the sample collected at 15 °C and 24-d HRT (43 % VSD), the gel points for other samples are within ± 0.001 v/v showing no significant variation with VSD.

The experimental hindered settling function values from the batch sedimentation and pressure filtration tests were combined and curve fit as a power law function of solids volume fraction (Eq. 5) across a range of 1 vol% to 40 vol% ($\phi = 0.01$ to 0.4).

$$R(\phi) = r_1\phi^{r_2} \tag{5}$$

The fitting parameters, r_1 and r_2 for the four digested sludge samples are presented in Table 4. Of these two fitting parameters, r_1 is linearly correlated to VSD ($R^2 = 0.92$); however, r_2 is approximately constant (4.68 ± 0.04) for all samples other than the one collected at 15 °C and 24-d HRT.

To understand the impact of digestion on the solid-liquid separation properties at lower solids volume fractions, a steady-state thickener model was used with fixed initial feed concentration, $\phi_0 = 0.005$. The solids flux (q) versus underflow solids volume fraction (ϕ_{uf}) were generated for the four digested sludge samples. The variation of q values at $\phi_{uf} = 0.03$ versus VSD is presented in Fig. 5(a) and the variation of ϕ_{uf} values at $q = 10^{-4}\,\mathrm{t\,m^{-2}\,h^{-1}}$ versus VSD is presented in Fig. 5(b). To understand the impact of decrease in solids volume fraction due to digestion, the inlet feed solids volume fractions for the four cases in the thickener model were varied using the outlet solids volume fractions calculated for a hypothetical digester fed with initial solids volume fraction of 0.01 (80 % VS/TS) and undergoing 43 %, 55 %, 67 % and 76 % VSD, respectively. The initial feed solids volume fractions (ϕ_0) for the thickener model at four VSD values were 0.00656, 0.0056, 0.00464 and 0.00392, respectively. The q values at $\phi_{uf} = 0.03$ versus VSD is presented in Fig. 5(a) and the ϕ_{uf} values at fixed solids flux $q = 10^{-4}\,\mathrm{t\,m^{-2}\,h^{-1}}$ versus VSD is presented in Fig. 5(b).

From Fig. 5(a) and (b), except for the sample at 43 % VSD, there is no variation in the solids flux (q) and underflow solids volume fraction (ϕ_{uf}) profile with VSD obtained for the fixed initial volume fraction ($\phi_0 = 0.005$) and varying initial solids volume fractions calculated using corresponding VSD values. This indicated that the microstructural changes due to digestion and reduction in solids volume fraction due to digestion might have no significant impact on the thickening process.

To investigate the impact of digestion on the solid-liquid separation properties at higher solids volume fractions, a fill and squeeze filtration model was used with fixed initial feed concentration, $\phi_0 = 0.02$. The specific throughput (Q) versus average cake solids volume fraction (ϕ_{av}) were generated for the four digested sludge samples. The variation of Q values at $\phi_{av} = 0.2$ versus VSD is presented in Fig. 6(a) and the variation of ϕ_{av} values at $Q = 10^{-6}$ m/s versus VSD is presented in Fig. 6(b). To investigate the impact of decrease in solids volume fraction due to digestion, the inlet feed solids volume fractions for the four cases in the filtration model were varied using the outlet solids volume fractions calculated for a hypothetical digester fed with initial solids volume fraction of 0.05 (80 % VS/TS) and undergoing 43 %, 55 %, 67 % and 76 % VSD, respectively. The initial feed solids volume fractions (ϕ_0) for the filtration model at four VSD values were 0.0328, 0.028, 0.0232 and 0.0196, respectively. The Q values at $\phi_{av} = 0.2$ versus VSD is presented in Fig. 6(a) and the ϕ_{av} values at fixed specific throughput $Q = 10^{-6}$ m/s versus VSD is presented in Fig. 6(b).

There is no general trend in the specific throughput (Q) versus VSD profile presented in Fig. 6(a), although the increase and decrease in the Q values might result from the interaction of reduction of solids volume fraction and changes in solid-liquid separation properties at different VSD ranges. Similarly, there is no specific trend in the ϕ_{av} with the VSD as presented in Fig. 6(b) for the fixed initial solids volume fraction ($\phi_0 =$

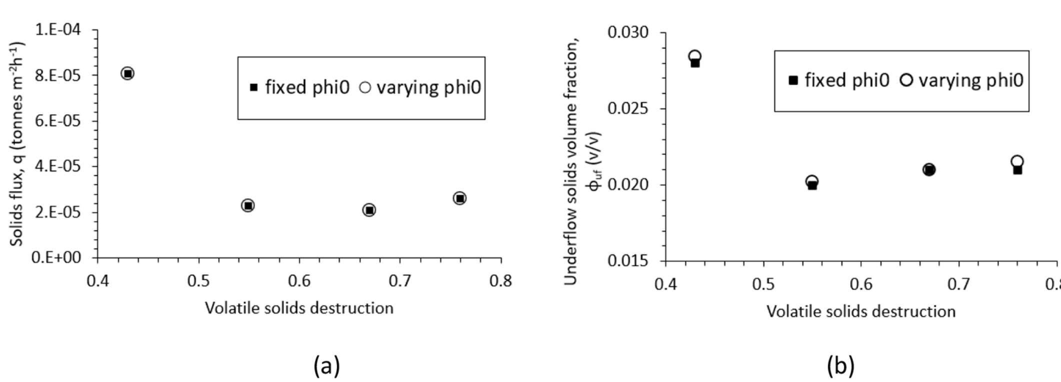

Fig. 5. (a) Solids flux (q) at underflow solids volume fraction $\phi_{uf} = 0.03$ and (b) ϕ_{uf} at $q = 10^{-4}\,\mathrm{t\,m^{-2}\,h^{-1}}$ versus VSD profiles for digested sludge samples for fixed initial solids volume fraction $\phi_O = 0.005$ and varying initial solids volume fractions calculated using corresponding VSD.

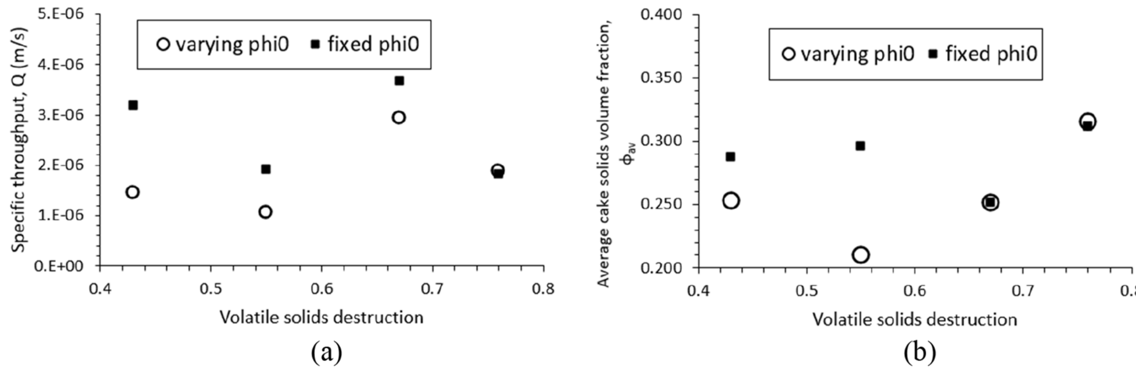

Fig. 6. (a) Specific throughput (Q) at average cakes solids volume fraction $\phi_{av} = 0.2$ and (b) ϕ_{av} at $Q = 10^{-6}$ m/s versus VSD for digested sludge samples in a membrane filter press for fixed initial solids volume fraction $\phi_0 = 0.02$ and varying initial solids volume fractions calculated using corresponding VSD.

0.005). In contrast, in the ϕ_{av} versus VSD profile corresponding to the decreasing initial solids volume fractions accounting for digestion of organic matter, there is an initial decrease in ϕ_{av} from 43 % to 55 % VSD and then a gradual increase in ϕ_{av} from 55 % to 76 % VSD, which indicated the impact of digestion on the filtration process. As VSD is inversely related to the VS/TS ratio of the digested sludge, this trend was similar to that observed by Skinner et al. (2015) where an increase of final cake solids volume fractions with the increase of VSS% in the sludge.

4. Conclusions

The operating temperature had significant impact on VSD and the anaerobic digestion process appeared significantly faster at 25 °C compared to 15 °C, with the hydrolysis constant increasing from 0.09 d^{-1} at 15 °C to 0.65 d^{-1} at 25 °C. In contrast, HRT had non-significant impact on VSD.

Herschel-Bulkley model fitting parameters, shear yield stress (τ_y), consistency (k) and flow behaviour index (n), were predicted as continuous functions of both solids volume fraction and VSD. At the same solids concentration, sludge viscosity (η) at the shear rate of $10s^{-1}$ increased 1.3 to 3.3 times as VSD increased from 43 % to 70 %. Analysis of a hypothetical digester indicated that there is an optimum VSD range 65–80 % VSD where the increase in viscosity due to the higher VSD is balanced by the reduction in solids concentration. However, this is asymmetric, and it is better to extend the HRT to achieve higher VSD to achieve optimal viscosities.

The solid-liquid separation properties, compressive yield stress, $P_y(\phi)$, hindered settling function, $R(\phi)$ and solids diffusivity, $D(\phi)$, were predicted as continuous functions of solids volume fractions. To compare the solid-liquid separation behaviour of the digested sludge samples at lower and higher solids concentrations, a thickener model and a filtration model was used, respectively. No specific trend in the solids flux (q), underflow solids volume fraction (ϕ_{uf}) and specific throughput (Q) with VSD was observed in the thickening and filtration processes. However, there was a general increase of average filter cake solids volume fraction (ϕ_{av}) from 21 % to 31 % with an increase of VSD from 55 % to 76 %, indicating better solid-liquid separation properties.

CRediT authorship contribution statement

Tanmoy Das – All experimental work, data analysis and writing first draft of the manuscript; Shane P. Usher – Interpretation of results; Damien J. Batstone – Analysis and interpretation of results, and review of manuscript; Maazuza Othman – Interpretation of results and review of manuscript; Catherine A. Rees – Provided industrial input and review of manuscript; Anthony D. Stickland – Corresponding author, experimental planning, concept design, interpretation of results, and review of manuscript; Nicky Eshtiaghi – Corresponding author, experimental planning, concept design, interpretation of results and review of manuscript.

Data availability

Data is available upon request.

Declaration of competing interest

The authors declare that they have no competing interests.

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Appendix A. Supplementary data

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