Rheological behaviour of residues in dry anaerobic digestion performed by Vane geometry

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Abstract

The knowledge of the rheology of residues involved in dry digestion is an essential factor for process optimization. Despite this fact, it is difficult to characterize the rheological behaviour of these residues with high total solid (TS) content. Indeed, the standard methods of rheological measurements are poorly adapted to these residues because of the sliding effects observed during the measurements. In this study, we used a non-standard method, the Vane geometry, for its particularity to limit the sliding effects. Two types of measurements were carried out on potato residues of 18 to 35% TS, extracted from the digesters. The first measurement was to submit to the sample a rising shear velocity ramp from 0 to 200 s^{-1} at 37°C and 55°C. The results of these measurements show a good correlation between the rheograms and Herschel-Bulkley model and we deduced a rheofluidifying threshold behaviour. The second measurement was performed in oscillations at 10 Hz at 25°C, 37°C and 55°C. The results show different evolutions of the elastic G' and viscous G'' modulus as a function of temperature. There is a crossing of G' and G'' at 25°C, G' greater than G'' at 37°C, and finally G' and G'' forming a plateau at 55°C, characteristic of a gel. The knowledge of these rheological behaviour is crucial for the design and operating instructions of anaerobic digestion reactors for residues with high TS content. The Vane geometry would, therefore, be an effective tool for characterizing the rheological behaviour of residues in dry digestion.

Keywords: Dry anaerobic digestion, Rheology, Vane geometry, Rheofluidifying, Threshold fluid, Gel.

Word count: 6956 words.

1. Introduction

Anaerobic digestion is a natural process that humans seek to understand and domesticate to optimize its functioning and make it more effective in removing organic residues in solid or dissolved form in water. This domesticated process allows both the depollution, the production of renewable energy and agricultural fertilizer [1]. Depending on the total solid (TS) content of the residues in the digester, two types of technologies are distinguished. Wet anaerobic digestion, characterized by total solid (TS) levels below 15% and dry anaerobic digestion, for which TS levels are between 15% and 40% [2, 3].

Anaerobic dry digestion, due to the many competitive advantages it has over the wet route, has experienced a real industrial boom in recent years [4]. Until the 1990s, wet digestion was widely used for the treatment of municipal waste. In 2010, it represented only 40% of the treatment capacity, compared to 60% for dry digestion [5]. This interest in dry digestion is motivated by the low volume of digester for the same organic load, the absence of excessive water addition to the substrate, and the post-treatment facilitated by the absence of solid-liquid phase separation [5, 6]. Already in the 1990s, biogas production and methane yields from digesters were shown to be higher or equal when solid residues were digested as they were or with little water added [7].

However, the dry matter content between 15% and 40% gives the solid water mixture a pasty consistency which induces noticeable differences in rheological behaviour and creates more severe technical constraints in terms of handling, pre-treatment or mixing compared to wet processes [8, 9].

Several authors have shown that the rheological behaviour of these residues with high dry matter content had significant impacts on food, agitation [10-16], monitoring and design of digesters [17]. They also noted that rheological behaviour influences chemical balances [6], transfer [18, 19], the mobility of these residues in digesters [20] and finally the rate of degradation [21-24]. Under these conditions, the optimization of a digester requires the characterization of the rheological behaviour of the substrate.

However, for these authors, the effective characterization of the rheological behaviour of these high dry matter residues remains a real scientific and technological challenge to which more appropriate solutions will have to be found [25]. Indeed, standard rheological measurement methods using rotary rheometers with plane-to-plane, cone-to-plane or cylindrical geometries are poorly adapted to these types of heterogeneous residues with high dry matter content [26-29]. Another approach of using a collapse test (used in the concrete industry) also demonstrated its limitations [11]. The effectiveness of most of these methods is undermined by sample slippage during measurements [11, 26-28].

In this study, we propose to use a rotating rheometer with a Vane geometry (coaxial cylinder with a cross as internal diameter), recognized for its ability to limit the sliding effects during rheological measurements of highly concentrated substrates [29-31], to characterize the rheological behaviour of potato residues extracted from four batch digesters with a TS content ranging from 18 to 35%.

2. Materials and methods

2.1. Rheological measuring device

2.1.1. Rheometer

We used an AR 2000 rheometer (TA Instruments, smart Swap technology, 200 mN.m Torque motor - France) for the different rheology measurements. With this type of rheometer, it is possible to make measurements on a very wide range of torque. This allows to describe the entire viscosity curve in a single measure. The rheograms obtained from the AR 2000 rheometer are distinguished by higher deformation resolutions, a wider frequency range, and interchangeable temperature control options made possible by Smart Drive technology. The rheometer is also equipped with the Mobius Drive control system, which makes it possible to carry out both constrained and imposed speed measurements. The rapid use of measurement data is made possible by the "Rheology Advantage" software. This software skillfully combined with the appropriate measuring accessories makes it possible to provide rapid characterization and complete mathematical modeling of data on a wide range of materials.

When the rheometer is mounted with a concentric cylinder system [32], it allows temperature control by the Peltier effect, and gives a whole new dimension of operational efficiency of the measurements and a significant time saving, especially when large temperature changes are necessary. The particularity of the use of the AR 2000 in this study was to mount a concentric cylinder system, more precisely the Vane geometry, which is a coaxial cylinder whose internal cylinder is a cross-brace made up of several blades (Fig. 2).

2.1.2. Vane Geometry

In rheology measurements, the choice of geometry models should generally meet four requirements, namely: (1) obtain the highest possible shear velocities and therefore use the lowest possible cone angle; (2) have a ratio between the mean dimension of the gap and the diameter of the particles as high as possible, which would lead to the use of the highest angle; (3) eventually, avoid any fracturing phenomena that may occur in the sample during measurements; and; (4) avoid any sliding phenomena during measurement. Compliance with these conditions depends on the substrate used. Therefore, it is necessary to choose the measurement geometry according to the characteristics of the material to be characterized. Generally, residues used in anaerobic dry digestion are characterized by high dry matter levels of between 15 and 45% [2, 3]. Municipal waste, agricultural waste, and industrial waste which is highly concentrated in dry and heterogeneous matter are most often found.

In our study, we used similar waste, crushed potato residues at dry matter levels between 15% and 35%. For these types of pasty, heterogeneous, dry matter-rich residues, it is difficult to effectively characterize their rheological behaviour through standard geometries such as plane-plane, cone-plane or even simple cylindrical geometry [29-31]. This is why we use a non-standard geometry but more adapted, the Vane geometry. Indeed, the Vane geometry (coaxial cylindrical cross-sectional geometry) replaces the simple cylindrical geometry in that the inner cylinder is replaced by a cross-sectional blade [33] (Fig. 2). This geometry is most often used to measure the properties of threshold fluids, particularly stress thresholds [29, 34, 35] which could be the case for most municipal or agricultural waste used in anaerobic dry digestion.

The main advantage we have of using this geometry is the ability to reduce the sliding effects of the sample during measurements. Indeed, the cross blades increase the contact surface between the sample and the measurement geometry, thus reducing the sliding effects during measurements. The Vane geometry has other advantages that make it particularly suitable for the type of substrate studied, namely that it makes it possible to measure large sample volumes and very heterogeneous substrates. Figure 2 shows the Vane geometry used during laboratory measurements. It consists of a coaxial cylinder with an external diameter of 33 mm and an internal diameter (the vane) of 28 mm.

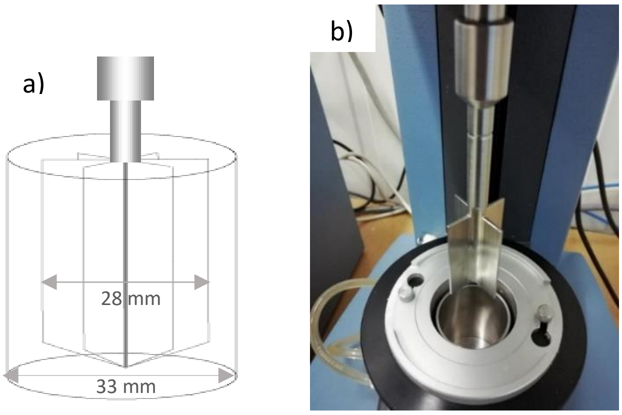


Fig. 2. Vane Geometry. a- Configuration of geometry Vane. b- Rheometer AR 2000 with geometry Vane

2.2. Rheological measurement protocol

2.2.1. Shear flow measurement

We performed two types of rheology measurements on 18% potato residue samples, 20%, 22%, 25%, 30% and 35% TS at 37°C, and 55°C. The first measurement consisted of shear flow tests to measure the flow ability of the sample subjected to certain stress or shear velocity. The curves (rheograms) obtained from these tests are then compared with the existing rheological models to describe the rheological behaviour of the measured sample. The second measurement consisted of oscillation tests (or dynamic regime) to characterize the viscoelastic nature, the internal structure of the samples. The rheometer AR 2000 used for these tests is fitted with a Vane geometry. We defined a measurement protocol and carried out the measurements in triplicate to ensure its repeatability and replication.

During shear flow, potato residues are first subjected to a precision of 100 s^{-1} for 2 min. After this step, the specified sample is kept at rest for two 2 min to prevent memory effects [36]. This technique makes it possible to homogenize the sample but also to break the bonds formed at the level of the particles. The actual shear flow measurement consisted of applying a rising shear velocity ramp from 0 to 200 s^{-1} to the sample after this precision step. The measurement temperatures are 37°C and 55°C, corresponding to the optimal mesophilic and thermophilic digestion temperatures, respectively [1, 37].

2.2.2. Oscillation measurement

To better understand the internal structure of the substrates, we made oscillations or dynamic measurements. These tests consisted of measuring the elastic or storage modulus G' and the viscous or loss modulus G'' at a frequency corresponding to the frequency for which the linear domain is reached. The elastic modulus G' corresponds to the elastic energy stored and returned over a given period. On the other hand, the viscous G'' modulus is the energy dissipated by viscous friction during the same period.

These rheological values (G' and G'' depend on the nature of the material, the temperature but also the frequency at which the material is requested. Therefore, a sample may have highly viscous behaviour at one frequency and highly elastic at another at the same temperature and vice versa. For our potato samples, we made measurements over a frequency range from 10^{-2} to 10^{2} Hz. The stresses applied to vary from $2x10^{-3}$ to $2x10^{-2}$ Pa and the linear range is reached at a frequency of 10 Hz. At this frequency, the stressed material is thus simply deformed, but its internal structure remains intact, the stresses applied to be sufficiently low not to cause a rupture of the inter-particulate bonds.

3. Results

3.1. Determination of threshold constraint

3.1.1 Shear flow measurement at 37°C

The curves obtained from shear flow measurements from 0 to 200 s^{-1} at a temperature of 37°C show three phases of stress evolution (fig.3). The first phase of evolution with a slight increase in stress for shears from 0.001 to 0.01 s^{-1}. A second phase is observed at a shear rate of 0.01 and 10 s^{-1}, where stress decreases. And the third phase of increased stress from shear velocities higher than 10 s^{-1}.

When we compare the curves obtained according to the dry matter content, we observe that the more concentrated the sample, the higher the stress. The first stress values observed on the curves are the threshold stresses, that is, the stress for which the sample flows. These threshold stresses are obtained for shears of 0.001 s^{-1}. The threshold constraints represented on the curves are 6.13 Pa, 6.13 Pa, 6.15 Pa, 11.1 Pa, 23.2 Pa, and 57.2 Pa, respectively for 18%, 20%, 22%, 25%, 30% and 35% TS.

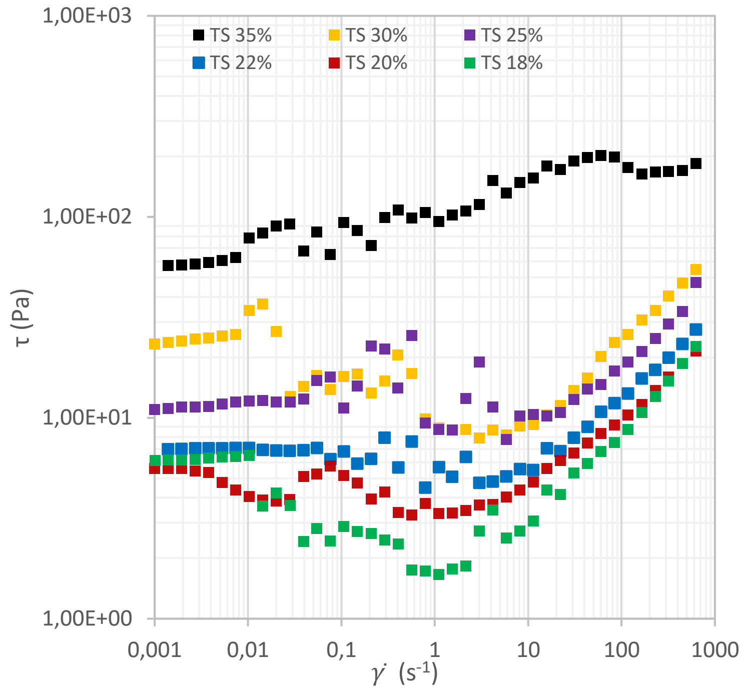


Fig. 3. Stress evolution curves as a function of the shear rate at 37°C

3.1.2. Shear flow measurement at 55°C

For shear flow measurements carried out at 55°C (Fig. 4), we observe, as in Fig 3, three phases of stress evolution. The first phase of low stress increases at a shear rate from 0.001 to 0.1 s^{-1}. A second phase between 0.1 and 10 s^{-1} where the stress decreases with shear. And the third phase of increased stress from shear velocities greater than 10 s^{-1}.

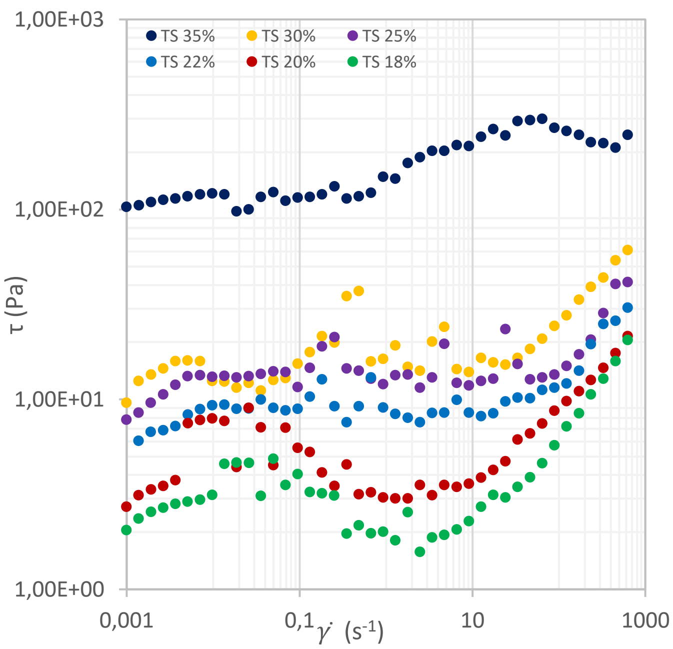


Fig. 4. Stress evolution curves as a function of the shear rate at 55°C

As previously observed (Fig.3), the more concentrated the sample, the higher the stress. By comparing the stress values obtained in Figures 3 and 4, we observe that the stress decreases with the temperature increase with the sole exception of 35% TS. The figure 4 also shows the existence of threshold stresses obtained at a shear rate of 0.001 s^{-1}. These threshold constraints are 2.05 Pa; 2.72 Pa; 6.05 Pa; 7.79 Pa; 9.57 Pa and 103 Pa, respectively for 18%, 20%, 22%, 25%, 30% and 35% TS. We see a significant increase in the threshold constraint (factor 10) between 30% and 35%, from 9.57 to 103 Pa.

The increase in temperature does not affect the overall speed of the curves. However, it influences the values of the threshold constraints. We observe (Fig. 3 and 4) that the higher the temperature the lower the threshold stress values. From the operational point of view, the increase in temperature in the digesters reduces the effort to be provided for the mixing of the medium, and thus energy and handling savings.

3.2. Determination of the limit viscosity

3.2.1. Shear flow measurement at 37°C

The figure 5 shows the evolution of viscosity as a function of the measured shear velocity following a rising ramp from 0 to 200 s^{-1} to 37°C for samples of 18%, 20%, 22%, 25%, 30% and 35% TS. We observe that viscosity decreases with increasing shear velocity. The curves represent a decrease in viscosity from 6.1×10^3 to 3.61×10^{-2} Pa.s; from 5.58×10^3 to 3.43×10^{-2} Pa.s; from 6.99×10^3 to 4.39×10^{-3} Pa.s; from 11×10^3 to 7.5×10^{-2} Pa.s; from 17×10^3 to 8.73×10^{-2} Pa.s and from 41×10^3 to 29.2×10^{-2} Pa.s, respectively for 18%, 20%, 25%, 30% and 35% TS.

The first apparent viscosity value represented on the rheograms corresponds to the sample limit viscosity, the viscosity value for which the first plateau of the rheograms is broken. Indeed, the viscosity limit is obtained towards a zero-shear rate, at the break of the plateau representing a Newtonian behavior. The measured viscosity limit are 6.1×10^3 Pa.s; 5.58×10^3 Pa.s; 6.99×10^3 Pa.s; 11×10^3 Pa.s; 17×10^3 Pa.s and 41×10^3 Pa.s, respectively for 18%, 20%, 22%, 25%, 30% and 35% TS.

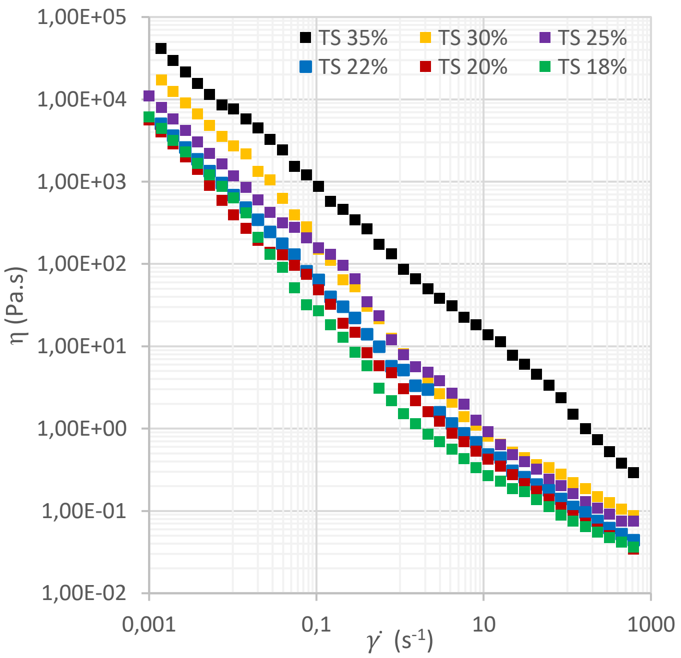


Fig. 5. Viscosity evolution as a function of shear velocity at 37°C

3.2.2. Shear flow measurement at 55°C

For shear flow measurements at a temperature of 55°C (Fig. 6), we observe, as in Figure 5, that viscosity decreases with the increase in shear velocity. Viscosity decreases from 2.04x10^3 to 3.26x10^{-2} Pa.s, from 3.7x10^3 to 3.42x10^{-2} Pa.s, from 4.38x10^3 to 4.82x10^{-2} Pa.s, from 7.78x10^3 to 6.57x10^{-2} Pa.s, from 10.54x10^3 to 6.72x10^{-2} Pa.s and from 103x10^3 to 46.5x10^{-2} Pa.s, for dry matter contents of 18%, 20%, 25%, 30% and 35% TS respectively. The viscosity limit obtained are 2.04x103 Pa.s; 3.7x10^3 Pa.s; 4.38x10^3 Pa.s; 7.78x10^3 Pa. s; 10.54x10^3 Pa.s and 103x10^3 Pa.s, respectively for 18%, 20%, 22%, 25%, 30% and 35% TS.

As observed in the evolution of the stress (Fig. 3 and 4), the increase in temperature does not affect the overall velocity of the viscosity evolution curves (Fig. 5 and 6). The temperature, on the other hand, influences the limit viscosity of the sample. The higher the temperature, the lower the apparent limit viscosity values. The increase in temperature, therefore, made it possible to reduce energy for brewing and save energy and handling.

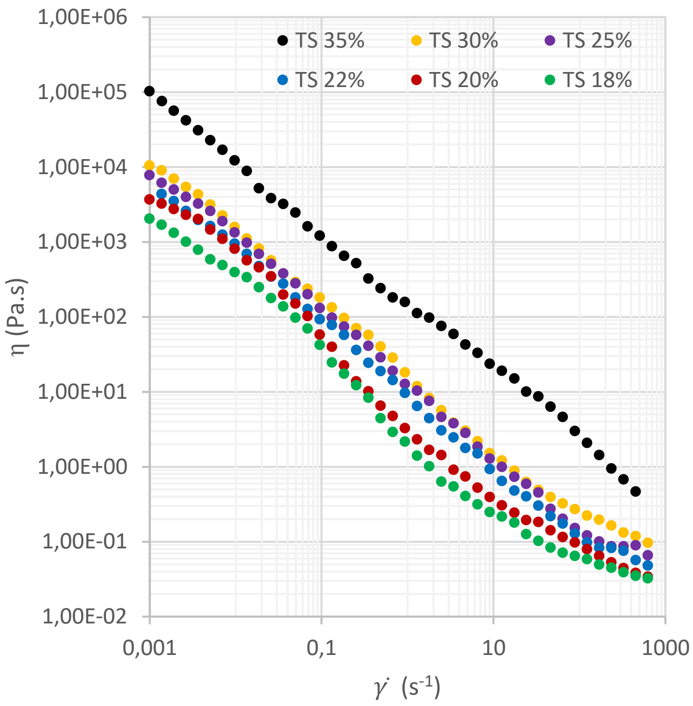


Fig. 6. Viscosity evolution as a function of shear velocity at 55°C

3.3. Determination of the rheological behaviour of residues at 37 and 55°C

The rheograms from Figure 5 obtained at 37°C were compared to different rheological models describing materials with threshold behaviour (Bingham and Herschel-Bulkley). We observe a good correlation between the rheograms and the Herschel-Bulkley model (fig. 7). The Herschel-Bulkley model is described by the equation below [38]:

$$\tau = \tau_0 + k \cdot \dot{\gamma}^n \qquad \textit{(eq.1)}$$

With τ stress, τ_0 the threshold stress, k the consistency and n the pseudoplastic index (flow index), and $\dot{\gamma}$ the shear velocity.

The correlation between the rheograms and the Herschel-Bulkley model is even better when the dry matter content of the samples is highest. The overall shape of the curves correlates well with the Herschel-Bulkley model. On the other hand, this correlation is less good below 35% TS for the shear rate higher than 10 s^{-1}.

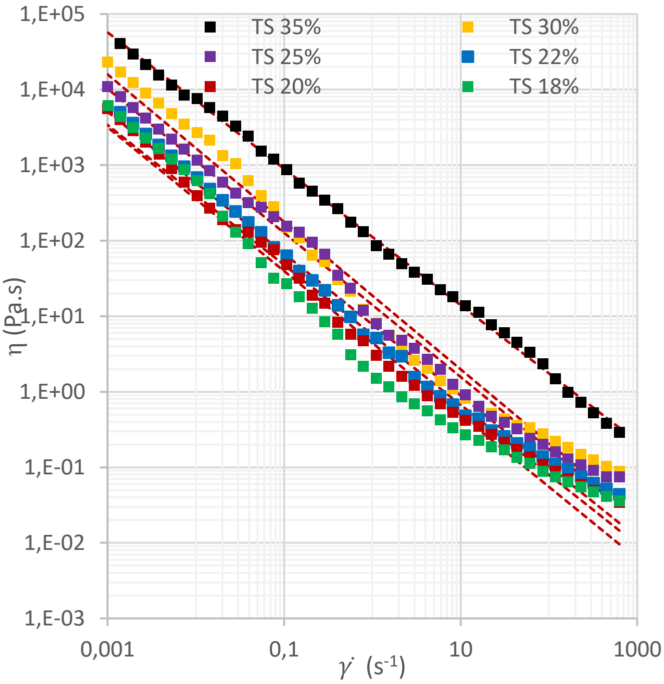


Fig. 7. Viscosity evolution as a function of shear velocity at 37°C, compared to the Herschel-Bulkley model.

The figure 8 shows the comparison of the rheograms obtained at 55°C (Fig. 6.) to the Herschel-Bulkley model. We see a good correlation between the different curves and the increase in dry matter content.

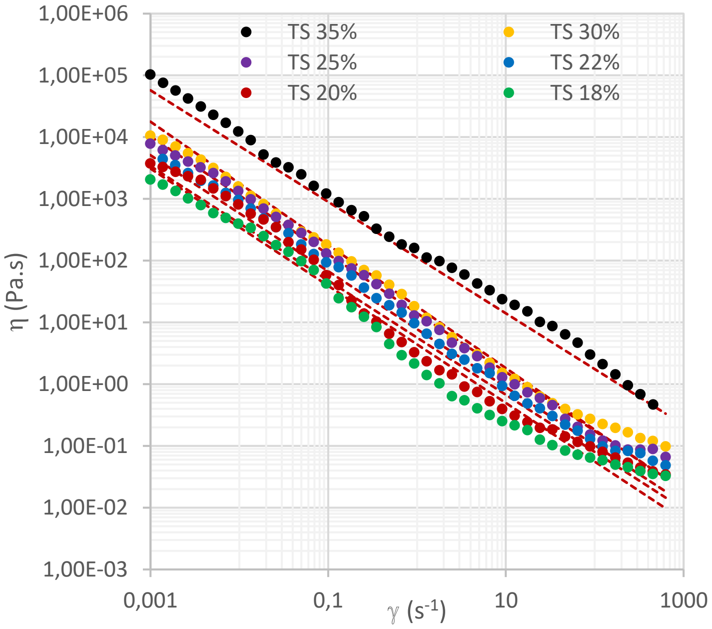


Fig. 8. Viscosity evolution as a function of shear velocity at 55°C, compared to the Herschel-Bulkley model.

As in Figure 7, the correlation between the rheograms and the Herschel-Bulkley model is even better at 35% TS. On the other hand, the correlation is less good for low dry matter contents, when the shear rate reaches 10 s^{-1}.

It should be noted that the Herschel-Bulkley model represents the rheological behaviour of the threshold fluids and according to the equation described above (eq.1), when the flow index n is less than 1, the fluid is rheofluidifying and when this index is greater than 1, the fluid is rheothickening. The flow indices calculated for the different measurements are all less than 1 (Tab.1). The threshold stress τ_0 and the consistency k increase with the dry matter content, while the flow index n decreases. These model parameters confirm that the higher the dry matter content of the residue, the higher the threshold stress and viscosity (Tab. 1).

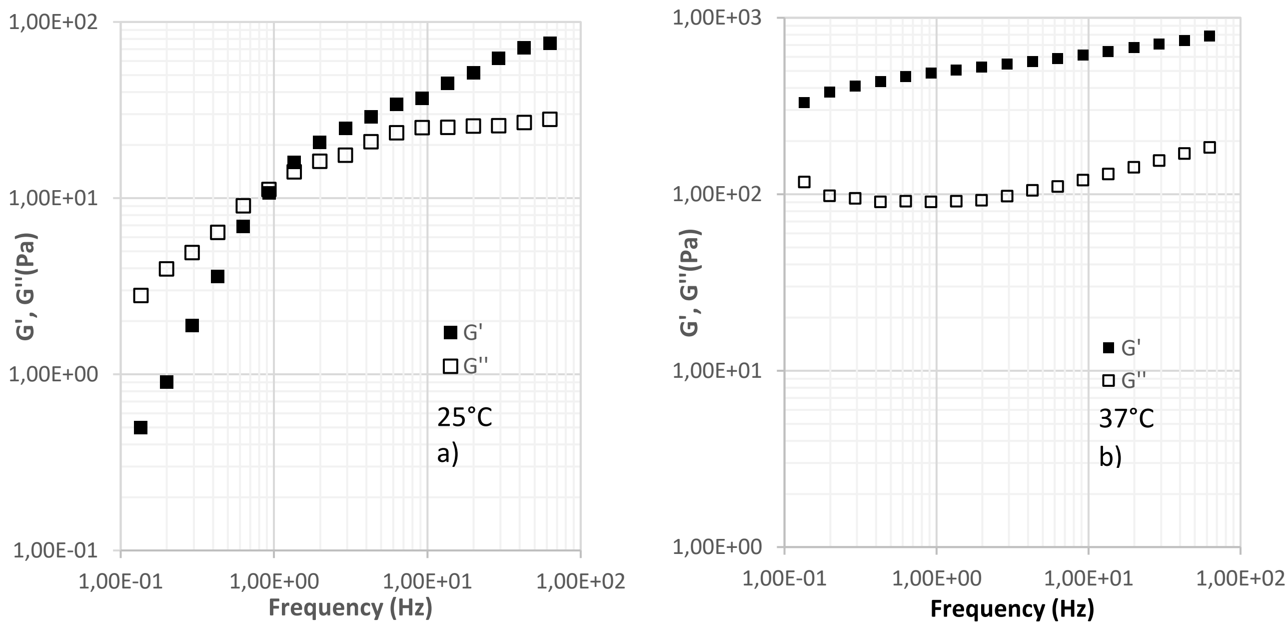
Tab. 1. Herschel-Bulkley model parameters at 37°C and 55°C

% TS	37°C			55°C		
	τ_0	n	k	τ_0	n	k
18	5.44	0.70	0.69	1.9	0.71	0.71
20	5.46	0.69	0.72	2.1	0.70	0.77
22	9.13	0.64	0.74	5.4	0.63	0.79
25	20.67	0.61	0.78	6.8	0,59	0.82
30	50.21	0.51	0.81	7.3	0.52	0.84
35	59.59	0.42	0.84	8.7	0.44	0.85

The results of the flow tests allow us to state that the potato residues studied are characterized by rheofluidifying material behaviour. Rheofluidifying materials are characterized by a decrease in viscosity under stress. Taking this behaviour into account would allow to adapt the brewing modes for optimization of digestion. Knowledge of the rheological behaviour of digestion residues is also an essential and useful parameter for the design and performance of digesters

3.4. Determination of rheological behaviour in oscillations

Oscillating measurements to characterize the viscoelastic behaviour of residues at 25°C, 37°C and 55°C (Fig. 9. a. b. c) showed notable differences with the evolution of the measuring temperature.



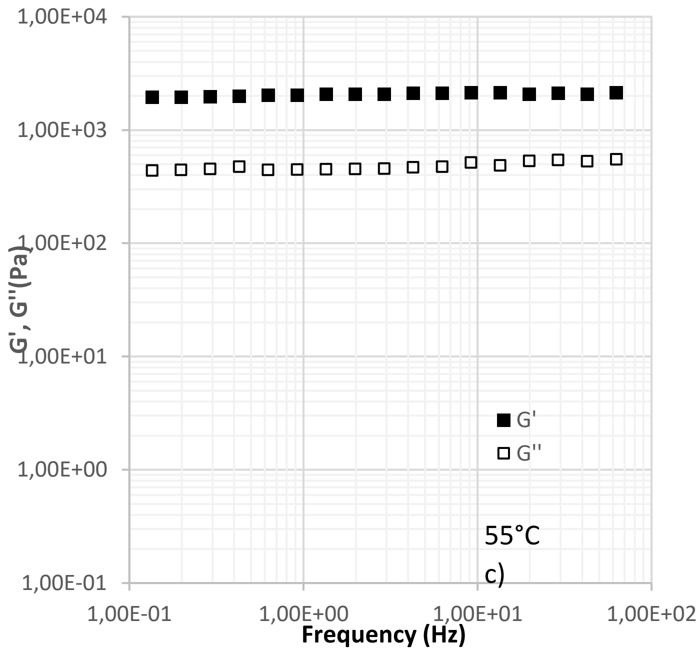


Fig. 9. Elastic G' and viscous G'' modulus, 22% TS measured at (a) 25°C, (b) 37°C and (c) 55°C

At 25°C, the curves of the G' and G' loss modulus have two phases (Fig. 9. a.): a first phase where G' is less than G' for frequencies at 1 Hz and a second phase where G' is more than G'' beyond 1 Hz. The inflection point between these two curves is at 1 Hz. The overall velocity of the curves shows an increase of G' and G'' modulus with frequency.

For measurements at 37°C (Fig. 9. b.), we observe a single-phase and there is no crossing between G' and G'', the modulus G' is predominant at G''. At 55°C, G' is always predominant at G'' (Fig. 9. c.), G' and G'' forming a plateau, characteristic of material that behave like a gel [39].

4. Discussion

4.1. Evolution of threshold stress with dry matter content and temperature

The shear flow measurements carried out on potato residues of 18 to 35% TS according to a viscosity ramp of 0 to 200 s^{-1} at 37°C and then at 55°C, showed the existence of threshold stress. As observed by Battistoni and coll. [26, 27, 40], this threshold constraint increases with the dry matter content. These authors determined a correlation between the threshold stress and the dry matter content for flow measurements on municipal waste using a cylindrical geometry and concluded that the inclusion of the threshold stress in the sizing and monitoring of digesters [26, 27, 40].

Mori and coll. [28] have used two types of geometries, simple cylindrical (quilt) and double cylindrical, to carry out flow measurements on sewage sludge. These authors concluded that the measurement geometry influenced the results and showed that the cylindrical geometry was the most suitable for measuring the threshold stress for these types of pasty waste. They hypothesized that a Vane geometry would be better adapted to these very heterogeneous pasty residues. This was demonstrated through this study by the use of Vane geometry to characterize the rheological behaviour of potato residues from 18 to 35% TS.

For shear flow measurements, we observe an increase in the threshold stress from 6.13 Pa to 57.2 Pa and from 2.05 Pa to 103 Pa, respectively for measurements at 37°C and 55°C. These results are comparable to those obtained by Battistoni which observed an increase in the threshold stress of 23.2 Pa at 57.2 Pa at 37°C and 57 Pa and 102 Pa at 55°C, respectively for 30% and 35% TS, for measures on municipal waste [27]. As observed by Pevere and coll. [41, 42], which have a simple cylindrical geometry, the threshold stress decreases with the temperature increase except at 35% TS. The threshold constraint changes from 23.2 Pa to 9.57 Pa for 37°C and 55°C, respectively.

Indeed, several authors have pointed to the impact of temperature on the evolution of the threshold stress [43, 44, 45]. For these authors, the decrease in the threshold stress with the increase in temperature could be explained by dilution of the solid particles under the influence of temperature, thus facilitating the flow of residues and consequently a decrease in the threshold stress. On the other hand, we observed an increase in the threshold stress to 35% TS from 57.2 Pa to 103 Pa, respectively for 37°C and 55°C. This result could be explained by the effect of evaporation during high-temperature measurements for highly concentrated samples.

Garcia-Bernet and coll. [11] which used collapse tests on sewage treatment plant sludge obtained a threshold stress increase of 250 Pa to 1500 Pa, a factor increase of 6, for 22% to 35% TS respectively, whereas we observe in our study an increase in the threshold constraint factor of 10 between 30% and 35% TS, from 9.57 Pa to 103 Pa. (Fig.4.). They concluded that the higher the dry matter content, the higher the effort required to release the material (the threshold stress) [11, 26, 27, 40, 45].

4.2. Evolution of viscosity limit with dry matter content and temperature

For the same shear flow measurements, different boundary viscosities were determined according to dry matter content and temperature. As for the threshold stress, the limit viscosity, corresponding to the viscosity value for which the first plate of the rheograms is broken, increases with the dry matter content. Thus, for potato residue shear tests of 18 to 35% TS measured at 37°C and 55°C, the apparent boundary viscosities decrease respectively by $6.1x10^3$ Pa.s to $41x10^3$ Pa.s and $2.04x10^3$ Pa.s to $103x10^3$ Pa.s. This decrease in the viscosity limit was also observed by several authors who studied the rheological behaviour of sewage treatment plant sludge and municipal waste [41, 42, 44, 46, 47].

There is also a decrease in viscosity as the shear velocity increases (Fig. 5, 6). This decrease in viscosity was observed by several authors [11, 42, 48]. According to these authors, this rheological behaviour could be explained by the fact that the increase in shear rate would allow the molecular chains of the substrate to stretch and orient themselves by forming parallel layers in the solution. This molecular alignment thus facilitates slippage between the particle chains and results in reduced viscosity for rheofluidifying materials [11, 42, 48]. This dependence of viscosity on shear velocity is characteristic of a non-Newtonian behaviour [42, 49].

As observed through our study, other authors have shown that temperature has an impact on the limit viscosity [42, 45, 49]. Indeed, the viscosity limit decreases with the increase in temperature. This is explained by the dilution of the solid particles under the effect of temperature, thus facilitating the flow of residues.

These authors used cylindrical geometry (Couette), double cylindrical geometry and for others, a collapse test to measure the rheological behaviour of residues used in dry digestion such as sewage sludge, farm waste, and municipal waste and have obtained results that confirm our observations. Some of these techniques have shown their limitations in the case of collapse tests, which lead to total solid levels above 25% of sample failure problems [11]. As for cylindrical or double cylindrical simple geometries without cross-section, some phenomena of sample slippage have sometimes been observed, a phenomenon which is very limited in the case of Vane geometry [26, 27, 29, 39, 42].

4.3. Rheological behaviour of potato residues

This study described a rheofluidifying threshold behaviour of potato residues used in dry anaerobic digestion using Vane geometry. The results of the measurements were compared with those obtained by several authors who used other measurement techniques such as cylindrical geometry, double cylindrical geometry or collapse tests [11, 27, 28, 30, 42, 45, 47, 50]

The rheograms were compared with the different rheological models available to determine the rheological nature of the residues. The curves were correlated with the Herschel-Bulkley rheological model [40, 45]. The flow index of the Herschel-Bulkley equation is less than 1 for all the samples measured, which allows us to conclude that the material studied is not only rheofluidifying but has a flow threshold or threshold stress. These observations were also made by Mori and coll., and Baudez and coll. [28, 47], which carried out flow measurements to characterize the rheological behaviour of sewage treatment plant sludge. By contrast, Battistoni and coll. [26, 27] have correlated with the Bingham model, another model describing flow-threshold materials.

Thus, using a more reliable measuring geometry, because limiting the sliding effects, it was possible to determine effectively the rheological behaviour of potato residues from 18 to 35% TS. This Vane geometry has enabled us to overcome operational constraints such as heterogeneity, high TS levels, particle size, and especially the sliding effects of the sample.

4.4. Viscoelastic behaviour

The viscoelastic behaviour of a material is intermediate between that of an elastic solid symbolized by a spring and that of a viscous Newtonian liquid symbolized by a shock absorber [51]. The determination of this characteristic for digestion residues makes it possible to understand their internal structure. Indeed, when its viscous characteristics predominate, the material resists a shear flow and exhibits a deformation that increases linearly when a stress is applied. On the other hand, when its elastic characteristics predominate, the material deforms with stress and quickly returns to its original state once the stress has been removed [51].

Results of measurements of viscoelastic properties of potato residues performed in oscillations at temperatures of 25°C, 37°C and 55°C (Fig.9), show that the evolution of the elastic or storage modulus G’ and the viscous or loss modulus G’’ is a function of temperature. These results are consistent with those obtained by Ren and coll., Agbenorhevi and coll., and Grundy and coll. [52-54].

In particular, we observed a crossing of G and G’’ at 25°C at 1 Hz, at which frequency the transition constraint is reached. Grundy and coll. [54] observed the same results during oscillation measurements of oat residue, a cross between G’ and G’’ modulus at 25°C at a frequency scan between 0.1 and 1000 rad s^{-1}.

In contrast, at temperatures 37 and 55°C and within the measured frequency range, G’ and G’’ evolve with the frequency without crossing. These results are also consistent with those obtained by various authors [55, 39] who suggest that at these high temperatures the G’ storage modulus is predominant over the G’ loss modulus for fluidifying fluids. At 37°C, the elastic G’ and viscous G’’ modulus increase linearly with frequency. However, at 55°C, G' and G'' modulus are constant according to frequency. According to Shrestha and coll. [39], the "flat" profile of the elastic G' and G'' modulus is characteristic of a material that behaves like a gel. For example, at 55°C, apple residue behaves like a gel.

5. Conclusion

This study showed that with a rotating rheometer it was possible to accurately characterize the rheological behaviour of residues with high dry matter content in anaerobic dry digestion using a Vane geometry. The Vane geometry spacers increase the contact surface between the geometry and the sample and considerably reduce the sliding effects observed with standard geometries (cone-plane, plane-plane and cylindrical). Using this geometry, we observed the following results during flow and oscillation measurements of potato residue samples with a total solid content of 18 to 35% at 25°C, 37°C and 55°C:

- the existence of threshold stress and limit viscosity which increase with dry matter rate;
- the reduction of the threshold stress and the limiting viscosity with the increase in temperature;
- the satisfactory correlation between the rheograms obtained and the Herschel-Bulkley model, with the flow index less than 1;
- different changes in the G' and G'' modulus depending on the temperature, a cross of G' and G'' at 25°C, G' above G'' at 37°C and G' and a "flat" profile at 55°C, characteristic of a gel.

In short, Vane geometry has enabled us to overcome operational constraints such as heterogeneity, high TS levels, particle size, and in particular the sliding effects of the sample. Thus, we were able to determine the rheofluidifying behaviour of potato residues at 37°C and that behaves like a gel at 55°C. Biochemically, 37°C is the optimum temperature of a mesophilic reactor while 55°C is the optimum temperature of a thermophilic reactor. Under these conditions, these rheological behaviour would affect both the design parameters and the operating conditions such as the feeding and agitation of the digesters, the biochemical balances of mass transfer, and residue mobility in dry anaerobic digestion.

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