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1 The impact of temperature on the rheological behaviour of anaerobic digested sludge

2  
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10  
11 Abstract

12  
13 The rheological properties of municipal anaerobic digested sludge rheology are temperature  
14 dependent. In this paper, we show that both solid and liquid characteristics decrease with  
15 temperature. We also show that the yield stress and the high shear (Bingham) viscosity are the two  
16 key parameters determining the rheological behaviour. By normalising the shear stress with the  
17 yield stress and the shear rate with the yield stress divided by the Bingham viscosity, a master  
18 curve was obtained, independent of both temperature and concentration. We also show that the  
19 rheological behaviour is irreversibly altered by the thermal history. Dissolution of some of the solids  
20 may cause a decrease of the yield stress and an increase of the Bingham viscosity. This result  
21 suggests that the usual laws used to describe the thermal evolution of the rheological behaviour of  
22 fluids are no longer valid with anaerobic digested sludge. Finally, the impact of temperature and  
23 thermal history have to be taken into account for the design of engineering hydrodynamic  
24 processes such as mixing and pumping.

25  
26 Keywords

27  
28 Digested sludge, Rheology, Structural change, Temperature, Thermal agitation, Viscosity, Yield  
29 stress

30

1

## 2 Introduction

3

4 Anaerobic digestion is one of the most important processes for reducing sludge volume by  
5 reducing of about 30% of the organic load (part of the solid organic matter is converted into gas)  
6 and for producing biogas. To be efficient, anaerobic digestion needs to satisfy at least three  
7 conditions: (i) controlled temperature, between 35 and 37°C for mesophilic processes or between  
8 55 and 58°C for thermophilic processes, (ii) homogeneous mixing conditions and (iii) no dramatic  
9 variation in organic load. To control temperature, biogas is partly used for heating large digesters  
10 through heat exchangers installed in recirculation loops (Gerardi, 2003). If possible sludge also has  
11 to be initially pre-heated to the appropriate temperature and raw sludge is sometimes mixed with  
12 digested sludge before entering the digester (Keeper, 1959). In other cases, the digester mixing  
13 system ensures that the cold, newly introduced sludge, is mixed with the warm older solids and  
14 the bacteria. Finally, the entire digester volume needs to be turned over once every 3-4 hours  
15 using pumps and recirculation loops. It is imperative that digester design take into account the  
16 energy requirements needed to maintain these essential operating conditions, such as the power  
17 consumption of pumps and mixing systems, which are directly related to the sludge rheology  
18 (Slatter, 2011).

19 Because flow velocities within the digester and the recirculation loops are not of the same order of  
20 magnitude, a better understanding of the rheological properties of digested sludge over a wide  
21 shear rate range would provide a sound basis for the efficient design of digesters. Monteiro (1997)  
22 showed that anaerobic digested sludge rheology can be described using the Herschel-Bulkley  
23 model, for which the rheological characteristics decreased with the degree of fermentation. More  
24 recently, Baudez et al. (2011a) highlighted a more complex behaviour with shear banding at low  
25 shear rates and a viscosity plateau at very high shear rates. However, these studies did not focus  
26 on the temperature dependence of the rheology of digested sludge, which would fundamentally  
27 affect the flow properties and consequent operating conditions of the digester.

28 Since the pioneering work of Manoliadis and Bishop (1984) who pointed out a decrease of  
29 rheological characteristics similar to what is usually observed with Newtonian fluids, there is very  
30 little literature on the impact of temperature on municipal sludge rheology. In a recent paper,

1 Baudez et al. (2011b) showed that the decrease of viscoelastic properties is proportional to the  
2 decrease of water viscosity with increase in temperature, suggesting thermal motion may be the  
3 key factor, in agreement with what was observed by Manoliadis and Bishop (ibid.) while Dieude-  
4 Fauvel et al. (2009) considered a VTF model (Vogel-Tamman-Fulcher) on secondary sludge.  
5 However, because municipal sludge is mainly composed of water, mineral particles and organic  
6 matter (polymeric and dissolved), one can imagine that the temperature dependence of sludge  
7 would be similar to what it is observed with mineral suspensions and organic polymers. If there is a  
8 general agreement in polymer science to consider that temperature decreases rheological  
9 characteristics, de Kretser and Scales (2008) showed that the solid characteristics of mineral  
10 suspensions can increase with an increase of temperature while liquid characteristics decrease.  
11 In this paper, we revisit the temperature dependence of digested sludge, both in the liquid and the  
12 solid regimes and we propose that thermal motion has the most important impact. We also show  
13 that it is not the only mechanism impacting sludge rheology. Similarities between the rheological  
14 behaviour of digested sludge at different temperatures were found, but temperature also  
15 irreversibly modified sludge structure. After being heated and cooled down, digested sludge  
16 showed a lower yield stress but a higher consistency index than the initial material, at the same  
17 temperature. Apart from the fundamental contribution this makes to understanding sludge  
18 behaviour, such behaviour has to be taken into account for the practical design and operation of  
19 anaerobic digesters, especially heat exchangers and pipe flow.

20

## 21 Material and methods

22

23 Anaerobic digested sludge was sampled at the Mount Martha waste water treatment plant  
24 (Melbourne, Victoria, Australia) at the outlet of the mesophilic anaerobic digester number 1. Its  
25 initial solids concentration was at  $18.5 \text{ g.L}^{-1}$  and was also gently concentrated to 25.5, 32.5 and 49  
26  $\text{g.L}^{-1}$  by using a Buchner vacuum. To reach these concentrations, sludge was basically poured into  
27 a cylinder onto a filter paper and vacuum suction was applied at the pressure of -0.5 bars. During  
28 all the filtration process, sludge was softly mixed to let the solid particles in suspension, avoiding  
29 clogging of the filter paper. Solid concentration was determined by drying the sludge at  $105^\circ\text{C}$  for  
30 24 hours.

1 Because fermentation alters sludge rheological characteristics mainly in the first 10 days of storage  
2 (Baudez and Coussot, 2001), and although anaerobic digestion is a stabilization process, our  
3 samples were stored at 4°C for 30 days before experiments, to ensure no temporal variability,  
4 allowing us to use the same material over several days. This technique was successfully used by  
5 Curvers et al. (2009).

6  
7 Rheological measurements were performed with a stress-controlled DSR200 from Rheometric  
8 Scientific, connected to a temperature controlled water bath. The rheometer was equipped with a  
9 cup and bob geometry (inner diameter: 29 mm, outer diameter: 32 mm, length: 44 mm). In order to  
10 highlight its impact on the rheological behaviour of digested sludge, temperature was varied from  
11 10 to 80°C using a temperature controlled water bath. To avoid evaporation, sludge was covered  
12 with a thin film of Newtonian oil (oil and sludge are not miscible, as evidenced by oil removal  
13 processes in wastewater treatment plants).

14 Before each measurement, the sludge was presheared for 10 minutes at a shear rate of  $1000 \text{ s}^{-1}$   
15 then left at rest for 10 minutes before each measurement. With such a procedure, measurements  
16 were repeatable and reproducible (Baudez and Coussot, 2001; Baudez et al., 2011a).

17 Because of shear banding, direct determination of the yield stress has to be conducted with  
18 caution in the sense that the data we could consider as the yield stress would be basically the  
19 stress at which shear localisation occurs. Thus, we focused our measurements on (i) the solid  
20 characteristics by applying a linear ramp of increasing shear stress as long as the strain remained  
21 limited and (ii) the liquid characteristics by applying decreasing stress ramps, starting from a high  
22 stress corresponding to a shear rate of approximately  $1000 \text{ s}^{-1}$  or lower for the less concentrated  
23 sludge (to avoid turbulent conditions).

24 The solid characteristics were only determined on the most concentrated sludge and below 60°C  
25 because at 80°C the measured values were too low to be considered consistent, with respect to  
26 the rheometer characteristics.

27 It must be noted that what will be called yield stress in the following should be seen as the  
28 extrapolation of the flow curve towards zero shear rate: it basically represents the limit below which  
29 there is no steady state flow.

1 Lastly, all the data fits shown in this paper were obtained using Excel software and its solver macro  
2 based on the “least squares” difference between model and experiment.

3

4 Definition of the general behaviour of anaerobic digested sludge

5 The following results will be interpreted according to the behaviour of digested sludge determined  
6 by Baudez et al. (2011a):

7 - a linear viscoelastic behaviour in the solid regime, well represented by a generalised Kelvin-Voigt  
8 model, with a wide relaxation times spectrum modelled by a stretched exponential:

$$9 \quad \gamma(t) = \tau \cdot \frac{1}{G} \cdot \left(1 - \exp\left(-(\lambda t)^m\right)\right) \quad (1)$$

10 where  $\gamma$  represents the strain,  $\tau$  the stress and  $\lambda = \frac{G}{\mu}$  with  $G$  (representative of the purely  
11 elastic spring) and  $\mu$  (representative of the purely viscous damper) the usual parameters of a  
12 Kelvin-Voigt model.

13 - a shear-thinning behaviour with a constant high-shear viscosity in the liquid regime, well  
14 represented by a Herschel-Bulkley model coupled with a Bingham model (Baudez et al., 2011a):

$$\begin{aligned} \tau &= \tau_c + K \cdot \dot{\gamma}^n + \alpha_0 \cdot \dot{\gamma} \\ \Leftrightarrow \frac{\tau}{\tau_c} &= 1 + \frac{K}{\tau_c} \cdot \dot{\gamma}^n + \frac{\alpha_0}{\tau_c} \cdot \dot{\gamma} \\ 15 \quad \Leftrightarrow \frac{\tau}{\tau_c} &= 1 + \xi \cdot \Gamma^n + \Gamma \quad (2) \\ \Gamma &= \frac{\alpha_0}{\tau_c} \cdot \dot{\gamma}, \quad \xi = \frac{K}{\tau_c} \cdot \left(\frac{\tau_c}{\alpha_0}\right)^n \end{aligned}$$

16 where  $\tau_c$  represents the extrapolated limit below which there is no steady-state flow (improperly  
17 named yield stress in the following),  $K$  the consistency index,  $\alpha_0$  the high shear viscosity rate  
18 (named Bingham viscosity in the following) and  $n$  the power-law index. Note that such a  
19 dimensionless form was previously used by Coussot (1995) with mineral suspensions. It comes  
20 from the identical shape of the flow curves. Thus, it is natural to scale the shear stress by the  
21 yield stress, such that all curves go through the same point  $(\dot{\gamma} = 0; \frac{\tau}{\tau_c} = 1)$ . This means we  
22 probably smooth the solid interactions since  $\tau_c$  represents the strength of particle interactions ‘at  
23 rest’. In parallel it is necessary to scale the shear rate by a factor also related to viscous

1 dissipation. This may be done by assuming that for high shear rates the energy dissipation  
 2 resulting from interactions between solid particles becomes negligible compared to hydrodynamic  
 3 dissipation. Under these conditions, like Coussot (1995) did for mineral suspensions, it is natural to  
 4 use a dimensionless number,  $\Gamma$  defined as  $\Gamma = \frac{\mu}{\tau_c} \cdot \dot{\gamma}$  where  $\mu$  is the viscosity of the equivalent  
 5 suspension of force-free particles in water. Following the work of Quemada (1998), we assumed  
 6 that at high shear particles are independent of each others, so  $\mu = \alpha_0$ .

7

8 Results and discussion

9 The impact of temperature on the solid characteristics

10

11 First focusing on increasing shear stress sweep, the model defined by Eq (1) is accurate at low  
 12 shear stress (fig. 1), and the higher the temperature, the higher the shear strain at a given shear  
 13 stress (fig. 1). Only the elasticity appears to change significantly with the temperature (Table 1),  
 14 and its changes with temperature are proportional to those of water viscosity (Fig. 2), indicating  
 15 that there are probably the same molecular movements which are involved in both water viscosity  
 16 and elastic coefficient changes with the temperature.

17

18 **Table 1: Values of the viscoelastic parameters given by Eq (1) at different temperatures.**

	10°C	25°C	40°C	60°C
G/100 [Pa]	0.559	0.385	0.263	0.167
$\lambda$ [t <sup>n</sup> ]	1.488	1.489	1.489	1.499
n [1]	0.416	0.421	0.374	0.479

19

20 The constant value of the parameter  $\lambda$  also indicated that both the elastic and viscous  
 21 characteristics of the Kelvin-Voigt model follow the same relationship regarding temperature.,  
 22 mainly caused by thermal agitation (due to the proportionality with water viscosities).

23

24 The impact of temperature on the liquid characteristics

25

1 The same temperature evolutions are observed with the flow curves, in the liquid regime (fig. 3):  
2 the higher the temperature, the higher the shear rate (and so, the lower the apparent viscosity) at a  
3 given shear stress. Sludge becomes less and less viscous as the temperature increases.

4 In the dimensionless form of (2), all the flow curves resolve to a single master curve, independent  
5 of both temperature and concentration (fig. 4), indicating that the (extrapolated) yield stress and the  
6 Bingham viscosity are the two key parameters to characterise the flowing behaviour of digested  
7 sludge. Note that the consistency index  $K$  appears to be proportional to  $\tau_c$  (data not shown) but  
8 at this stage, we have no explanation for that.

9 Moreover, in the temperature range of [10-60°C], both the yield stress (fig. 5) and the Bingham  
10 viscosity decreased, this latter followed a linear relationship with water viscosity (fig. 6), meaning  
11 thermal agitation also had a major influence in the change of the liquid characteristics. Surprisingly,  
12 the temperature changes of  $\tau_c$  are not proportional to the water viscosity changes, which could  
13 have been expected because of the impact of temperature on the elasticity. This probably comes  
14 from the determination of  $\tau_c$  which is not a 'real' rheological characteristic but rather a fitting  
15 parameter (due to shear banding,  $\tau_c$  cannot be measured directly).

16 Following the results of Rodd et al. (2001), Baudez (2008) and Baudez et al. (2011a), the yield  
17 stress and the Bingham viscosity can be expressed as follows:

$$18 \quad \tau_c = a \cdot (\phi - \phi_0)^m \quad (3)$$

$$19 \quad \alpha_0 = \mu_0 \cdot \exp(\beta \cdot \phi) \quad (4)$$

20 where  $\phi_0$  is the lowest concentration below which there is no yield stress (also called gel point in  
21 polymer science),  $m$  is a parameter which can be related to the fractal dimension of sludge flocs  
22 (Pignon et al., 1996),  $\mu_0$  is the viscosity of the liquid medium, and  $a$  and  $\beta$  are model  
23 parameters.

24 From the fitted values of  $\tau_c$  and  $\alpha_0$  (see appendix for all the rheological values), we have  
25 determined the numerical values for the parameters of (3) and (4):  $\mu_0$  and  $\beta$  of (4) and  $a$  and  $m$   
26 of (3) decreased when the temperature increased while  $\phi_0$  increased (fig. 7 and table 2)).

27



1 **Table 2: Values of parameters of (3) and (4) at different temperatures.**

	10°C	25°C	40°C	60°C
$\phi_0$ [%]	0.787	0.792	0.825	0.948
$a$ [Pa]	0.0855	0.0817	0.0803	0.0654
$m$ [1]	2.482	2.366	2.352	2.153
$\mu_0$ [mPa.s]	1.826	1792	1.777	1.762
$\beta$ [1]	0.6935	0.605	0.517	0.426

2

3 Assuming that  $m$  reflects the fractal dimension of the flocs (Pignon et al., 1996), its decrease  
 4 means that the flocs became less dense and compact (Li and Ganczarczyk, 1989). From a more  
 5 fundamental perspective, this also means that if flocs are seen as spherical agglomerates of  
 6 particles, the decrease in fractal dimension implies that (i) floc surface remained constant while the  
 7 number of voids increased or (ii) floc surface increased. In both cases, it would mean that sludge  
 8 structure is modified when the temperature increases..

9

10 The impact of thermal history

11 If sludge is heated and cooled before measurement, its rheological behaviour is irreversibly altered  
 12 compared to its initial behaviour with no such thermal history (fig. 8): the hotter the preheat, the  
 13 smaller the yield stress and the higher the Bingham viscosity (fig. 9), the consistency being almost  
 14 constant. This result strengthened the assumption we previously made and it also indicated that  
 15 the structural changes induced by an increase of temperature are globally irreversible.

16 At this time, it must be strongly emphasised that our initial anaerobic digested sludge was picked  
 17 up at the outlet of a mesophilic digester which runs between 35 and 38°C. So our results obtained  
 18 at the temperature below the digester operating temperature were already influenced by the  
 19 previous thermal history of the sludge. That's may explain why the variation of the rheological  
 20 characteristics are weak below 40°C and also why there is no significant change in fig.6 between  
 21 25 and 40°C: in this range, the yield stress and the water viscosity appear to be proportional (data  
 22 not shown), suggesting that (i) thermal fluctuation is the main factor responsible of the yield stress  
 23 decrease below 40°C and (ii) the alteration of the composition with the temperature is irreversible.

1 However, by reducing shear rate and shear stress to dimensionless form as proposed in Eq(2), a  
2 master curve was also obtained (fig. 10), indicating that ultimately the yield stress and the Bingham  
3 viscosity can again be considered as the two key parameters which drive the rheological behaviour  
4 of anaerobic digested sludge and its (physical) evolution with temperature.

5  
6 As suggested above, thermal history apparently irreversibly modified the structural characteristics  
7 of the sludge, because of the change of the rheological characteristics, but did not change the total  
8 solid concentration. Indeed, we did not find significant changes in the total solid concentration  
9 regarding temperature history, which is quite logical in the sense that water content determination  
10 imply to heat sludge at a higher temperature that we did. So, assuming the total solids  
11 concentration was kept constant by preheating the sludge, from Eq (4), we can deduce the  
12 increase of Bingham viscosity originates only from an increase in  $\mu_0$  (since  $\beta$  decreases with  
13 temperature) and in parallel, the yield stress decreases.

14 Keeping in mind that the fractal dimension of the flocs decrease with the temperature, if the  
15 viscosity of the medium increased while the yield stress decreased, it implies that there is a  
16 transfer from solids constituents to dissolved constituents, the total solids concentration being  
17 constant. This assumption has to be confirmed, by measuring the liquor viscosity before and after  
18 heating. However such a result was already reported by Appel et al. (2010) who pointed out that  
19 temperature increase promoted dissolution of the main organic (proteins, carbohydrates and  
20 volatile fatty acids) and inorganic (heavy metals, S and P) sludge constituents. Paul et al. (2006)  
21 suggested a preferential dissolution of proteins over carbohydrates by measuring COD released  
22 after thermal treatment of digested sludge.

23 Because material composition changed with temperature, it is then quite impossible to define  
24 temperature evolution laws with consistent physical meaning for parameters from Eqns (3) and (4):  
25 it would be fundamentally inaccurate to consider constant activation energies, as the composition  
26 is changing with temperature.

27  
28 Conclusion

29 The impact of temperature on the rheological behaviour of digested sludge has been investigated.  
30 Sludge became progressively more fluid when the temperature was increased, but by normalising

1 the shear stress with the yield stress and the shear rate with the yield stress divided by the  
2 Bingham viscosity, a master curve was obtained. Both yield stress and Bingham viscosity  
3 decreased with increasing temperature, indicating that thermal agitation had a major influence.  
4 However, it is not the only parameter influencing the fluidisation of sludge: thermal history also  
5 played an important role.

6 If sludge was preheated and cooled before experiment, the initial yield stress decreased while  
7 initial Bingham viscosity increased: this may result from a conversion of solid to dissolved  
8 constituents, a process which is partially irreversible. Thus, the usual laws used to model  
9 temperature dependence would be no longer valid because such laws can only be used when the  
10 composition remains constant. This point cannot be asserted in the case of anaerobic digestion  
11 sludge as our results suggest that the sludge composition may be altered by temperature. In order  
12 to confirm this assumption, further work is needed to measure liquor viscosity by filtering sludge at  
13 different temperatures. The apparatus to enable such an experiment is under construction.

14 Such behaviour has to be taken into account in the hydrodynamic modelling of industrial flow  
15 processes in which temperature is modified, such as in recirculation loops through sludge heaters,  
16 where head loss determination can be significantly affected.

17

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19

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6

7 Appendix : numerical values of rheological characteristics allowing us to plot the master curve

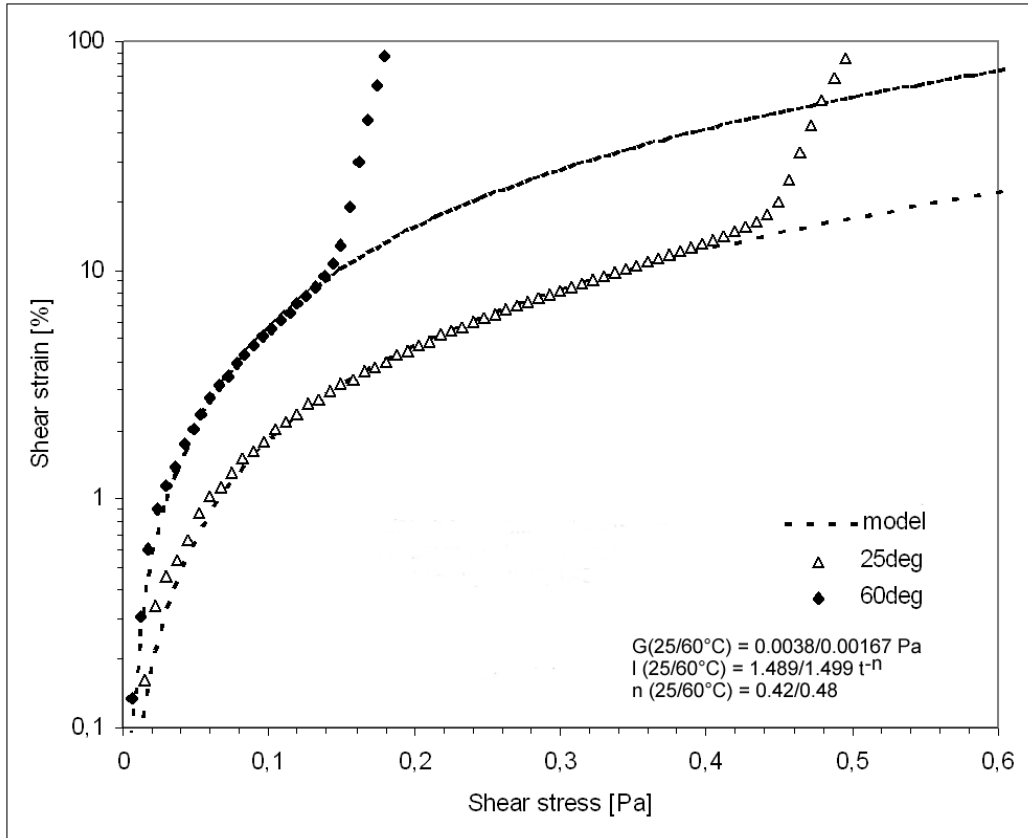
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	10°C	25°C	40°C	60°C
	18.5 g.L <sup>-1</sup>			
$\tau_c$	0.100	0.092	0.088	0.052
K	0.194	0.169	0.148	0.097
n	0.308	0.308	0.308	0.308
$\alpha_0$	0.007	0.005	0.004	0.003
	25.5 g.L <sup>-1</sup>			
$\tau_c$	0.335	0.293	0.201	0.146
K	0.531	0.436	0.331	0.240
n	0.308	0.308	0.308	0.308
$\alpha_0$	0.012	0.009	0.008	0.006
	32.5 g.L <sup>-1</sup>			
$\tau_c$	0.711	0.711	0.606	0.376
K	0.976	0.905	0.770	0.499
n	0.308	0.308	0.308	0.308
$\alpha_0$	0.016	0.013	0.011	0.008
	49.0 g.L <sup>-1</sup>			
$\tau_c$	2.844	2.300	2.175	1.255
K	3.686	2.769	2.327	1.383
n	0.308	0.308	0.308	0.308

$\alpha_0$	0.054	0.035	0.022	0.014
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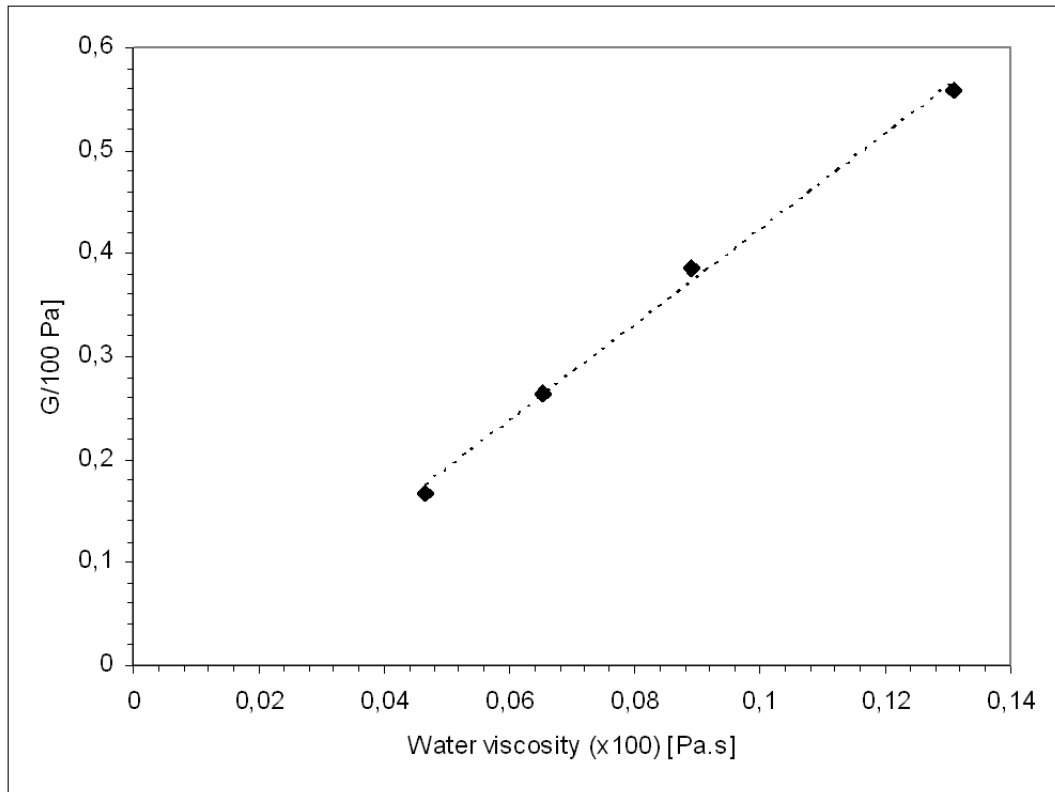
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Captions



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**Figure 1: Viscoelastic behaviour of the 4.8% sludge. at 25 and 60°C. The dotted lines represent the fit of the generalised Kelvin-Voigt model Equation (1)**

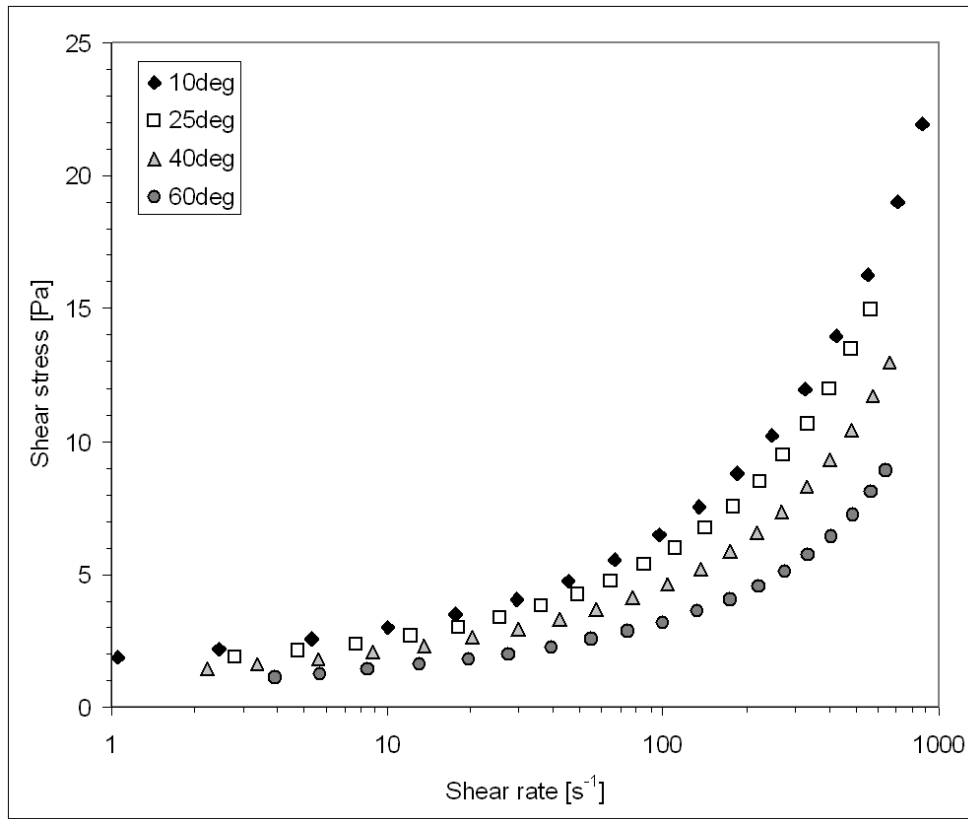


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**Figure 2: Linear relationship between the elastic coefficient and water viscosity**

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2



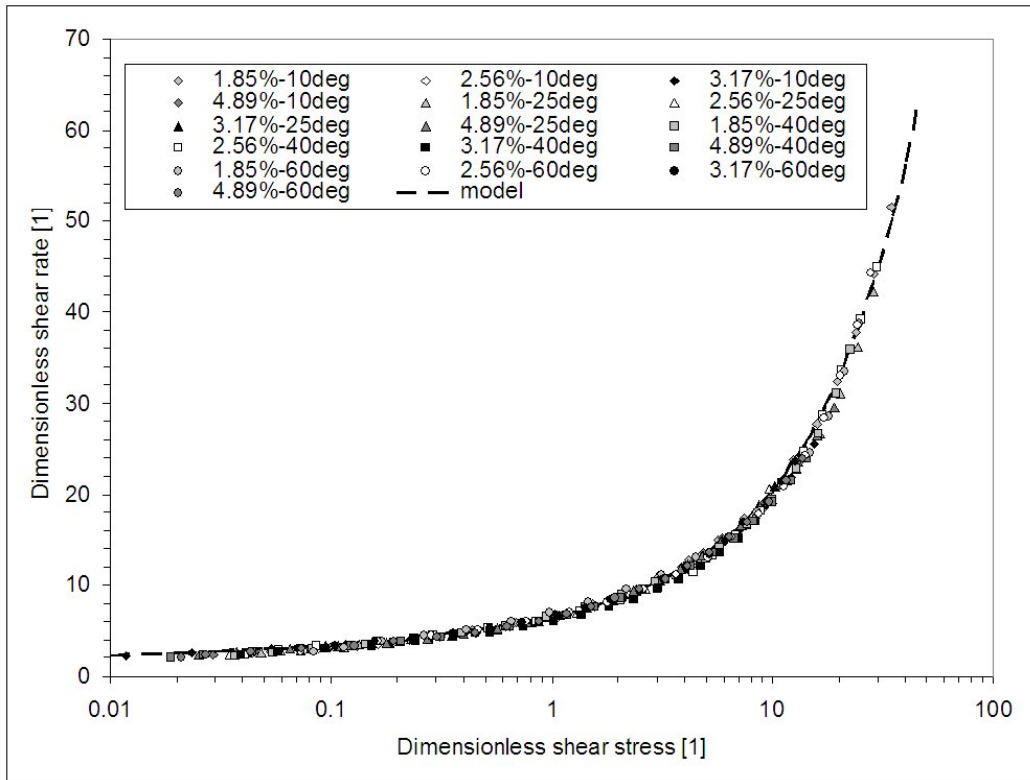
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**Figure 3: Flow curves of the 3.2% digested sludge at different temperatures.**

5





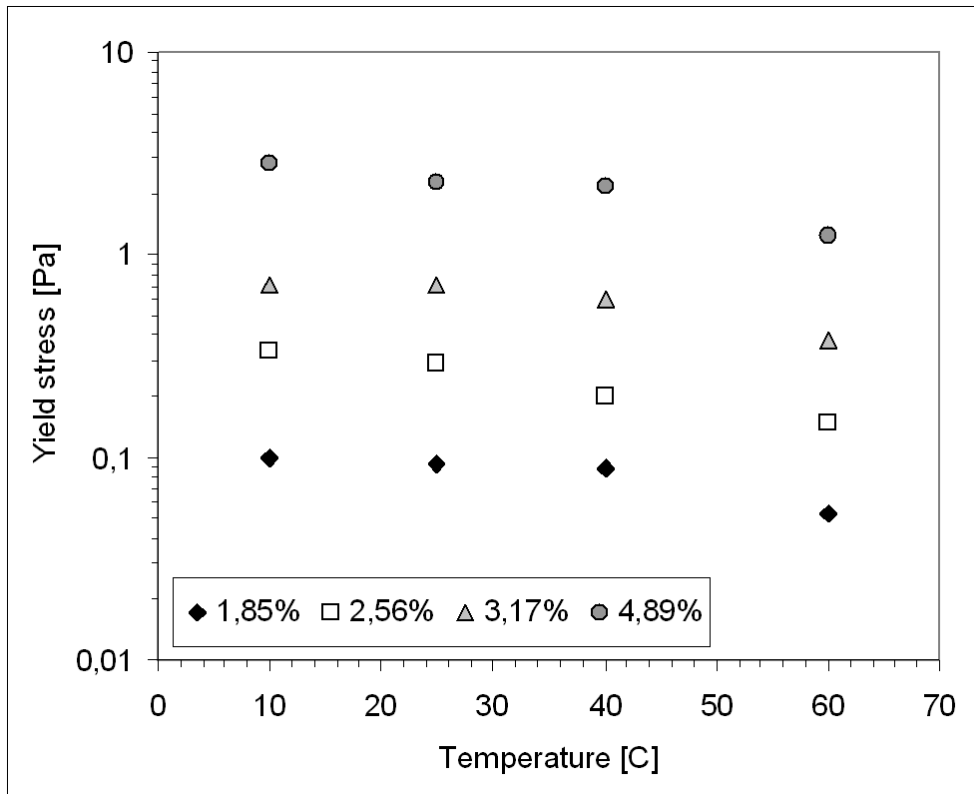
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2 **Figure 4: Dimensionless flow curves of the digested sludge at different solids concentration and**  
 3 **temperature. The dotted line represents the model of Equation (2) with the following parameters:**

4

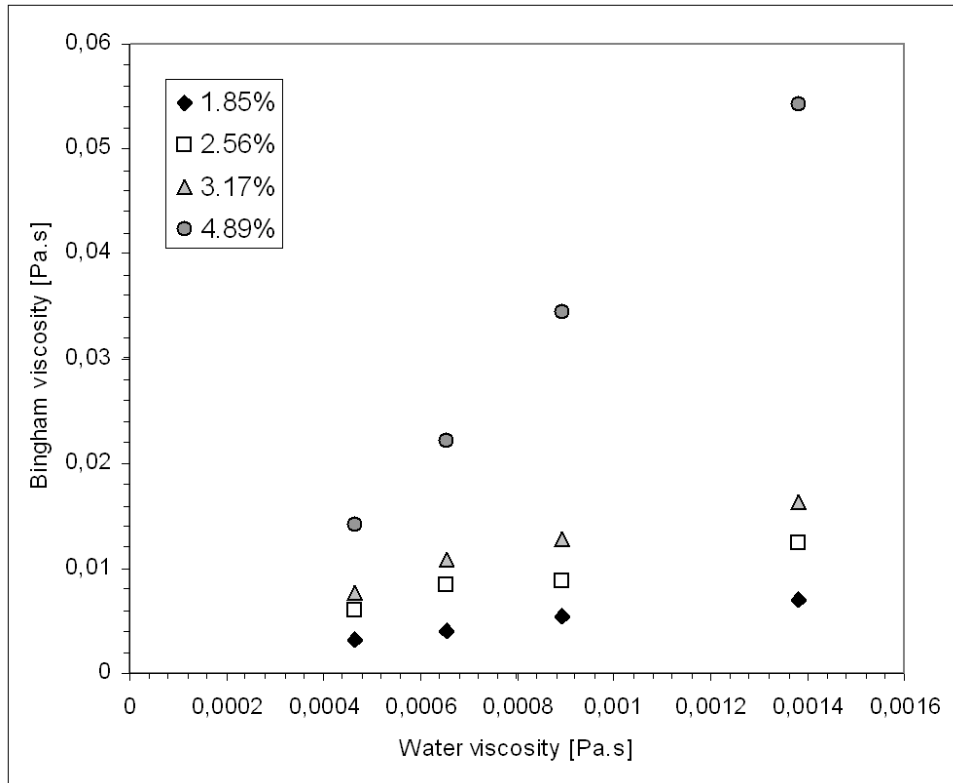
$$\xi = 4.57, n = 0.3$$

5



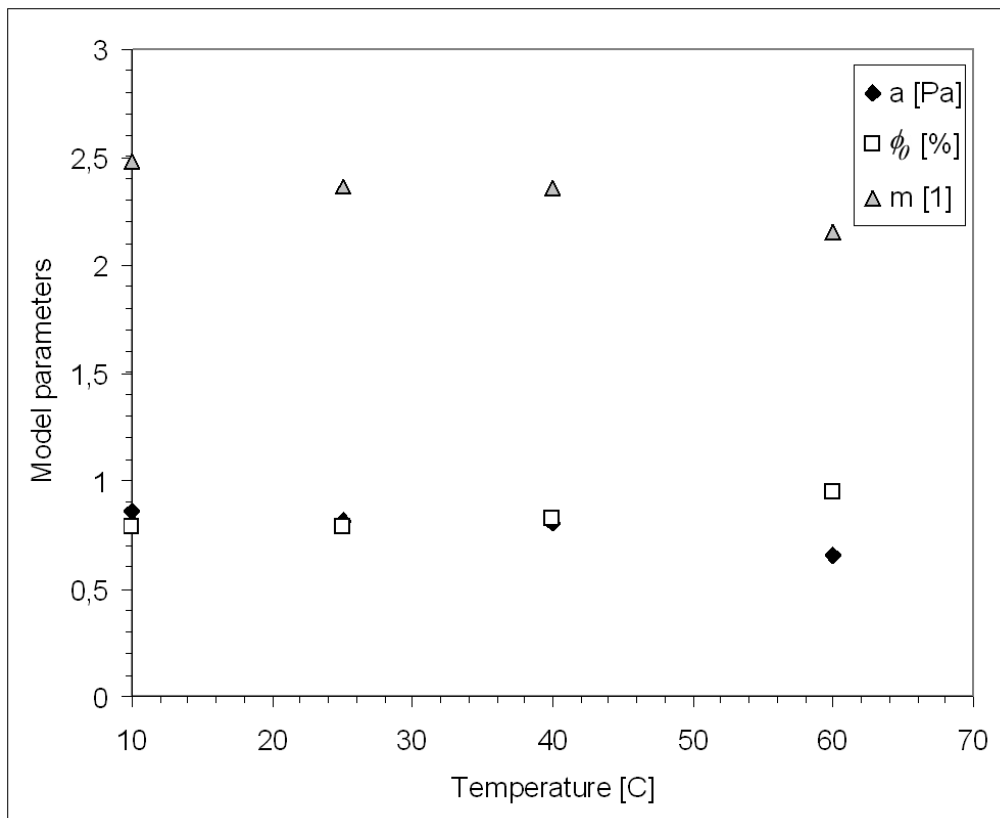
1  
2  
3  
4  
5

**Figure 5: Evolution of the yield stress with temperature for the digested sludge at different concentrations.**



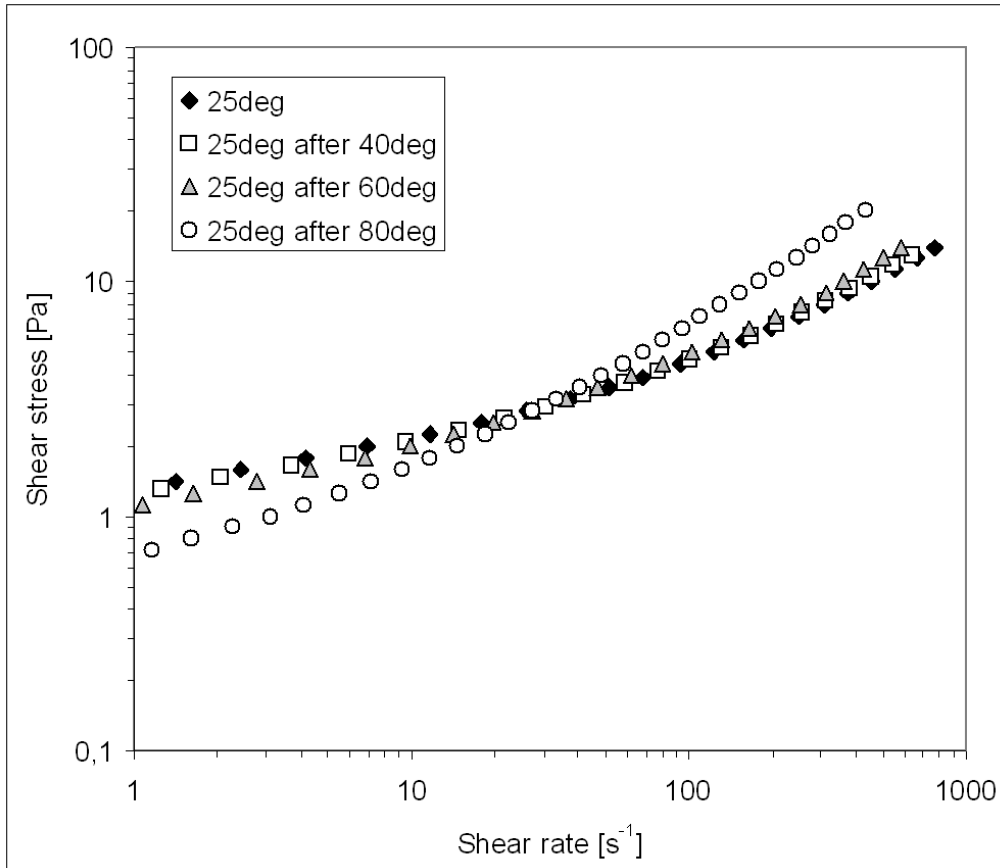
1  
2  
3  
4

**Figure 6: Thermal evolution of the Bingham viscosity of the digested sludge at different concentrations plotted against the viscosity of water at the same temperatures.**



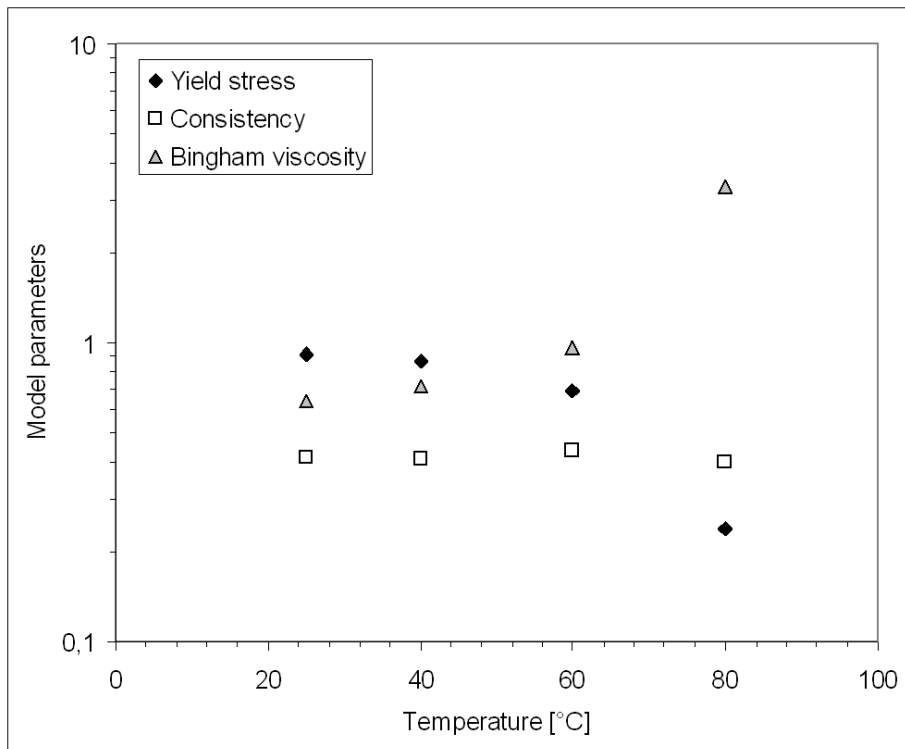
5  
6  
7

**Figure 7: Evolution of the parameters of Equation (3) with temperature.**



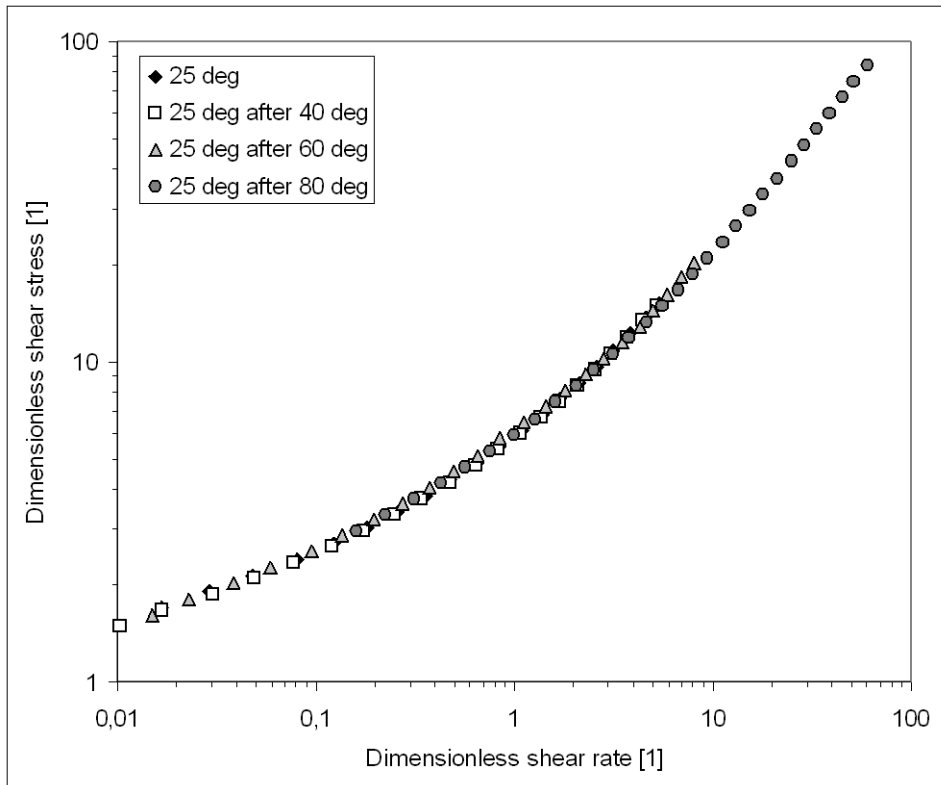
1  
2  
3

**Figure 8: Flow curve of the 3.2% digested sludge at 25°C after being heated to 40, 60 and 80°C.**



4  
5  
6  
7

**Figure 9: Evolution of the parameters of Equation (2) for the 3.2% sludge at 25°C with respect to its thermal history**



1

2 **Figure 10: Dimensionless flow curve of the 3.2% digested sludge at 25°C after being heated to 40. 60**

3

**and 80°C.**

4

5

6

7

8