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Comparative analysis of anaerobically digested wastes flow properties



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ABSTRACT

The flow curve of anaerobically digested wastes from different origins was determined through rheological measurements. Regardless of their origin, samples can be divided into two families: simple non-Newtonian liquids well modelled by basic power law below 10%DC and viscoelastic liquids with a yield stress, well modelled by a Herschel–Bulkley model above. In all the cases, the rheological behaviour is driven by both the organic content and the volatile fraction (organic content/solid content), indicating that anaerobic digestion tends to smooth the rheological characteristics of organic wastes, whichever their origins.

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1. Introduction

Nowadays, the process of anaerobic digestion is widely used for waste management. It is also one of the most sustainable ways for producing renewable energy such as biogas (Holm-Nielsen et al., 2009). Given this situation, there is a strong motivation for extending the anaerobic digestion process to a wide range of waste materials (plant residues, organic, domestic or food wastes, manures, etc.), particularly interesting for their anaerobic decomposition (Sharma et al., 1999). However, despite its numerous advantages, anaerobic digestion still produces a residue, the anaerobic digested waste, which has to be reused. Due to their attractive agronomic potential (Favoino, 2008), anaerobic digested wastes are mainly reused in agriculture but additional treatments are required to allow handling regarding their solid concentration (Flotats et al., 2009). Handling is directly connected to mechanical properties of materials. Thus the knowledge of rheological characteristics of anaerobic digested wastes will facilitate the design of processes for handling them.

Publications found in the literature about the rheology of anaerobic digested wastes mainly concern sewage sludge. They clearly highlight some of the effects resulting from the process of anaerobic digestion such as shear-thinning (Monteiro, 1997; Mu and Yu, 2006) and the decrease of viscosity influenced by the duration of the fermentation period (Aranowski et al., 2010). Also, Baudez et al. (2011) confirmed this shear-thinning behaviour for anaerobic digested sludge, and showed that its rheological behaviour depended on the shear stress level, with possible shear banding

instability at low shear stress, and power law model behaviour at higher stress.

Nevertheless, a lack of information remains concerning the wide range of anaerobically digested waste materials. Battistoni et al. (1993) and Battistoni (1997) focused their research on organic fraction of municipal solid waste and highlighted that the rheological characteristics are correlated with the total volatile solids by following an exponential. More recently, Garcia-Bernet et al. (2011) found an exponential relationship between yield stress and total solids concentration with the same materials.

However probably owing to their heterogeneity, there are no publications describing the flow behaviour of these waste materials. No comparative analysis of rheology was found for digested wastes from different origins, solid concentration as well as particles interactions. In this paper, we compared different types of anaerobically digested wastes and we demonstrated that regardless of their origin, their rheological behaviour is mainly driven by both the total solids and the total volatile solids, as stated by Garcia-Bernet et al. (2011) but also by Battistoni (1997) and Battistoni et al. (1993).

2. Materials and methods

2.1. Samples nature and preparation

Different samples of anaerobically digested wastes from five origins were considered in this study (Table 1). They were picked up at the outlet of the anaerobic digester and stored at 4 $^{\circ}$ C before being used. Although anaerobic digestion is a stabilization process, our samples were stored at 4 $^{\circ}$ C for 30 days before experimentation, in order to ensure that no temporal variability occurred,

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 Table 1

 Information about the anaerobically digested wastes considered in this study.

| Type of digested waste and nature of raw wastes | Corresponding notation | Averaged total solid content (%) | Averaged organic matter (%) | Process |
|---|------------------------|----------------------------------|-----------------------------|--------------|
| Agricultural (manure + cereals) | A1 | 17.1 | 11.22 | Mesophilic |
| Agricultural (manure + cereals) | A2 | 5.63 | 3.97 | Mesophilic |
| Green wastes | GW | 20.03 | 10.24 | Thermophilic |
| Municipal wastes | MW | 26.66 | 11.05 | Thermophilic |
| Territorial (manure and organic wastes) | TW | 5.74 | 4.09 | Mesophilic |

allowing us to use the same material over several days. This technique was successfully used by Curvers et al. (2009).

Moreover, in order to highlight time variability, four monthly samplings were done for each digester.

The total solid concentration of each sample was determined by drying at $105\,^{\circ}\text{C}$ during 24 h, according to the ASAE standard and the mineral residue was measured by burning the dried sample at $550\,^{\circ}\text{C}$ for 2 h. The total volatile concentration was considered to be the difference of mass losses at $550\,^{\circ}\text{C}$ and $105\,^{\circ}\text{C}$.

Lastly, because most of the anaerobically digested wastes still contained coarse particles (i.e. glass, metal, wood, rubber), samples were sieved using a 7 mm sieve prior to rheological measurements.

2.2. Rheological measurements

Rheological measurements were performed using a stress controlled rheometer MCR300 (Anton Paar) equipped with wide gap coaxial cylinders and rough surfaces to avoid wall slip (inner radius 12.5 mm, outer radius 19.5 mm, height 70 mm). As established by Baudez et al. (2011), temperature only quantitatively impacts rheological measurement: curves and fitting models are qualitatively the same between 20 and 60 °C. Thus, the temperature test was regulated at 20 ± 0.3 °C by a thermal water bath.

Before each measurement, samples were presheared at a rotational speed of 600 rpm in order to determine the corresponding range of torque adapted to the determination of the samples flow curve.

Then, samples were sheared according to the following two steps procedure:

- Torque maintained at M_{max} for 1 min to reach a permanent state, M_{max} corresponding to the torque for which the shear rate is equivalent to a rotational speed of 600 rpm.
- Logarithmic ramp of decreasing torque from M_{max} to 0.

Because all of the samples do not flow in the same range of torque, the slope of torque ramp was examined to ensure that the slope was the same for each test. In order to not specifically focus on high velocities and neglect lowest values, the logarithmic ramp allowed us to have the same numbers of points in each decade.

Shear rate and shear stress were calculated from the raw data. The shear stress τ was calculated from the torque M according to the relation $\tau = \frac{M}{2\pi h R^2}$, where h is the height of the mobile and R its radius.

Using wide gap geometry, the usual straight gap approximation to determine the shear rate is not convenient and the shear rate has be calculated as defined by Piau (1979), cited by Baudez and Coussot (2001) (see Appendix A).

3. Results and discussion

With the materials used in this work, all the samples showed a non-Newtonian shear-thinning behaviour as the viscosity decreases when the shear rate increases (Fig. 1) and two distinct groups can be defined:

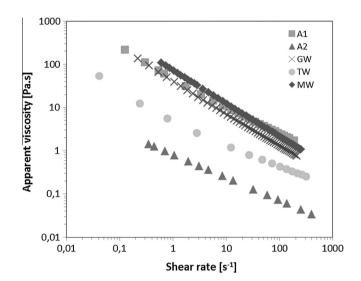


Fig. 1. Apparent viscosity vs. shear rate for different anaerobically digested wastes.

- First, materials which are basically liquids, with no yield stress
- Second, materials which highlight a solid-liquid transition defined by the existence of a yield stress (a critical shear stress has to be reached to initiate flow) (Table 2).

Globally, samples above 10%DC have a yield stress whereas samples below 10%DC do not have one and the range of yield stress values is consistent with the literature (Garcia-Bernet et al., 2011).

First group materials, such as samples A2 and TW are well described by an Ostwald model (Fig. 2):

$$\tau = K\dot{\gamma}^n \tag{1}$$

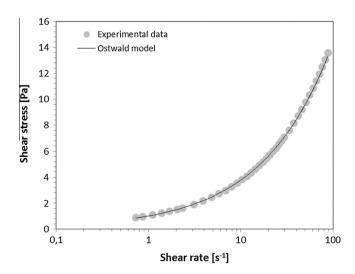


Fig. 2. Flow curve and Ostwald model on TW sample4 (k = 0.3419 and n = 0.6438) ($R^2 = 0.9997$).

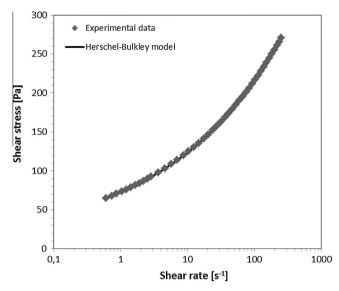


Fig. 3. Flow curve and Herschel–Bulkley model on MW sample1 ($\tau c = 9$. 05, k = 63.77 and n = 0.256) ($R^2 = 0.999$).

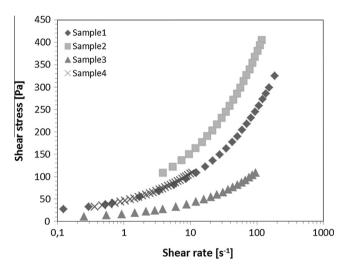


Fig. 4. Flow curves of different A1 samples (after sieving) taken at different times.

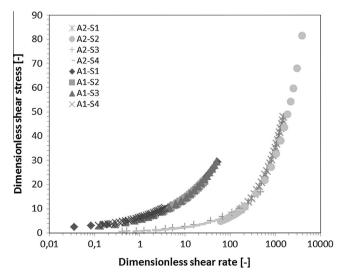


Fig. 5. Flow curve master curves for A2 and A1 samples taken at different times.

Table 3Dimensionless parameters of the master curves.

| Type of digested waste | Averaged solid content (%) | Dimensionless yield stress (–) | Dimensionless consistency (–) | Dimensionless index (–) |
|------------------------------|----------------------------------|-----------------------------------|----------------------------------|----------------------------|
| TW | 5.74 | 0 | 0.977 | 0.584 |
| A2 | 5.63 | 0 | 0.744 | 0.524 |
| A1 | 17.1 | 1 | 5.216 | 0.424 |
| GW | 20.03 | 1 | 23.936 | 0.271 |
| MW | 26.66 | 1 | 1.364 | 0.282 |

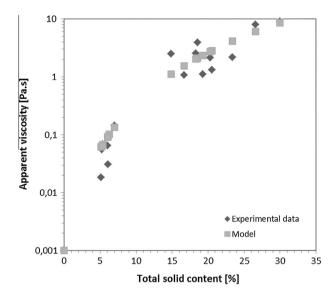


Fig. 6. Apparent viscosity (at $100 \, \text{s}^{-1}$) as a function of the total solid content for the experimental data (dark diamonds) and the corresponding model (empty squares). The model parameters are $\eta_0 = 0.001$, a = 1.487, b = 0.423 and c = 0.3 ($R^2 = 0.924$).

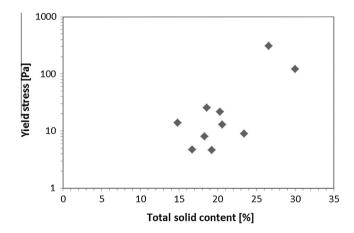


Fig. 7. Yield stress as a function of the total solid content for A1, GW and MW samples. No clear relationship can be established.

where τ is the shear stress, $\dot{\gamma}$ the shear rate, K the consistency and n is the consistency index.

They do not show a yield stress which means they are flowing whichever the applied shear stress.

Second group materials, such as samples A1, GW and MW are well fitted by a Herschel–Bulkley model (Fig. 3):

$$\tau = \tau_c + K \dot{\gamma}^n \tag{2}$$

Where τ is the shear stress, τ_c the yield stress, $\dot{\gamma}$ the shear rate, K the consistency and n is the consistency index.

Table 2Flow curves parameters for all the samples, adjusted from the master curve (i.e. index is kept constant for a given type of waste).

| Type of digested waste | Yield stress (Pa) | Consistency (Pa. s ⁿ) | Index (-) | Total solid content (TSC) (%) | Organic content (OC) (%) | Volatile fraction (OC/TSC) (-) |
|------------------------|-------------------|-----------------------------------|-----------|-------------------------------|--------------------------|--------------------------------|
| A1 | 11.063 | 33.785 | 0.424 | 14.84 | 10.74 | 0.72 |
| | 18.965 | 52.694 | 0.424 | 18.57 | 12.14 | 0.65 |
| | 3.688 | 14.602 | 0.424 | 16.69 | 10.56 | 0.63 |
| | 10.536 | 34.76 | 0.424 | 18.31 | 11.45 | 0.63 |
| A2 | _ | 0.39 | 0.451 | 6.09 | 4.38 | 0.72 |
| | - | 0.229 | 0.451 | 5.16 | 3.58 | 0.69 |
| | - | 0.699 | 0.451 | 5.25 | 3.79 | 0.72 |
| | - | 0.812 | 0.451 | 6.02 | 4.11 | 0.68 |
| GW | 1.085 | 37.4 | 0.271 | 20.55 | 10.48 | 0.51 |
| | 1.22 | 31.227 | 0.271 | 19.25 | 10.74 | 0.56 |
| | 0.271 | 10.109 | 0.271 | 17.95 | 10.68 | 0.6 |
| | 2.712 | 60.41 | 0.271 | 20.30 | 9.50 | 0.47 |
| MW | 25.039 | 51.999 | 0.282 | 23.41 | 10.21 | 0.44 |
| | 166.927 | 172.201 | 0.282 | 26.6 | 10.4 | 0.39 |
| | 208.659 | 186.414 | 0.282 | 29.98 | 12.55 | 0.42 |
| TW | _ | 0.455 | 0.587 | 5.37 | 3.82 | 0.71 |
| | _ | 0.621 | 0.587 | 6.28 | 4.50 | 0.72 |
| | - | 0.964 | 0.587 | 7.00 | 5.05 | 0.72 |

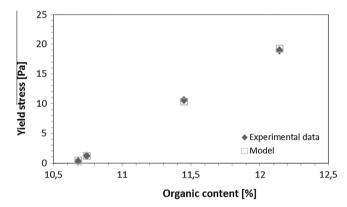


Fig. 8. Yield stress as a function of the organic content for samples with similar total solid content (\approx 18%DC), experimental data (dark diamonds) and corresponding model (empty squares). The relationship is linear τ_c = 12.83 (ϕ_{oc} -10.65), (R^2 = 0.9997).

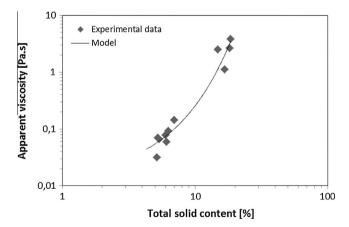


Fig. 9. Apparent viscosity (at $100 \, \text{s}^{-1}$) as a function of the total solid content for samples with similar ratio organic content/total solid content (\approx 0.7), for the experimental data (dark diamonds) and the corresponding model (straight line). The model equation is η_{apparent} = 0.0118 e^{0.3067 ϕ} (R^2 = 0.9507).

Focusing on a single type of waste, the rheological behaviour of samples is self-similar as they show the same shape (Fig. 4). By reducing shear rate and shear stress in a dimensionless form (Baudez et al., 2004), a master curve can also be defined for each

digested waste family (Fig. 5), indicating that the same kind of interactions are governing the rheological behaviour of each material (Table 3). Such master curve allows us to determine the flow curve only by determining the dimensionless parameters.

If the apparent viscosity monotonically increases with the solid content (Fig. 6), independently of the sample, no clear correlation was found between the yield stress and the solid concentration (Fig. 7). The solids dependence of the viscosity can be easily modelled (Fig. 6) by considering the digestate as a moderately concentrated suspension for which the viscosity is a third order limited development (Ammari et al., 2012) of the form:

$$\eta_{annarent} = \eta_0 (1 + a.\phi + b.\phi^2 + c.\phi^3)$$
(3)

where $\eta_{apparent}$ is the apparent viscosity (corresponding to a given shear rate), η_0 is the water viscosity, ϕ is the total solid content, and where a, b and c are model parameters.

However, the experimental data is very noisy between 10% and 20% of solids, the range of concentrations where a yield stress can be noticed.

As shown in Table 2, our samples displayed different solid concentrations but also different organic loads such that the ratio organic/solid is not constant. In 2001, Baudez and Coussot highlighted that the organic and mineral content of pasty sludge directly affected the rheological behaviour: for a given and constant solid concentration, the higher the organic content, the higher the rheological characteristics of sludge.

Straight from this result, when the solid concentration is considered constant, the amount of organic content significantly impacts the rheological behaviour of anaerobically digested wastes: the higher the organic load, the higher the yield stress (and the viscosity), independently of material origin. A linear relationship was found between the yield stress and the organic content when the total solids is kept constant (Fig. 8):

$$\tau_{\rm c} = \alpha.(\phi_{\rm OC} - \phi_{\rm 0_{\rm organic}}) \tag{4}$$

where τ_c is the yield stress, ϕ_{OC} is the percentage of organic content, $\phi_{0_{organic}}$ a critical concentration, and α is a constant parameter.

The fitting value of $\phi_{0_{organic}}$ is equal to 10.68% which is consistent with the two distinct groups we defined above. Comparing this with the literature about sewage sludge (Baudez, 2008; Baudez et al., 2011), $\phi_{0_{organic}}$ can be assimilated to the concentration below which there is no significant yield stress.

In parallel, when the volatile fraction (organic/solids) is considered constant, an exponential relationship is observed between the apparent viscosity (some samples having no yield stress) and the solid concentration (Fig. 9) as well as a power-law relationship with the yield stress (when exists):

$$\eta_{apparent} = Ae^{B\phi} \tag{5}$$

where $\eta_{apparent}$ is the apparent viscosity (for a given shear rate), ϕ is the total solid content, and A and B are constant parameters.

These results are consistent with the literature (Battistoni et al., 1993; Battistoni, 1997; Baudez, 2008; Baudez et al., 2011) and close to those obtained by Garcia-Bernet et al. (2011). The slight differences observed between all those results may be explained either by the type of digested wastes considered in each paper or by the fact that we consider the apparent viscosity instead of the yield stress.

To summarize, despite their different origins, all those anaerobically digested wastes behave similarly and present close rheological properties, for which both the total solid and the total volatile contents are the key parameters to define their flowing behaviour.

Even if it was never established before, these results are quite logical because anaerobic digestion is a biochemical process for which the same pool of reactions occurs whichever the material (Pretorius, 1983; Pavlostathis and Giraldo-Gomez, 1991). Basically, organic polymers are hydrolysed into a well-known bunch of monomers or at least smaller molecules (amino acids, sugars, fatty acids, alcohol), then acetate and methane are produced. Thus, based on this chain of reactions, if the digestion process is complete, the final residue contains more or less the same hardly biodegradable compounds (though in different quantities). Anaerobically digested wastes can be qualitatively considered as homogeneous materials, meaning that the same interactions are dominant. In other words, anaerobic digestion process tends to smooth the rheological behaviour of wastes.

4. Conclusion

This paper investigated the flow properties of different types of anaerobically digested wastes including agricultural, green and municipal wastes. We observed that regardless of their origin, materials can be divided into two groups: flow properties of samples below 10%DC can be described by a single power law (Ostwald model), whereas samples above 10%DC have a yield stress and are well described by a Herschel–Bulkley model.

By reducing the shear rate and the shear stress in a dimensionless form, a master curve was obtained for each of the materials families, indicating the samples from the same digester highlight strong similarities.

By going deeper through these similarities, for materials which have the same solid concentration – regardless of their origin – rheological properties are strongly correlated with the total volatile concentration. Moreover, when the volatile fraction is imposed, the well-known correlations with the solid concentrations are found

These results allow us to consider anaerobic digestion modifies material composition to tend towards a qualitatively homogeneous material for which the rheological behaviour is governed by the same kind of interactions. Thus, regarding the materials considered in this study, two anaerobically digested wastes, with the same solid concentration and the same volatile fraction, would have the same rheological behaviour.

The direct consequence of these results is abacus of the rheological behaviour of anaerobically digested wastes can be drawn,

basically by knowing their total solid and total volatile solid concentrations. It opens new insight to improve post-treatment processes.

Moreover, because the chemical reactions involved in the anaerobic digestion process are always the same, further study needs to be done in order to investigate the biochemical composition of anaerobically digested wastes and to look at its relationships with rheological properties. This will be the following step.

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Appendix A. Shear rate wide gap calculation

Using wide gap coaxial cylinders, both shear stress and shear rate are heterogeneous within the gap.

The material rheological behaviour is described by:

$$\tau = f(\dot{\gamma}) \iff \dot{\gamma} = \xi(\tau) = \xi\left(\frac{M}{2\pi R^2 L}\right)$$
 (A1)

R and L being the internal mobile cylinder dimensions (radius and height).

The rotational speed at a distance r from the central axis is given by:

$$\omega_{r} - \omega_{R_{1}} = \int_{R_{1}}^{r} \frac{1}{x} \dot{\gamma}_{x} dx = \int_{R_{1}}^{r} \frac{1}{x} \xi \left(\frac{M}{2\pi x^{2} L} \right) dx = \frac{1}{2} \int_{\frac{M}{2\pi r^{2} L}}^{\frac{M}{2\pi R_{1}^{2} L}} \frac{\xi(\tau)}{\tau} d\tau$$
(A2)

Applied to the whole sample volume, Eq. (A2) becomes:

$$0 - \omega_{R_1} = \frac{1}{2} \int_{\frac{M}{2\pi R_1^2 L}}^{\frac{M}{2\pi R_1^2 L}} \frac{\zeta(\tau)}{\tau} d\tau = \delta \omega \tag{A3}$$

Experimentally, a series of torque (n; M) is determined. The torque by length unit, M/L, is represented as a function of the angular speed $\delta\omega$ in order to determine the function ξ . To do that, Eq. (A3) is differentiated considering M/L:

$$2\frac{M}{L} \cdot \frac{d(\delta\omega)}{d(\frac{M}{L})} = \xi \left(\frac{M}{2\pi R_1^2 L}\right) - \xi \left(\frac{M}{2\pi R_2^2 L}\right) \tag{A4}$$

The variable β is defined:

$$\beta = \left(\frac{R_1}{R_2}\right)^2 < 1$$

Using β , Eq. (A4) becomes:

$$2\frac{M}{L} \cdot \frac{d(\delta\omega)}{d(\frac{M}{L})} = \xi \left(\frac{M}{2\pi R_1^2 L}\right) - \xi \left(\beta \frac{M}{2\pi R_1^2 L}\right)$$

$$\Rightarrow \sum_{n=0}^{\infty} 2\beta^n \frac{M}{L} \frac{d(\delta\omega)}{d(\beta^n \frac{M}{L})} = \xi \left(\frac{M}{2\pi R_1^2 L}\right) - \xi(0) \tag{A5}$$

At rest, the material is not submitted to any shear stress, and $\xi(0)=0$, so that:

$$\dot{\gamma} = \xi \left(\frac{M}{2\pi R_1^2 L}\right) = \sum_{n=0}^{\infty} 2\left(\frac{R_1}{R_2}\right)^{2n} \frac{M}{L} \frac{d(\delta\omega)}{d\left(\left[\frac{R_1}{R_2}\right]^{2n} \frac{M}{L}\right)} \text{ and } \tau = \frac{M}{2\pi R_1^2 L}$$
 (A6)

To determine the flow curve, we first plot the curve $M/L = f(\delta\omega)$ (calculated from the rotational speed). The best fitting mathematical model is then determined from the experimental points (best correlation coefficient). This model can be written as:

$$\frac{M}{L} = \left(\frac{M}{L}\right)_0 + a(\delta\omega)^b, \left(\frac{M}{L}\right)_0 \in [0; \infty[, a \in]0; \infty[, b \in]0; 1] \tag{A7}$$

Practically, we plot the curve $\frac{M}{L}-(\frac{M}{L})_0=f(\delta\omega)$ which can be fitted by a power law and we determine the value of $(\frac{M}{L})_0$ in order to obtain the best correlation coefficient.

Eq. (A7) is differentiated considering $\left(\frac{R_1}{R_n}\right)^{2n} \frac{M}{I}$, leading to Eq. (A8):

$$\frac{d[\delta\omega]}{d\left\lceil \left(\frac{R_1}{R_2}\right)^{2n}\frac{M}{L}\right\rceil} = \frac{1}{a.b} \left(\frac{\left(\frac{R_1}{R_2}\right)^{2n}\frac{M}{L} - \left(\frac{M}{L}\right)_0}{a}\right)^{\frac{1-b}{b}}$$
(A8)

Based on Eq. (A8), we calculate one by one the terms of the Eq. (A6) for many successive values of the coefficient n until the series converges then we plot the rheogram $(\dot{\gamma}; \tau)$.

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