Substrate and operational conditions as regulators of fluid properties in full-scale continuous stirred-tank biogas reactors – implications for rheology-driven power requirements

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ABSTRACT

Understanding fluid rheology is important for optimal design and operation of continuous stirred-tank biogas reactors (CSTBRs) and is the basis for power requirement estimates. Conflicting results have been reported regarding the applicability of total solid (TS) and/or total volatile solid (TVS) contents of CSTBR fluids as proxies for rheological properties. Thus, the present study investigates relationships between rheological properties of 12 full-scale CSTBR fluids, their substrate profiles, and major operational conditions, including pH, TS and TVS contents, organic loading rate, hydraulic retention time, and temperature. Rheology-driven power requirements based on various fluid characteristics were evaluated for a general biogas reactor setup. The results revealed a significant correlation only between the rheological fluid properties and TS or TVS contents for sewage sludge digesters and thermophilic co-digesters (CD), but not for mesophilic CD. Furthermore, the calculated power requirements for pumping and mixing, based on the various fluid characteristics of the studied CSTBRs, varied broadly irrespective of TS and TVS contents. Thus, this study shows that the TS and/or TVS contents of digester fluid are not reliable estimators of the rheological properties in CSTBRs digesting substrates other than sewage sludge.

Key words | anaerobic digestion, biogas, continuous stirred-tank biogas reactor, power requirement, rheology, viscosity

NOMENCLATURE

AD | Anaerobic digestion
CD | Co-digestion
CSTBR | Continuous stirred-tank biogas reactor
FIW | Food industry waste
OFMSW | Organic fraction of municipal solid waste
PBSS | Primary and biological sewage sludge
PCA | Principal component analysis
SHW | Slaughterhouse waste
SS | Sewage sludge

β | Correction factor for non-uniformity of flow [dimensionless]
ρ | Fluid density [kg/m³]
η₂₀ | Apparent viscosity at shear rate 20 s⁻¹ [mPa·s]
ηₘₐₘ | Limit viscosity (reported at shear rate 800 s⁻¹) [mPa·s]
D | Impeller diameter [m]
γ | shear rate [s⁻¹]
τ | shear stress [Pa]
τ₀ | Yield stress [Pa]
HRT | Hydraulic retention time [days]
K | Consistency index [Pas]
m | Mass flow rate [kg/s]
n | Flow behaviour index [dimensionless]
**INTRODUCTION**

Application of anaerobic digestion (AD) of organic wastes and production of biogas, containing methane as a renewable energy carrier, is widely recognized (Kampman et al. 2016). Continuous stirred-tank biogas reactors (CSTBRs) are extensively used for AD processes, where effective mixing plays a central role in sustaining stable and high-yield performance. Consequently, a substantial share of the energy required for CSTBR operation is spent on mixing, contributing to a considerable part of the biogas production costs (Lindmark et al. 2014). Thus, the mixing needs to be efficient, implying that it should use as little energy as possible, while allowing a homogeneous distribution of the substrate and heat as well as a complete utilization of CSTBR operational volume (Lindmark et al. 2014). The amount of energy required to reach this level of efficient mixing is linked to rheological properties of the reactor fluid and the scale of the system (Nienow 2014), in addition to energy needs associated with, for example, friction or inherent inefficiencies of the electric motors. Furthermore, non-Newtonian fluids impose additional challenges for maintaining uniform shear distribution through the bulk of the reactor fluid (Nienow 2014). For example, thixotropic fluids, which build up internal structures and gel-like consistency in the absence of shear stress (Seyssiecq et al. 2005), may partially contribute to the increased power demand at the start of mixing in CSTBR systems (Lindmark et al. 2014). In addition, inadequate mixing, damaged mixing equipment, foaming, and sludge bulking may occur when fluid behaviour in a reactor changes (Nordberg & Edström 2005; Lindorfer & Demmig 2016).

A variety of mixing systems with different advantages and disadvantages in relation to energy consumption and mixing efficiency are applied in CSTBRs. In short, the mixing systems can be mechanical, hydraulic, or pneumatic in their design, and reactors may be mixed continuously or intermittently (Lindmark et al. 2014). The mixing energy requirements for all types of systems are related to the need for optimal mixing turnover rates and particle suspension, which directly depend on the fluid’s rheological characteristics as well as the amount, size, and density of the particles (Nienow 2014). Accordingly, evaluations of fluid rheological properties are central for the design and optimization purposes. Comprehensive research has been carried out on rheological characterization of primary and biological sewage sludge (PBSS) in wastewater treatment systems, including the reactor fluids of their AD units. However, information on rheological properties of CSTBR fluids digesting substrates other than PBSS is relatively scarce (Mbaye et al. 2014). This information has an immense practical relevance, considering that around 72% of the biogas produced within the European Union during 2014 was produced by digesting substrates other than PBSS (Kampman et al. 2016).

For AD processes digesting PBSS, total solid (TS) and/or total volatile solid (TVS) contents are frequently reported as the most important parameters determining the rheological properties (e.g. Battistoni 1997; Baudez et al. 2011). Similar results have also been found for reactors digesting other types of organic wastes, such as agricultural wastes and the organic fraction of municipal solid waste (OFMSW) (e.g. Battistoni 1997; Mbaye et al. 2014). Particularly viscosity and/or yield stress have been found to be positively correlated to TS and/or TVS (e.g. Battistoni 1997; Baudez et al. 2011; Mbaye et al. 2014), which is why these parameters are suggested as general proxies for fluid behaviour in AD systems. In contrast, a number of studies have shown that parameters such as temperature, degree of degradation, substrate composition, and particle size have a more prominent effect on rheological behaviour of reactor fluids compared to the TS and TVS contents. For example, Ometto et al. (2017) showed that thermophilic reactors co-digesting PBSS and macro-algae had lower apparent and limit viscosities than their mesophilic counterparts and that changes in TS over time did not correlate with changes of limit or apparent viscosities. On a similar note Monteiro (1997) showed that the degree of degradation of PBSS during AD resulted in a decrease of the apparent viscosity and yield stress. They also observed that variations in TS did not cause changes in viscosity. Finally,
Nordberg & Edström (2005) added another dimension to these interactions by demonstrating that substrate composition (i.e., different ratios between ley crop silage and the organic fraction of municipal solid waste) and particle size of the substrate at a constant TS lead to significant differences in rheological properties of the resulting CSTBR fluids. More specifically, lower contents of ley crop silage and smaller particle sizes resulted in lower apparent viscosities of the digester fluids. It is therefore unclear whether the TS and/or TVS contents represent the CSTBR fluid rheology in AD processes, signifying the need for a better understanding of the relationship of the rheological characteristics with substrate composition and operational conditions.

Accordingly, the objective of the present study was to elucidate possible relationships of the rheological properties of a range of full-scale CSTBRs with their substrate profile and main operational conditions, including organic loading rate (OLR), hydraulic retention time (HRT), pH, TS, TVS, and temperature. To assess the rheological properties, we determined shear stress at different shear rates, apparent and limit viscosities, as well as fluid behaviour by rheological modelling. Pairwise correlations and principal component analysis (PCA) were used to evaluate potential relationships between rheological and operational parameters. Finally, the effect of rheological properties of these fluids on the power requirements for mixing and pumping were compared for a hypothetical reactor setup in order to evaluate the contribution of rheology-driven power requirement based on fluid characteristics of the studied AD fluids.

MATERIALS AND METHODS

Sample sources and chemical characterization

Twelve CSTBRs were sampled during an initial campaign, seven of which were re-sampled 9 months later. The reactors were either PBSS-based digesters (SS; designated SS1*, SS2T and SS4) or co-digesters (CD; designated CD2T, CD3T, CD4, CD5a, CD5b, CD7a, CD7b, CD8T and CD11, where T stands for thermophilic). Eight of the CSTBRs were operated under mesophilic (36–38 °C), and four under thermophilic (52–55 °C) conditions. Information on operational conditions and substrate compositions of the CSTBRs is presented in Table 1. The substrate of reactor SS1* included food industry waste (FIW) in addition to PBSS. The asterisk signifies the different substrate profile of SS1* compared to SS2T and SS4, which received only PBSS as the substrate. Substrates of the CD reactors included different combinations of FIW, slaughterhouse waste (SHW), OFMSW, fodder residues, fat, starch, and manure.

The pH of the reactor liquid was determined using a PHM93 meter (Radiometer, Copenhagen, Denmark) according to the Swedish standard method SS-12176. The TS content (in % of wet weight) was analysed by drying samples at 105 °C for 20 h and the TVS (in % of wet weight) was measured after heating the dried samples in a furnace at 550 °C for 2 h according to the Swedish standard method SS-028113.

Rheological characterization

A shear rate-controlled Searle-type rotational rheometer (RheolabQC SN80609650) with a CC27-SN19237 measuring system and a C-LTD80/QC cell, coupled to Rheoplus software (Anton Paar, Ostfildern, Germany), was used for rheological characterization of the samples. The measuring system consisted of concentric smooth surface cylinders with an inner diameter of 27 mm, outer diameter of 29 mm, and height of 40 mm. Samples (17 ml) were analysed at 37 ± 0.2 °C, 52 ± 0.2 °C or 53 ± 0.2 °C corresponding to operational temperatures of the CSTBRs (Table 1). We minimized extensive sample pre-treatments before rheological analyses to avoid perturbation of the original rheological characteristics of the reactor fluids, e.g., through alteration of parameters, such as floc structure or concentrations of extracellular polymeric substances, which may affect the rheology (More et al. 2014). To account for inhomogeneity of the samples with respect to presence of particles and flocs, the rheological analyses were performed on three to four replicates. Samples were analysed at the day of sampling or after storage at 4 °C overnight. A previous assessment of the effect of overnight sample storage on fluid properties showed no significant changes in the rheological parameters (data not shown).

The rheological measurements followed a three-step protocol adopted from Pevere et al. (2007), where the shear rate was (1) increased linearly from 0 to 800 s−1 over a period of 800 s, with measuring points every 10 s (referred to as up-curve), (2) maintained at 800 s−1 for 300 s, with measuring points every 30 s, and (3) decreased linearly from 800 to 0 s−1 over a period of 800 s, with measuring points every 10 s (referred to as down-curve). The rheological parameters including apparent viscosity (η) and limit viscosity (ηlim) were determined based on the relationship between shear rate and shear stress (rheogram). Apparent viscosity at a shear rate of 20 s−1, η20 [Pas], was selected for our analyses, as AD fluids in CSTBRs are exposed
to local shear rates in the range of 0–20 s⁻¹ throughout most of their volume (Sindall et al. 2013). Limit viscosity, ηlim [Pas], was estimated at a shear rate of 800 s⁻¹, at which the apparent viscosity of all fluids remained constant in relation to the applied shear stress. When ηlim and ηυ were compared between the two sampling occasions, a t-test was used to evaluate the differences between the measurements.

### Determination of fluid type

To assess the fluid types of the samples, three models including Herschel–Bulkley (τ = τ₀ + Kγ²), Ostwald (τ = Kγ), and Bingham (τ = τ₀ + Kγ) were used according to Pevere et al. (2006) and Seyssiecq et al. (2005). The parameters τ and γ are shear stress [Pa] and shear rate [s⁻¹], respectively, τ₀ is yield stress (the shear stress required for the material to start flowing) [Pa], K consistency index (the general slope of the curve) [Pas], and n flow behaviour index [dimensionless]. The latter signifies if the fluid exhibits any shear thinning (n < 1) or shear thickening (n > 1), i.e. dilatant behaviour (Ratkovich et al. 2013). The model for each fluid was selected based on goodness-of-fit (R² values), and the distribution of least-square fitting residuals (i.e. by evaluating if the residuals are independent and normally distributed). When possible, a model with fewer parameters was selected in order to avoid ‘over fitting’. For the same reason, we avoided the use of other common rheological models with higher numbers of adjustable parameters (e.g. Carreau or Cross models) and degrees of freedom for curve fitting purposes (cf. Ratkovich et al. 2013).

Since the three-step protocol generates two sets of data over the same shear rates (i.e. the so-called ‘up’ and ‘down’ curves from intervals 1 and 3; Figure S1 in the supplementary information, available with the online version of this paper), the modelling was performed on both datasets. The ‘down’ curves of the rheograms represent the fluid behaviour under which the thixotropic structures are completely disrupted and simulate the behaviour after exposure to prolonged mixing. The ‘up’ curves of the rheograms represent the fluid behaviour during the start-up of mixing. The modelling results of both intervals as well as the possible presence of a hysteresis area between the two curves were used to ascertain the fluid type. PCA was carried out to obtain an overview of the dataset and evaluate possible multivariate relationships between the fluid behaviour indices (from modelling performed on the ‘down’ curves) and overall fluid properties.

### Calculations of power requirement for pumping and mixing

The modelling parameters, n, K, and τ₀, were used to estimate the part of power requirements for pumping and mixing associated with fluid rheological properties in a hypothetical CSTBR (i.e. without taking friction or other losses into account), following the procedure developed by Farno et al. (2017) with some modifications. The specifications of the hypothetical CSTBR as well as details on power requirement calculations are presented in the supplementary information (Figure S2, available online). In short, the CSTBR configuration was assumed to be a
cylinder with a working volume of 1,000 m³ and a diameter equal to the height of the fluid phase (10.84 m). The stirrer type was assumed to be a down-pumping 45° pitched blade impeller, centrally positioned at one third of fluid height (3.58 m). The impeller diameter (D) was assumed to be equal to 0.4 times the reactor diameter (D = 4.34 m), which is within the interval suggested for calculating the impeller power number by Chapple et al. (2002).

It was assumed that the reactor has a liquid recirculation loop through heat exchangers and that the substrate is continuously added into the recirculation loop at a rate needed to achieve an HRT of 27 days, representing the average HRT of CSTBRS in this study. The recirculation pipe was assumed to have a diameter of 100 mm. Data from the ‘down-curve’ of the rheograms were used for the calculations to account for the power requirement for pumping/mixing of the fluid under prolonged mixing conditions.

The transition point from laminar to turbulent flow was assumed as the optimal condition for pumping since it represents the minimal power required to avoid the creation of stagnant zones and laminar flow particle settling, which may otherwise lead to pipe blockage (Slater 2008; Eshtiaghi et al. 2012). Accordingly, the critical pumping velocity, \( V_c \) [m/s], for the laminar–turbulent transition can be defined by the following equation (Myers et al. 2017):

\[
V_c = \frac{2n + 1}{n + 1} \sqrt{\frac{Re_5 \tau_0}{8 \rho}}
\]

where \( n \) is the flow behaviour index [dimensionless], \( \tau_0 \) the yield stress [Pa], and \( \rho \) is fluid density [kg/m³]. For simplicity, the fluid density was assumed to be similar to the density of water (i.e., 1,000 kg/m³) due to relatively low TS values in AD fluids (<6% of wet weight in this study) (Eshtiaghi et al. 2012). Parameter \( Re_5 \) represents the Slatter laminar to turbulent Reynolds number, where the transition to turbulent conditions is predicted at \( Re_5 = 2,100 \) (Slater 2008). For fluids, where \( \tau_0 = 0 \) was calculated based on the rheogram (i.e., \( V_c = 0 \) m/s), we assumed that the minimum flow velocity (\( V_{min} \)) is equal to continuous inflow rate of 0.06 m/s, which is needed to maintain 27 days HRT in a 1,000 m³ vessel through a 100 mm diameter pipe. Either \( V_c \) or \( V_{min} \) were then used to calculate the pumping power requirement, \( P_p \) [W], according to Farno et al. (2017).

\[
P_p = \frac{m V^2}{2 \beta}
\]

where \( \beta \) is the correction factor for non-uniformity of flow ([dimensionless]; calculations presented in the supplementary information) (Farno et al. 2017) and \( m \) is mass flow rate [kg/s]. The latter was calculated for each fluid using pipe diameter and critical velocity. The mixing speed to achieve a turbulent mixing regime, \( N_{it} \) [rpm], was assumed as the required criterion for mixing and was calculated according to the following equation (Metzner & Otto 1957):

\[
N_{it} = 2 - \frac{n}{8} \left( \frac{n}{18n + 1} \right) D^2 \rho
\]

where \( n \) is the flow behaviour index, \( K \) the consistency index, \( D \) the diameter of the impeller [m], and \( \rho \) the fluid density [kg/m³]. Parameter \( Re_m \) is the Reynolds number [dimensionless] for mixing of non-Newtonian fluids at which the transition to turbulent flow occurs and was assumed to be 20,000 (Chapple et al. 2002). It is important to highlight that Equation (3) was developed based on experiments on pseudo-plastic fluids (i.e., \( n < 1 \)). Application of Equation (3) for dilatant fluids (\( n > 1 \)) probably leads to inaccurate results, and particularly so when \( n \) approaches or surpasses 2. For comparative purposes, the dilatant fluids in this study were approximated to be Newtonian (\( n = 1 \)) by using their Bingham model-derived \( K \).

The minimum power requirement [W] for mixing was calculated as \( P_m = P_0 \rho N_{it}^2 D^5 \) (Farno et al. 2017), where \( P_0 \) represents the power number [dimensionless], calculated for the hypothetical CSTBR according to Chapple et al. (2002), \( \rho \) is fluid density [kg/m³], \( N_{it} \) [s⁻¹] impeller speed for achieving turbulent mixing, and \( D \) [m] is the impeller diameter. More details on assumptions and power requirement calculations are found in the supplementary information.

**RESULTS AND DISCUSSION**

**Digester liquid and substrate characteristics**

The pH of the digester liquids ranged between 7.2 and 8.1. The lowest pH values were observed for SS reactor liquids (pH < 7.5), while the pH values for CD reactor liquids were generally higher (pH > 7.6; Table 2). The TS and TVS contents were in the range of 1.8 to 5.3% and 1.0 to 3.8% of wet weight, respectively. Reactors CD11 and CD3T, with SHW as their major substrate, had the highest TS values (5.3 and 5.0% of wet weight). The lowest TS of 1.8% of wet weight was observed for SS1's. Comparisons between the sampling occasions showed that the TS...
contents remained relatively constant for the majority of the reactors (Table 2). However, the TS content was lower for SS1’s and CD5as, while it was higher for SS4s as compared to the samples taken from these reactors during the first campaign. The TS and TVS contents of the substrate mixtures were higher than the corresponding digested liquid (Table S1 in the supplementary information, available with the online version of this paper). The TVS contents of all substrates were between 4.4 and 8.4% of wet weight, except for the PBSS for SS2T (2.8% of wet weight).

### Viscosity of the digester fluids

Both \( \eta_{20} \) and \( \eta_{\text{lim}} \) values were generally higher for substrate samples compared to their corresponding reactor fluids (Table S1 and Table 2), which indicates an overall thinning effect of AD on fluid properties of organic waste substrates. Variations between replicate rheograms were also higher for substrate samples than CSTBR samples, probably due to larger particle sizes, which may have interfered with the rheological measurements. For CSTBR fluids, \( \eta_{\text{lim}} \) measurements yielded consistent results between replicates, with only two samples (CD7a and CD5as) exhibiting relative standard deviation higher than 50% (Table 2). The highest \( \eta_{\text{lim}} \) values were observed for reactor fluids of SS4s (14 ± 0.2 mPa·s), CD11 (15 ± 1.5 mPa·s), and CD4s (10 ± 0.5 mPa·s), while the lowest \( \eta_{\text{lim}} \) values were observed for CD2T, CD8T, and CD8Ts, (3.8 ± 0.4, 3.6 ± 0.3, and 3.5 ± 0.3 mPa·s respectively; Table 2).

The \( \eta_{20} \) values were not consistent in replicate measurements, particularly for the CD systems, signifying the effect of reactor fluid inhomogeneity on rheological measurements at low shear rates. Overall, the replicate \( \eta_{20} \) measurements for 10 samples demonstrated relative standard deviations >50% (Table 2). The \( \eta_{20} \) values represent fluid viscosity at a shear rate of 20 s\(^{-1}\) in the up-curve of the rheograms, under which aggregates and flocs in the samples are not exposed to long-term shear, contributing to inconsistencies in measurements. It is also argued that the up-curves of rheograms may actually reflect the gradual breakdown of

### Table 2 | Characteristics of reactor liquids including pH, total solid (TS), total volatile solid (TVS), apparent viscosity and limit viscosity

<table>
<thead>
<tr>
<th>Reactor</th>
<th>pH</th>
<th>TS (% of wet weight)</th>
<th>VS (% of TS)</th>
<th>TVS (% of wet weight)</th>
<th>( N_{\text{app}} )</th>
<th>( \eta_{20} ) (mPa·s)</th>
<th>( \eta_{\text{lim}} ) (mPa·s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SS1</td>
<td>7.2 ± 0.0</td>
<td>3.7 ± 0.1</td>
<td>31 ± 0.2</td>
<td>1.2 ± 0.0</td>
<td>2</td>
<td>45 ± 6</td>
<td>5.9 ± 0.4</td>
</tr>
<tr>
<td>SS1’s</td>
<td>7.2 ± 0.0</td>
<td>1.8 ± 0.0</td>
<td>53 ± 0.3</td>
<td>1.0 ± 0.0</td>
<td>2</td>
<td>13 ± 16</td>
<td>4.6 ± 0.1</td>
</tr>
<tr>
<td>SS2T</td>
<td>7.3 ± 0.1</td>
<td>2.4 ± 0.0</td>
<td>66 ± 0.2</td>
<td>1.6 ± 0.0</td>
<td>2</td>
<td>42 ± 4</td>
<td>5.5 ± 0.1</td>
</tr>
<tr>
<td>SS4</td>
<td>7.5 ± 0.1</td>
<td>3.0 ± 0.0</td>
<td>61 ± 0.0</td>
<td>1.8 ± 0.0</td>
<td>3</td>
<td>83 ± 8</td>
<td>8.2 ± 0.5</td>
</tr>
<tr>
<td>SS4s</td>
<td>7.3 ± 0.0</td>
<td>3.9 ± 0.0</td>
<td>62 ± 0.0</td>
<td>2.4 ± 0.0</td>
<td>4</td>
<td>150 ± 14</td>
<td>14.4 ± 0.2</td>
</tr>
<tr>
<td>CD2T</td>
<td>8.0 ± 0.0</td>
<td>2.7 ± 0.0</td>
<td>59 ± 0.1</td>
<td>1.6 ± 0.0</td>
<td>2</td>
<td>8.2 ± 3</td>
<td>3.8 ± 0.4</td>
</tr>
<tr>
<td>CD2Ts</td>
<td>8.0 ± 0.0</td>
<td>3.6 ± 0.0</td>
<td>51 ± 0.1</td>
<td>1.8 ± 0.0</td>
<td>3</td>
<td>nd</td>
<td>4.7 ± 0.2</td>
</tr>
<tr>
<td>CD3T</td>
<td>7.9 ± 0.0</td>
<td>5.0 ± 0.3</td>
<td>75 ± 1.6</td>
<td>3.8 ± 0.2</td>
<td>3</td>
<td>4.6 ± 2.7</td>
<td>5.3 ± 0.3</td>
</tr>
<tr>
<td>CD4</td>
<td>7.8 ± 0.0</td>
<td>3.9 ± 0.1</td>
<td>72 ± 0.3</td>
<td>2.8 ± 0.1</td>
<td>2</td>
<td>3.8 ± 24</td>
<td>8.1 ± 0.6</td>
</tr>
<tr>
<td>CD4s</td>
<td>7.9 ± 0.0</td>
<td>3.9 ± 0.0</td>
<td>71 ± 0.0</td>
<td>2.8 ± 0.0</td>
<td>3</td>
<td>110 ± 21</td>
<td>10.4 ± 0.5</td>
</tr>
<tr>
<td>CD5a</td>
<td>8.0 ± 0.0</td>
<td>4.0 ± 0.1</td>
<td>72 ± 0.1</td>
<td>2.9 ± 0.1</td>
<td>2</td>
<td>160 ± 200</td>
<td>9.1 ± 3.7</td>
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<tr>
<td>CD5as</td>
<td>7.7 ± 0.0</td>
<td>5.5 ± 0.0</td>
<td>70 ± 0.6</td>
<td>2.5 ± 0.0</td>
<td>2</td>
<td>1,300 ± 1,800</td>
<td>9.9 ± 6.4</td>
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<tr>
<td>CD5b</td>
<td>na</td>
<td>4.0 ± 0.1</td>
<td>72 ± 0.6</td>
<td>2.9 ± 0.1</td>
<td>2</td>
<td>40 ± 9</td>
<td>5.8 ± 0.7</td>
</tr>
<tr>
<td>CD7a</td>
<td>7.9 ± 0.0</td>
<td>3.9 ± 0.0</td>
<td>61 ± 0.3</td>
<td>2.4 ± 0.0</td>
<td>3</td>
<td>1,000 ± 1,800</td>
<td>28 ± 20</td>
</tr>
<tr>
<td>CD7as</td>
<td>7.9 ± 0.0</td>
<td>4.2 ± 0.0</td>
<td>59 ± 0.4</td>
<td>2.5 ± 0.0</td>
<td>2</td>
<td>150 ± 100</td>
<td>9.5 ± 0.7</td>
</tr>
<tr>
<td>CD7b</td>
<td>na</td>
<td>4.1 ± 0.0</td>
<td>61 ± 0.3</td>
<td>2.5 ± 0.0</td>
<td>2</td>
<td>180 ± 150</td>
<td>10 ± 0.1</td>
</tr>
<tr>
<td>CD8T</td>
<td>7.6 ± 0.0</td>
<td>2.1 ± 0.0</td>
<td>69 ± 0.2</td>
<td>1.5 ± 0.0</td>
<td>2</td>
<td>59 ± 66</td>
<td>3.6 ± 0.3</td>
</tr>
<tr>
<td>CD8Ts</td>
<td>7.7 ± 0.0</td>
<td>2.0 ± 0.0</td>
<td>66 ± 0.1</td>
<td>1.3 ± 0.0</td>
<td>3</td>
<td>nd</td>
<td>3.5 ± 0.3</td>
</tr>
<tr>
<td>CD11</td>
<td>8.1 ± 0.0</td>
<td>5.3 ± 0.0</td>
<td>71 ± 0.1</td>
<td>3.8 ± 0.0</td>
<td>2</td>
<td>340 ± 360</td>
<td>13 ± 1.5</td>
</tr>
</tbody>
</table>

\( N_{\text{app}} \) and \( N_{\text{lim}} \) denote numbers of replicate samples used for obtaining apparent and limit viscosity values, respectively. na stands for not analysed and nd stands for not detected (measurement was within the measuring noise).

\( ^a \) Reactors SS1, SS4, CD2T, CD4, CD5a, CD7b, and CD8 were sampled at two occasions, shown by letter ‘s’ after the reactors’ names.

\( ^b \) Apparent viscosity at shear rate if 20 s\(^{-1}\), using data from the first measuring interval of the rheogram (i.e. ‘up’ curve).

\( ^c \) Limit viscosity, measured at shear rate of 800 s\(^{-1}\).
elastic-solid structures due to incomplete shear across the measuring gap at low shear ranges rather than actual fluid rheology (Baudez 2006). The \( \eta_{20} \) measurements with relative SD below 50% ranged from 0 to 150 mPa·s (Table 2). The highest reliable \( \eta_{20} \) values were measured for SS4 (150 ± 14 mPa-s), SS4s (110 ± 21 mPa-s) and CD4s (85 ± 8.1 mPa-s), while the lowest values were observed for fluids from thermophilic reactor samples CD2T (8.2 ± 3.1 mPa-s), as well as CD2Ts, and CD8T with \( \eta_{20} \) values within the baseline noise of the measurements. Thus, the viscosities of the digester fluids from the thermophilic CD processes were the lowest among the samples, probably due to the fact that the generally higher degradation efficiencies of thermophilic processes lead to smaller and less abundant particles and polymers (Appels et al. 2008). Our results indicate that thermophilic reactor operation may also have less viscous reactor fluids as a side effect.

Pairwise correlation analyses did not result in any conclusive relationships between the operational parameters (OLR, HRT, pH, TS, and TVS) and fluid viscosity (\( \eta_{20} \) and \( \eta_{\text{lim}} \)) when data from all samples were considered for the analysis (Figure 1(a) and Figure S4, supplementary information, available online). It is therefore evident that none of the operational parameters can be used as a single proxy for fluid viscosity. However, we observed that the TS and TVS significantly correlated with \( \eta_{\text{lim}} \) values \( (r = 0.67, p = 0.012 \) and \( r = 0.93, p < 0.001 \) respectively) for SS reactor fluids (SS1, SS1s, SS2T, SS4, and SS4s). A correlation was also observed between \( \eta_{\text{lim}} \) and TS or TVS \( (r = 0.93, p < 0.001 \) and \( r = 0.84, p < 0.001 \)) of the thermophilic CD reactor fluids. No such correlations were observed between TS or TVS and \( \eta_{\text{lim}} \) for the mesophilic CD reactor fluids \( (r = 0.01, p = 0.976 \) and \( r = -0.16, p = 0.462, \) respectively) (Figure 1(b)). Similar to \( \eta_{\text{lim}} \), a positive and significant correlation was observed between \( \eta_{20} \) and TS \( (r = 0.79, p = 0.001 \) for SS, but not for CD or CDT reactor fluids \( (r = -0.17, p = 0.464 \) and \( r = -0.19, p = 0.528, \) respectively). The correlation between \( \eta_{20} \) and TVS was even stronger for SS reactor fluids \( (r = 0.95, p < 0.001). \) Thus, the relationship between fluid viscosity and operational parameters appears
to be different for PBSS digesters, thermophilic CD, and mesophilic CD. Particularly, the CSTBR substrate and operational temperature are important factors, determining the suitability of TS and TVS as proxies of fluid rheology. Based on the correlation analyses, it is clear that TS and TVS contents do not represent the rheological characteristics of CSTBR fluids. Although rheological parameters may be correlated to the TS and TVS of the SS reactor fluids, such correlation cannot be implemented for assessments of reactor fluids in CD systems. For the CD reactors operated under mesophilic conditions, the rheological behaviour of the reactor fluids seemed to vary with the substrate profiles and operational conditions rather than with the TS and TVS contents of the reactor fluids. This was also pointed out by Nordberg & Edström (2005), who studied the effects of ratios between ley crop silage and OFMSW and particle size of the substrate and found that both parameters play a more important role on the rheology of the fluid in the reactor than TS content. It should be noted that the TS and TVS contents of the digesters in our study were all lower than 5.3% and 3.8%, respectively. Further research may be needed to assess the applicability of these results for CSTBRs with TS and VS contents higher than these values.

Samples from the two sampling occasions differed significantly in \( \eta_{20} \) values for reactor fluids SS4 and CD2T \( (p = 0.001 \) and \( p = 0.028 \), respectively) and statistically significant differences in \( \eta_{\text{lim}} \) were observed for reactor fluids SS1° \( (p = 0.029) \), SS4 \( (p = 0.001) \), CD2T \( (p = 0.026) \), and CD4 \( (p = 0.008) \). The reactor fluids showing statistical differences in \( \eta_{\text{lim}} \) or \( \eta_{20} \) between the two sampling occasions also demonstrated different TS and TVS contents, except for CD4 (Table 2). In other words, the dependence of viscosities on TS or TVS could only be observed for SS and CDT but not for CD reactor fluids.

Digester fluid behaviour

Only digester fluids from CD4 and CD8T (second sampling) behaved as Newtonian fluids, exhibiting a linear relationship between shear stress and shear rate as well as no yield stress. The CDT reactor fluids, except CD8T, expressed various degrees of dilatant behaviour, with an increasing viscosity at higher shear rates. Strongly dilatant fluid behaviour may have important implications in determining the optimal mixing strategy for a process, since increasing mixing speed may lead to an increased apparent viscosity. However, the degree of dilatant behaviour described by the flow behaviour and consistency indices \( n \) and \( K \) in thermophilic CD reactor fluids was relatively low, as also demonstrated by comparatively low apparent viscosities throughout the shear range.

Yield stress could be detected in fluids from SS2T and SS4 reactors, which is in line with previously reported fluid behaviour for PBSS-digesting CSTBR systems (Baudez et al. 2011). The SS1° reactor fluid exhibited yield stress only at the first sampling occasion. Among the CD reactors, CD4, CD4s, CD5a, CD7as, CD7b and CD11 exhibited yield stress (Table 3). The measured negative yield stress of CD8Ts is due to limitations caused by relatively low apparent viscosities of the fluid as compared to the measurement noise (i.e. the low shear rates were within the baseline noise of the measurement). Reactor fluids exhibiting yield stress may induce the creation of a cavern (i.e. a small mixed zone) in the immediate surrounding of the stirrer, while the rest of the reactor volume remains unmixed (Nienow 2014). Such fluids are also problematic when pumped through pipes, since stagnant zones may appear if the fluid is not flowing fast enough to overcome the shear stress, leading to increased particle sedimentation and eventually pipe blockage (Farno et al. 2017).

Some of the CSTBR fluids from SS and mesophilic CD reactors featured a hysteresis area between the up- and down-curves in the rheograms, indicating possible thixotropic behaviour. Intermittent mixing of thixotropic fluids with long resting periods between the mixing occasions may lead to an increased energy consumption due to the additional power needed to break down the thixotropic structures every time the mixing initiates. In three cases (SS2T, SS4, and CD7b), the two curves even corresponded to distinctly different fluid types, indicating that the optimal speeds for intermittent stirring might be different than those required for continuous stirring (Table 3). It is noteworthy that the observed hysteresis area in rheograms may be caused by simultaneous presence of sheared and unsheared zones in the measuring gap of the rheometer during the ‘up-curve’ measurements. Such a phenomenon creates an apparent hysteresis area that is not caused by thixotropy and is related to the additional shear stress required for breaking down the elastic solid structure at the interface of the sheared and unsheared zones (Baudez 2006). Nevertheless, the consistent use of the same measuring protocol still allows for comparison between samples (Baudez 2006).

Rheological modelling on data from some samples (SS2T, CD5a, and CD7b) resulted in relatively low \( R^2 \) values for model fits. This was caused by individual outlier points in the rheograms or differences between replicate rheograms.
Among operational parameters, TVS correlated significantly with $\tau_0$ and $K$ ($r > 0.99$, $p < 0.001$ and $r = 0.93$, $p = 0.024$, respectively) for SS reactor fluids (Figure 1(d)), while pH was observed to correlate with $\tau_0$ of mesophilic CD fluids ($r = 0.88$, $p = 0.021$). However, this happened only across a relatively narrow pH interval of 7.7 to 8.1. To our knowledge, such correlation has not been reported before and further analyses of a more diverse...
set of fluids from CD reactors at mesophilic conditions are needed.

**Principal component analysis**

The PCA resulted in three principal components (PCs) that together described 86% of the variation in the data. All variables had relatively low contributions to the PCs, which is in line with the results of pair-wise correlation analyses (Figure S4). The results showed that HRT, TS, TVS, limit viscosity ($\eta_{\text{lim}}$), and consistency index ($K$) positively contributed to PC1, while temperature had the highest negative contribution. The consistency index ($K$) had the highest negative loading for the PC2 ($-0.51$), while pH and OLR had high positive contributions ($>0.5$). Therefore, the negative values of the second PC explain mainly the variation in rheological parameters, while the positive values describe the variation in operational and process parameters. Based on the PCA results, the CDT fluids were separated from SS and CD reactor liquids with their lower negative scores of PC1 (Figure 2), mainly associated with the contributions from temperature and flow behaviour index ($n$). For SS reactor fluids, PC2 had negative scores, thus implying a major contribution of the operational conditions of these processes. The SS1° and SS1*°'s samples, which have a combination of PBSS and FIW as substrate, can be found between the SS and the CD reactors, signifying the transitional characteristics of these fluids as compared to purely PBSS- and co-digesters. Digester CD11 was separated from all the other reactors probably related to its high pH, TS, and HRT compared to the other processes (Tables 1 and 2).

Our findings contradict those of Mbaye et al. (2014) who suggested that the rheological behaviours of AD fluids with the same TS or TVS are similar. In this study we show that fluids from CSTBRs digesting different substrates clearly do not behave in the same way. Since it has been shown before that the chemical composition of different CSTBR fluids is highly dependent on the substrates (Tambone et al. 2013), it is likely that the observed differences in rheological behaviour are a reflection of such characteristics. It is therefore clear that the rheology of CSTBR fluids is highly complex and dependent on an array of factors with different degree of interactions. Therefore, it is not plausible that a few parameters would enable a description of these interactions across the different substrate profiles of AD in CD reactors. Thus, this area calls for further research.

**Power requirement calculations**

Power requirements were calculated for pumping and mixing, based on rheological properties of the CSTBR fluids (cf. Table 3). It should be emphasized that our calculations are merely used to highlight differences in power requirements associated with different rheological characteristics of CSTBR fluids and their validity is restricted to the specified simplifying assumptions. Furthermore, the threshold $Re$ used in this study in place of $Re_m$ in Equation (3), was originally based on Newtonian fluids (Chapple et al. 2002). Since non-Newtonian fluids tend to reach this transition at higher $Re$ (Metzner & Otto 1957), the optimal mixing speeds based on the assumed $Re_m$ represent the lower-end threshold for power requirement.

Table 4 shows the estimated optimum pumping and mixing power requirements caused by varying rheological characteristics of the reactor fluids in the hypothetical CSTBR system. The rheology-driven power requirements for mixing varied from $10^{-7}$ to 0.002 kW/m$^3$ of digester volume, which was lower than the typical range of 0.005 to 0.008 kW/m$^3$, given in the literature (Appels et al. 2008). A lower estimate of power requirement based on rheological
In general, the power demands for operating a CD reactor under thermophilic conditions were always in the lowest range of the calculations. Our results thus highlight that operation of CD reactors at thermophilic conditions may be associated with lower power and, thus, energy requirements for pumping and mixing compared to the mesophilic condition. The observed differences may partially offset the increased energy requirement to maintain the higher temperature of the reactor and may be considered as an advantage of AD under thermophilic conditions. However, the fluid from the only SST reactor in this study, SS2T, still had a comparably high pumping power demand, indicating that thermophilic operation alone does not necessarily guarantee lower mixing and pumping power demands.

Although the method used for power requirement estimations enables a practically relevant interpretation of the rheological data, it does not take into account important parameters which may have substantial influence on regulation of the power requirements for mixing and pumping (e.g. its application is not suitable for dilatant fluids and yield stress is not accounted for when estimating the mixing power requirement). Additionally, the calculations only account for the power requirements based on fluid characteristics, and other important parameters such as the required minimal mixing speeds for achieving appropriate reactor volume turnover rates or minimal mass flows through the heat exchangers to compensate for heat losses of the reactor are not represented. To account for incomplete mixing associated with the change of the scale (e.g. larger volume) in full scale systems more accurate models such as computational fluid dynamics, which account for the shear distribution in reactor fluids, may be applied (e.g. López-Jiménez et al. 2015). Nevertheless, the relatively simple calculation approach presented in this study and its supplementary information allowed comparison of the contribution of rheology-driven power requirement based on different fluid characteristics of the CSTBRs with different substrate profiles and operational conditions. This approach could also be adapted to calculate the contribution of rheological parameters for reactors with dimensions differing from the one assumed here by replacing those specific values in the presented formulas.

**CONCLUSIONS**

The rheological characterization of fluids from 12 full-scale CSTBRs and their substrates showed distinct differences based on substrate and operational conditions of the processes. The TVS content showed the best correlation with

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### Table 4 | Pumping and mixing power requirement calculated from the modelling parameters of each sampled fluid

<table>
<thead>
<tr>
<th><em>Reactor</em></th>
<th>Pumping (W)</th>
<th>Mixing (W)</th>
<th>(V_c) (m/s)</th>
<th>(N_{tr}) (rpm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SS1s</td>
<td>680</td>
<td>0.10</td>
<td>0.44</td>
<td>0.23</td>
</tr>
<tr>
<td>SS1s</td>
<td>1.3</td>
<td>0.16</td>
<td>0.06</td>
<td>0.26</td>
</tr>
<tr>
<td>SS2T</td>
<td>1,800</td>
<td>0.16</td>
<td>0.62</td>
<td>0.26</td>
</tr>
<tr>
<td>SS4</td>
<td>2,500</td>
<td>200</td>
<td>0.70</td>
<td>2.8</td>
</tr>
<tr>
<td>SS4s</td>
<td>5,200</td>
<td>1,800</td>
<td>0.89</td>
<td>6.0</td>
</tr>
<tr>
<td>CD2T</td>
<td>1.3</td>
<td>0.09</td>
<td>0.06</td>
<td>0.22</td>
</tr>
<tr>
<td>CD2Ts</td>
<td>1.3</td>
<td>0.14</td>
<td>0.06</td>
<td>0.25</td>
</tr>
<tr>
<td>CD3T</td>
<td>1.3</td>
<td>0.15</td>
<td>0.06</td>
<td>0.26</td>
</tr>
<tr>
<td>CD4</td>
<td>190</td>
<td>0.62</td>
<td>0.29</td>
<td>0.42</td>
</tr>
<tr>
<td>CD4s</td>
<td>1,100</td>
<td>1.6</td>
<td>0.52</td>
<td>0.57</td>
</tr>
<tr>
<td>CD5a</td>
<td>1.3</td>
<td>0.56</td>
<td>0.06</td>
<td>0.35</td>
</tr>
<tr>
<td>CD5as</td>
<td>1.3</td>
<td>0.06</td>
<td>0.06</td>
<td>0.19</td>
</tr>
<tr>
<td>CD5b</td>
<td>1.3</td>
<td>0.17</td>
<td>0.06</td>
<td>0.27</td>
</tr>
<tr>
<td>CD7a</td>
<td>na</td>
<td>na</td>
<td>na</td>
<td>na</td>
</tr>
<tr>
<td>CD7as</td>
<td>1,300</td>
<td>23</td>
<td>0.56</td>
<td>1.4</td>
</tr>
<tr>
<td>CD7b</td>
<td>3,300</td>
<td>39</td>
<td>0.76</td>
<td>1.7</td>
</tr>
<tr>
<td>CD8T</td>
<td>1.3</td>
<td>0.13</td>
<td>0.06</td>
<td>0.24</td>
</tr>
<tr>
<td>CD8Ts</td>
<td>1.3</td>
<td>0.24</td>
<td>0.06</td>
<td>0.30</td>
</tr>
<tr>
<td>CD11</td>
<td>2,600</td>
<td>2.5</td>
<td>0.69</td>
<td>0.66</td>
</tr>
</tbody>
</table>

The results are presented in W. Critical flow velocity (\(V_c\)) to achieve turbulent flow and minimal stirring speed (\(N_{tr}\)) to achieve turbulent mixing in the hypothetical 1,000 m3 reactor are presented in m/s and rpm, respectively.

*Reactors SS1*, SS4, CD2T, CD4, CD5a, CD7b, and CD8 were sampled at two occasions, showed by letter 's' in front of the reactors' name.

The value of 1.3 W was calculated for fluids without a yield stress for the minimal assumed flow speed of 0.06 m/s, corresponding to the required substrate flow speed.
\( \eta_{20} \) and \( \eta_{\text{lim}} \) of SS reactors, while in the case of thermophilic CD reactors, TS revealed the strongest correlation with \( \eta_{\text{lim}} \). No such relationships were found for the CD reactors under mesophilic conditions. The results showed that using TS or TVS as estimators of CSTBR fluid rheology may lead to erroneous predictions of fluid behaviour and power demand. Not accounting for the process specifications concerning substrate origin and operational temperature may be the reason behind the discrepancies in the literature regarding the importance of TS and TVS for CSTBR fluid rheology. A range of different fluid behaviours was observed among the reactor liquids. The CDT reactors often demonstrated weak dilatant fluid behaviour. Estimated power requirements based on the rheological characteristics of the CSTBRs fluids showed a large variation irrespective of the TS and TVS contents. The CDT reactors were found to generally have the lowest power requirements, which may be considered as a general advantage of thermophilic operation of CD systems. Rheological characterization of CSTBR fluids with a broader range of substrate profiles including a wide range of operating parameters is needed to elucidate mechanisms regulating fluid rheology particularly for CD systems.

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DECLARATIONS OF INTEREST

None.

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