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Rheological Characterization of Sludge Coming from a Wastewater Treatment Plant

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Abstract: Problem statement: The aim of this study was that of studying the rheology of sewage sludge using two different rheological test protocols taken from literature and comparing them in order to evaluate which useful information are given from every protocol. **Approach:** Two different protocols have been used taking particularly into account the problems connected to sludge heterogeneous composition and to the interaction between solid-solid and solid-water particles in order to completely understand the rheological behavior of this suspension; moreover, the consequences of particular effects connected to test geometry and conditions have been considered. Two fundamental parameters have been modified in the samples: The total solids content and the polyelectrolyte addition. Sludge with 3 and 5% of total solids have been investigated, with or without polyelectrolyte using also microscope analysis to understand the effect of polyelectrolyte on the sludge. **Results:** As expected, it was noticed that sludge viscosity grows up increasing the total solids content and with the presence of polyelectrolyte. The effect of polyelectrolyte is that of separating the liquid-phase from the solid-phase of the sludge giving a more space-heterogeneous suspension with higher viscosity and higher non-Newtonian behavior. **Conclusion:** This study proved that combining two different protocols of analysis can be useful to furnish important and complementary information on sludge rheology especially when some parameters change from sample to sample. Moreover, in order to have good and consistent results, it is necessary to use particular attention on samples pretreatments.

Key words: Conditioned, polyelectrolyte, rheology, sewage sludge, suspended solids

INTRODUCTION

Rheology is a science which has had its main development in the transport field, in particular with the studies on bitumen and conglomerate used to pave roads, motorways and civil areas.

Every fluid can be studied by a rheological point of view especially when it is necessary to understand its behavior in certain mechanical conditions. Rheological studies on cement and food industry are two examples of further applications of rheology to different fluids used in industrial applications. At last, a contribution to rheology is given also by the environmental sector especially concerning sewage sludge. Sewage sludge is a big problem for the environment because a complex treatment is necessary to obtain a final product useful for disposal or in agriculture. Sewage sludge can be defined as a suspension of organic and inorganic particles into a fluid; the presence of the suspended particles involves the fact that some interactions happen among solid-solid and solid-water particles whose characteristics depend on many factors such as particle dimensions, pH, temperature and more. The main rheological parameter that is important to investigate is the viscosity; this parameter provides information about sludge flow characteristics when it is subjected to deformations in flow conditions. An other important parameter is the sludge yield point because it is directly correlated to sludge inner forces between particles and to particles concentration. Many studies have been developed in order to understand how these parameters could affect the rheological measurements (Sanin, 2002; Dentel, 1997; Mori *et al*., 2006; Guibaud *et al*., 2004; Forster, 2002). Sanin (2002) in particular has detected some aspects that influence the sludge rheological behavior: The viscosity of the dispersion medium, the particle concentration, the particle size and shape, the particle-particle and particle-dispersion medium interactions. The solids concentration is the main parameter influencing the sludge viscosity, in particular an increase of solids concentration determine an increase of sludge non-Newtonian behavior instead

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of the Newtonian one that is typical of water and solutions with low solids concentration. The pH growth determine a viscosity increase, this effect is amplified if it's followed by a solids concentration growth; at last, the presence of polyelectrolyte used to improve the sludge dewatering process, modifies the sludge viscosity determining an increase of its values. Dentel (1997) has underlined the fact that in many cases it is quite difficult to correlate the theoretically described physical properties of the sludge (the viscosity that derives from rheological studies is one of these parameters) with the operational physical properties that are important in plant processes such as pumping characteristics and dewatering; despite of this, many studies furnish important correlations between these two aspects. In particular, the dewatering efficiency can be correlated with different squeezing velocities applied to the sludge (Chaari *et al*., 2003); the obtained results show that the dewatering increases with the decreasing of the squeezing velocity. The aim of this study is that of defining a method to perform rheological tests with sewage sludge keeping into account the sludge pre-treatment phase, comparing two different protocols taken from literature in order to achieve the best conditions for rheological tests. Moreover the effects of polyelectrolyte addition to sewage sludge are investigated both by the physical and rheological point of view using also an optical microscope to investigate the physical characteristics of the sludge. The rheological behavior (shear stress-shear rate curves), the viscosity values, the yield stress values and the maximum shear stress are the observed parameters that furnish information on sewage sludge rheological characteristics.

MATERIALS AND METHODS

Sludge samples: The sludge samples employed for the experiments come from a wastewater treatment plant located near the city of Turin in the North-west of Italy and run by the Smat S.p.a Society. The considered sludge come from two different sections of the plant at two different steps of treatment; the first one is a pre-thickening sludge, a mixture of sludge coming from the primary and the secondary settling phase before the thickening necessary to obtain the optimal solids concentration in the digesters. The second one is a sludge coming out from the primary settling phase and conditioned with polyelectrolyte, a reactant normally used to improve sludge dewatering. These sludge samples were treated in the Smat laboratories in order to modify their solids content at two different Concentrations: 3 and 5% w/w. The obtained samples were stocked in a fridge at the temperature of +4°C before being submitted to the laboratory and tested at the Politecnico di Torino.

Microscope analysis: In order to understand sludge physical characteristics, before starting the rheological tests, a series of photos using an optical microscope was performed (Tixier *et al*., 2003). The employed microscope is a LEICA DMLP; the adopted enlargements were 2.5 and 5x the photos have been done with a digital camera JVC TK-C1380. Figure 1a shows a microscope photo (2.5x) representing a sludge sample (3% w/w total solids TS) coming from the exit of the primary settling phase and conditioned with polyelectrolyte. Figure 1b shows a sample of sludge (2.5x and 3% w/w total solids TS) without polyelectrolyte; it's clear how the polyelectrolyte carries out the function of separating solids particles of sludge from water creating a non-homogeneous solution with a different structure in comparison with a normal sludge. Water is physically separated from solids as it's shown in Fig 1a.

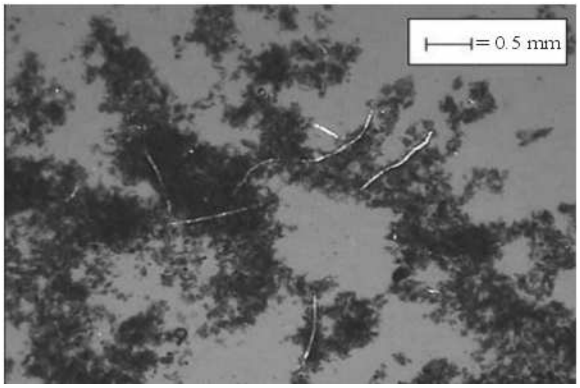

Fig. 1a: Photo of a sludge (2,5x; 3% w/w TS) conditioned with polyelectrolyte

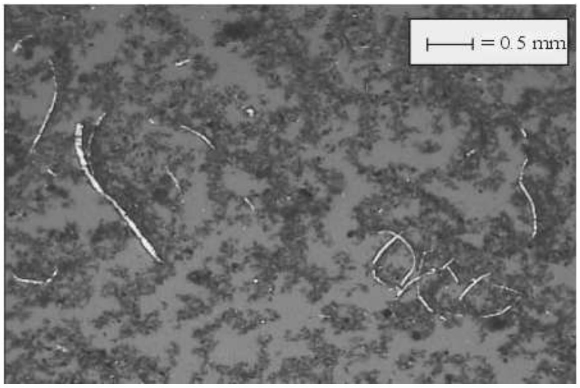

Fig. 1b: Photo of a sludge (2,5x; 3% w/w TS) without polyelectrolyte

Moreover, these photos show that sludge complexity is not only due to the fact that it is a dispersion made of (minimum) two phases (a liquid phase and a solid phase), but also to the fact that particles (solid phase) are heterogeneous both by the physical and by the dimensional point of view. In the sludge there are organic and mineral particles with a very different mechanical behavior; in addition there are particles with a much stretched shape made of organic materials that, during the tests, tend to twist themselves creating heterogeneity into the sample that could affect the measures of the rheological test.

Rheological behavior: The rheometer employed to perform laboratory tests is an Anton Paar Physica MCR 301 that works controlling torque with a sensibility of the bending moment and torsion angle respectively of 0.1 µN·m e 1 µrad. The used geometry is that of the coaxial cylinders with a fixed gap of 1.13 mm; this kind of test called rotational test is particularly suitable in case of low-viscosity liquids, polymer solutions emulsions and solid-in-liquid suspensions (Gupta, 2000) because the water remains in the sample without the possibility of being ejected as it happens in dynamic tests (cone-and-plate or plate-and-plate tests) especially at high shear rates. On the other hand, the tests must be performed immediately after having put the sample in the cylinder because the particles in the sludge tend to precipitate causing a particle size distribution and concentration between the cylinders (Seyssiecq *et al*., 2003); for this reason, sludge preconditioning with the thermostatic bath at the same temperature of the rheometer has been fundamental otherwise the sample would have been conditioned into the rheometer with some precipitation problems.

The adopted test methods were two and are subsequently described.

Protocol 1: Double repeated ramp tests: Increase of the shear rate from 0-100 $\sec^{-1}$ in 90 sec and return to 0 $\sec^{-1}$ measuring at the same time the shear stress (PA) applied to the sludge. Immediately after the first test, a second one was performed with the same sludge sample in order to investigate its time-dependant properties and the effect of inner and outer factors as it will be illustrated later on.

Protocol 2: step-tests: Use of 8 fixed shear rates (0.9, 4.5, 15, 40.5, 67.5, 81, 135, 243 $\sec^{-1}$) (Lotito *et al*., 1997) for a time included between 300 and 100 sec in order to understand the time required by the sludge to reach a constant viscosity value at different shear rates.

The number of samples to analyze was not decided before the execution of the tests, so, the reported results in this study have to be considered as typical, even though modest data dispersion has been noticed.

Pre-treatments: a thermostatic bath was employed to obtain a sludge constant temperature equal to +35°C and the rheometer cell too was conditioned to that temperature. This procedure has, as a consequence, the possibility of starting the rheological tests immediately after having put the sludge in the rheometer in order to avoid the sludge particles sedimentation that could affect the rheological test results. This temperature has been chosen because it is proper of an anaerobic digester operating in mesophyle conditions. A 500 µm screen (32 mesh) was employed to sieve the sewage in order to eliminate a small part of the solid fraction before putting the sample into the rheometer. This solution (Lotito *et al*., 1997) was necessary because of the small distance between the coaxial cylinders of the rheometer (1.13 mm) and the presence in the sludge of few over-size particles that could damage the rheometer.

RESULTS

In order to analyze the sludge rheological behavior, it's fundamental to consider a series of inner and outer factors that could play an important role during the rheological tests, this is particularly important when the investigated fluid is extremely heterogeneous. These factors have to be kept into account in order to furnish a correct data interpretation.

The inner factors are due to the nature of the fluid, the main inner factors are TS percentage, dispersant phase viscosity, interactions among particles and among particles and fluid phase, particles shape and dimension. The outer factors are due to the test geometry and conditions. As it will be illustrated, both inner and outer factors are responsible of the rheological curves trend.

Protocol 1, double repeated ramp test: Figure 2a, b, 3a and b show the typical results of these tests; every graph has on the x-axis the values of shear rate ($\sec^{-1}$) and on the y-axis the values of shear stress (Pa); so the viscosity (Pa·s) can be simply obtained by the ratio between the shear stress and the shear rate. The non-Newtonian behavior of these fluids due to the non-linearity of the curves is evident; the consequence is that the viscosity varies when the shear rate values change.

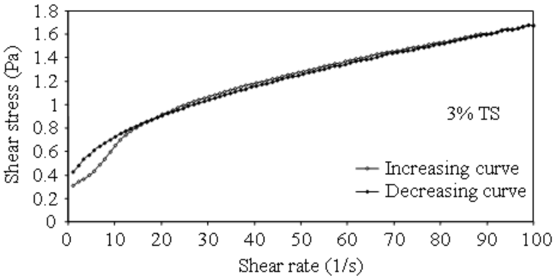

Fig. 2a: Double repeated ramp test results (3% TS sludge without polyelectrolyte)

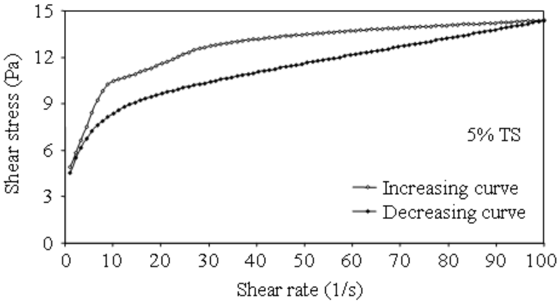

Fig. 2b: Double repeated ramp test results (5% TS sludge without polyelectrolyte)

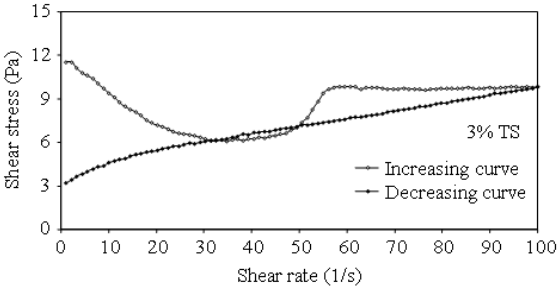

Fig. 3a: Double repeated ramp test results (3% TS sludge with polyelectrolyte)

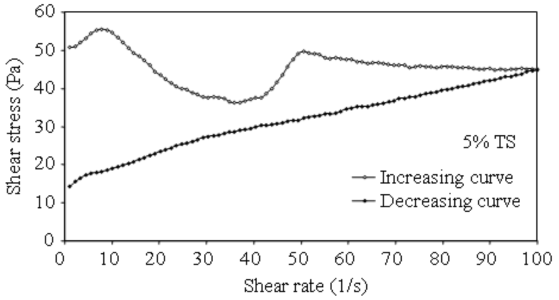

Fig. 3b: Double repeated ramp test results (5% TS sludge with polyelectrolyte)

In all the curves the presence of a yield stress is evident; this can be defined as the minimum stress that is necessary to apply to the sludge in order to reach flow conditions; moreover, this value grows up both with the increase of TS content and with the presence of polyelectrolyte. Water-particles and particle-particle connections are the reasons of this yield stress, in fact more particles means more connections and links that have to be disrupted. The sludge with polyelectrolyte shows a yield stress that is higher in comparison with that one of the sludge without polyelectrolyte; this is due to the fact that, besides of the links between water and particles, the polyelectrolyte has got a kind of binder effect towards the organic solid particles of sludge. Aggregates of organic particles are formed and have to be disrupted in order to move the sludge. The presence of these aggregates is also important in order to explain the reasons of the shear stress peak that occurs after the yield stress at low shear rate values in case of sludge with polyelectrolyte. After the disruption, the polyelectrolyte effect vanishes and the viscosity rapidly decreases.

All the phenomena that have been explained can be associated to inner factors, therefore, to factors that are directly connected to sludge characteristics.

The influence of outer factors is also evident in some graphs, especially in that one concerning the sludge with polyelectrolyte. After the quick reduction of viscosity that occurs after the maximum shear stress, a new increase of the shear stress and than a phase of quite constant values can be noticed. This is not a normal behavior of a fluid, the particular flow conditions into the cylinder may be considered responsible of this trend.

Finally, it’s important to notice that increasing the TS content and introducing polyelectrolyte, the gap between the curves of ramp-up and ramp-down becomes higher and higher. The 3% sludge without polyelectrolyte presents an almost perfect overlap between the two curves, in all the other cases the increasing curve is above the decreasing one. This fact can only be due to particles reorganization and orientation that occur during the test.

After every test made on a sample of sludge, a new test was performed on the same sample immediately after the first one. So, every sample was tested two times; this procedure has permitted to analyze in a better way the effects of inner and outer factors, as mentioned before. Figure 4a, b, 5a and b show these results; in the same graph are represented both the curves of the main test (primary) and those of the repeated one (secondary).

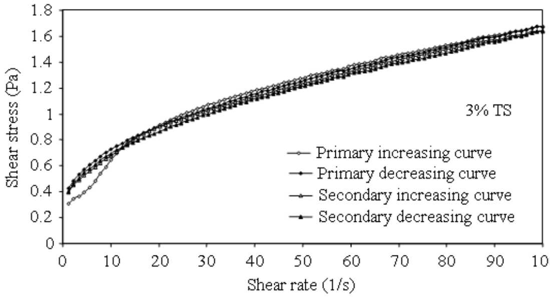

Fig. 4a: Double repeated ramp test results (3% TS sludge without polyelectrolyte; primary and secondary test)

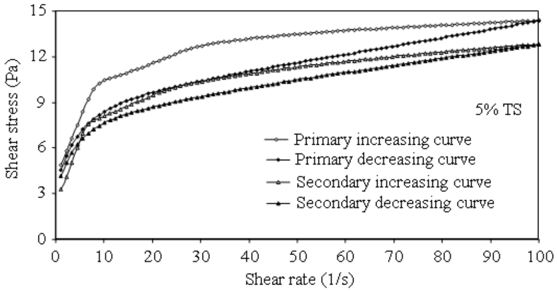

Fig. 4b: Double repeated ramp test results (5% TS sludge without polyelectrolyte; primary and secondary test)

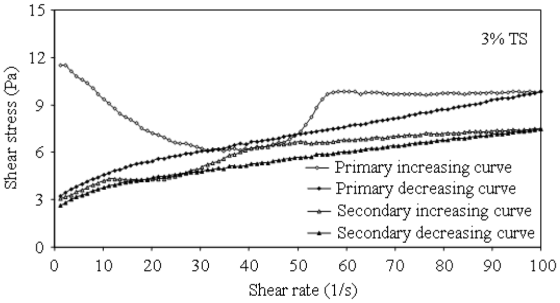

Fig. 5a: Double repeated ramp test results (3% TS sludge with polyelectrolyte; primary and secondary test)

It's evident that, in the case of 3% sludge without polyelectrolyte, all the curves are very close one to the other, without gap. This means that time-dependant factors, not specifically investigated in this study, may be excluded. In all the other cases, the curves of the already employed samples (secondary test) are translated toward the bottom: the measured values of the shear stress, at the same conditions of shear rate, are lower in the second test than in the first one. This fact is the consequence of the agglomerated particles disruption that occurs during the first test.

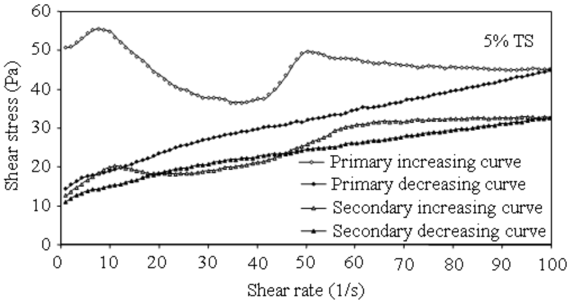

Fig. 5b: Double repeated ramp test results (5% TS sludge with polyelectrolyte; primary and secondary test)

Time-dependant factors and particles reorganization are responsible of the difference between the increasing and the decreasing shear rate curves, as it may be noticed in the graphs. This difference is less evident during the second test. In addition, yield stress values are lower during the second test, especially for the sludge with polyelectrolyte; this is a further consideration concerning the disruption of the link among particles and polyelectrolyte (when it's present).

In all the rheograms, but in particular in that one of the sludge with polyelectrolyte, the increasing shear rates curves maintain their particular trend with a double flexure, the outer factors due to the geometry and conditions are still effective, while it can be noticed a pronounced decrease of the initial maximum value of shear stress. This is an other consequence of the particles disruption; in fact the polyelectrolyte hasn't the time to re-agglomerate sludge particles between the first and the second test. Inner factors and outer factors, time-dependant behavior and tixotropy are all factors that occur during a rheological test, they play an important role and in many cases their actions and interactions are very difficult to demonstrate, separate and recognize.

Protocol, 2 step-tests: Typical results of this kind of tests are shown in Fig. 7a, b, 8a and b. As said before, 8 values of shear rates have been used and kept constant for a certain time between 100 and 300 sec. Figure 6 illustrates a typical obtained rheogram with the employed shear rates values. Each single part of the curves in Fig. 7a, b, 8a and b, represents the viscosity values at a certain value of shear rate (from 0.9-243 sec^{-1}) while the discontinuities between every part of the curves are due to the change of shear rate.

This protocol gives directly the viscosity values calculated as the ratio between the measured shear stress and the adopted shear rate; even though it's not possible to investigate the time-dependant factors that have been shown in the previous protocol, in this case the viscosity values are measurable at each step of shear rate. These values correspond to the steady state conditions reached by the sludge at the end of every step.

Outer factors are responsible of viscosity variations that occurs especially at low shear rates when the flow is not constant; the necessary time used to reach steady state conditions is less and less high increasing the shear rates.

As in the previous protocol, the viscosity values decrease increasing the shear rates, but the comparison between the observed viscosity values, measured with the two different protocols, is interesting. For protocol 1, it is necessary to distinguish between viscosity values measured increasing the shear rates (protocol 1a) and those ones measured decreasing the shear rates (protocol 1b). Table 1 and 2 report all the measured viscosity values, in particular, Table 1 shows the values obtained for increasing shear rates, while Table 2 shows the values obtained for decreasing shear rates.

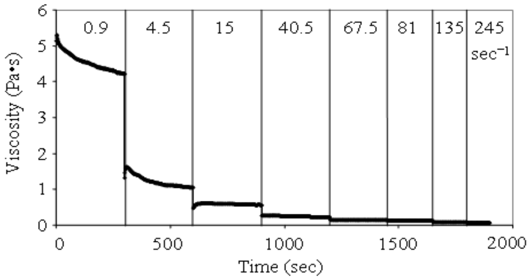

Fig. 6: Example of step-test results

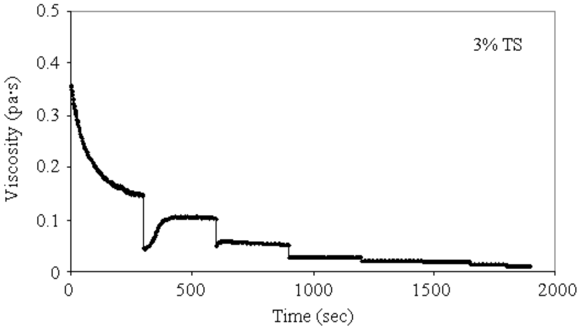

Fig. 7a: Step-test results (3% TS sludge without polyelectrolyte)

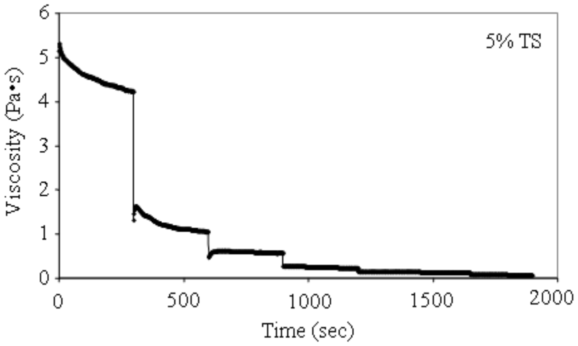

Fig. 7b: Step-test results (5% TS sludge without polyelectrolyte)

Table 1: Comparison between viscosity values measured in protocol 1a and protocol 2

	3% TS			5% TS		
Shear rate (sec^{-1})	Viscosity (Pa·s) Protocol 1a	Viscosity (Pa·s) Protocol 2	Δ	Viscosity (Pa·s) Protocol 1a	Viscosity (Pa·s) Protocol 2	Δ
Without polyelectrolyte						
0.9	-	0.145	-	-	4.218	-
4.5	0.088	0.102	-0.014	1.602	1.040	0.562
15	0.054	0.052	0.002	0.705	0.563	0.142
40.5	0.029	0.027	0.002	0.322	0.226	0.096
67.5	0.021	0.020	0.001	0.204	0.137	0.067
81	0.019	0.018	0.001	0.172	0.113	0.059
135	-	0.013	-	-	0.077	-
243	-	0.010	-	-	0.055	-
With polyelectrolyte						
0.9	-	4.472	-	-	27.080	-
4.5	2.404	0.985	1.419	10.360	6.404	3.956
15	0.539	0.309	0.230	2.916	1.133	1.783
40.5	0.154	0.138	0.016	1.002	0.525	0.477
67.5	0.144	0.086	0.058	0.573	0.285	0.288
81	0.120	0.072	0.048	0.472	0.221	0.251
135	-	0.048	-	-	0.138	-
243	-	0.030	-	-	0.087	-

Table 2: Comparison between viscosity values measured in protocol 1b and protocol 2

	3% TS			5% TS		
Shear rate (sec^{-1})	Viscosity (Pa·s) Protocol 1b	Viscosity (Pa·s) Protocol 2	Δ	Viscosity (Pa·s) Protocol 1b	Viscosity (Pa·s) Protocol 2	Δ
Without polyelectrolyte						
0.9	-	0.145	-	-	4.218	-
4.5	0.127	0.102	0.025	1.498	1.040	0.458
15	0.054	0.052	0.002	0.603	0.563	0.040
40.5	0.029	0.027	0.002	0.273	0.226	0.047
67.5	0.021	0.020	0.001	0.186	0.137	0.049
81	0.019	0.018	0.001	0.164	0.113	0.051
135	-	0.013	-	-	0.077	-
243	-	0.010	-	-	0.055	-
With polyelectrolyte						
0.9	-	4.472	-	-	27.080	-
4.5	0.855	0.985	-0.130	3.840	6.404	-2.564
15	0.336	0.309	0.027	1.387	1.133	0.254
40.5	0.165	0.138	0.027	0.737	0.525	0.212
67.5	0.118	0.086	0.032	0.537	0.285	0.252
81	0.108	0.072	0.036	0.492	0.221	0.271
135	-	0.048	-	-	0.138	-
243	-	0.030	-	-	0.087	-

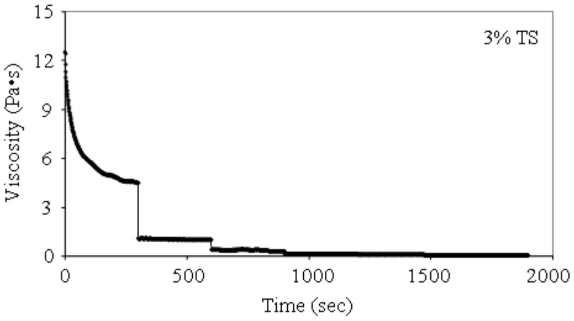

Fig. 8a: Step-test results (3% TS sludge with polyelectrolyte)

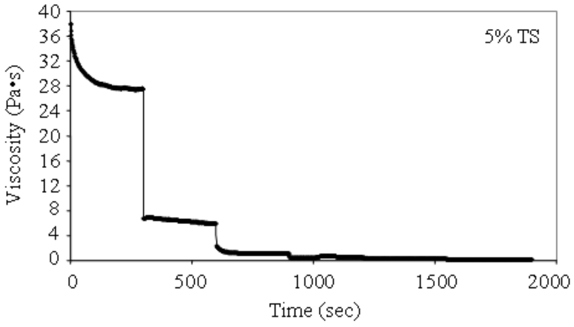

Fig. 8b: Step-test results (5% TS sludge with polyelectrolyte)

DISCUSSION

Table 1 and 2 report all the data obtained with the two different test protocols; in particular these tables are necessary to understand what the differences between the two protocols are in terms of measured viscosity.

Concerning Table 1, the measured viscosity values are of the same order of magnitude, but the differences (Δ = protocol 1-protocol 2) between the two protocols change either with the sludge or with the shear rate. In general, protocol 1a gives viscosity values that are higher than protocol 2. This is due to the fact that viscosity values measured with protocol 1 are not that of the sludge in steady state conditions as the values gathered in protocol 2 at the end of every shear rate step. In addition, this difference is less important when the shear rate is higher.

Considering the viscosity values gathered with protocol 1b in comparison with those ones of protocol 2, a further reduction of the differences between viscosity values is evident especially for the sludge with polyelectrolyte; at low shear rates an inversion of this tendency can be noticed, with higher measured viscosities for protocol 1b than for protocol 2. In all the examined cases, over 15 sec^{-1} the differences between viscosities are lower than 0.5 Pa·s, only for shear rates lower than 15 sec^{-1} these differences are higher than 1 Pa·s. At last, the measured viscosities in protocol 1 decrease passing from increasing shear rates to decreasing shear rates (comparison between Table 1 and 2). As said before, this fact is directly correlated to time-dependant factors and inner factors. In order to give further information about viscosity values measured with protocol 2 at the end of every step at constant shear rate, an interpolation of these data by means of a power law model taken from literature (Coussot, 2005; Gupta, 2000; Larson, 1999; Barnes *et al*., 1989; Macosko, 1994) has been performed.

Table 3: Adopted model results

	Without polyelectrolyte		With polyelectrolyte	
	3% TS	5% TS	3% TS	5% TS
k	0.148	3.535	4.051	24.824
n	0.579	0.233	0.062	0.002
R^2	0.977	0.999	0.999	0.999

The adopted model is:

$$\eta = k \cdot \dot{\gamma}^{(n-1)}$$

where, k and n are two parameters representing respectively the sludge thickness rate and the sludge dependence rate by shear rate variations.

The Table 3 shows the obtained results.

In all the analyzed samples the R^2 values are very close to 1, this means that the model have a good consistency and that the relationship between the viscosity and the shear rate is well represented by the applied power law model.

Considering k and n parameters, k values increase with increasing the TS content and with the presence of polyelectrolyte, while n values decrease. This means that, increasing the TS content and using polyelectrolyte, the sludge thickness grows up and its behavior in flow conditions becomes more sensitive to shear rate variations.

CONCLUSION

In this study the rheological behavior of four different kind of sludge was analyzed using two test protocols. First of all, important and complementary information about sewage sludge behavior in flow conditions have been gained and the adopted protocols have furnished similar results. This fact is also the consequence of a correct preconditioning procedure which has guaranteed a constant feed to the rheometer, controlling the main parameters that are responsible of viscosity changes in fluids. Sewage sludge are a very complex suspension, so it’s necessary to take in account many different factors that could be responsible of the curve trends reported in the rheograms; in particular both inner and outer factors typical of the sludge characteristics and of the test geometry and conditions were considered. Moreover, the preliminary microscope analysis has furnished a fundamental basis in order to understand the sludge rheological behavior and to take in account the phenomena of particles aggregation, aggregates disruption and particles orientation that occur when a sludge conditioned with polyelectrolyte is tested. The viscosity values gathered by means of the two protocols are of the same order of magnitude; only at low shear rates a certain difference may be appreciated but this one is reduced considering only the viscosity values from the decreasing shear rate curves in protocol 1. In general, the viscosity grows up increasing the sludge TS content and this effect is more evident when polyelectrolyte is added. In order to complete the work, a power law model has been used to investigate the relationship between viscosities and shear rate values obtained by the tests. At last, rheology may be considered as an innovative and useful way to study sewage sludge characteristics, giving important information about the sludge behavior in certain flow conditions, especially when some parameters are changed. The TS content and the presence of polyelectrolyte are two of these parameters; in particular the TS content has a great importance both in designing and in managing a sewage sludge plant. In every part of the plant where the sludge is moved, the viscosity is fundamental and it may be considered as a parameter useful to understand the sludge resistance to the movement: rheology could be an interesting way to improve plant performances.

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