## SHOULD RHEOLOGICAL PROPERTIES OF ACTIVATED SLUDGE BE MEASURED?

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#### **ABSTRACT:**

The core of activated sludge monitoring lies in the biological analyses. Anyway, the knowledge of sludge physical characteristics is crucial for a proper management of WWTPs (Waste Water Treatment Plants). One of these physical features is viscosity that, notwithstanding its valuable role has not yet become a routine analysis. This study examined the evolution of rheological properties of two sludges alongside the "purification route" (from the biological reactor up to the sludge treatments). It could been shown that sludges behaved like non-Newtonian fluids and dry solids content strongly affected viscosity values, which reached relatively high values. Microscopic observation of flocs was carried out. Both the sludges revealed similar features, in particular an over-proliferation of filamentous bacteria. This work showed how rheological measurements can be a tool to obtain information on microbiological composition of activated sludge and how it could be related to settleability properties.

### **KEY WORDS:**

activated sludge, rheology, filamentous bacteria, viscosity, waste water

## 1 INTRODUCTION

The activated sludge (AS) process is so far the most common biological method used worldwide for wastewater treatment. Water contamination is removed through biomass activity leading to the unavoidable generation of a large amount of biosolids (waste sludge), which has to be managed. Despite its small volume more than 40% of the wastewater treatment cost is spent on sludge treatment and disposal processes [1]. In order to improve sludge handling (e.g. pumping, transporting, mixing, and dewatering operations) and to reduce costs consequently a proper technical design and management of biosolids treatment and reuse/disposal facilities is needed. This requirement can be fulfilled mainly through the knowledge of physical characteristics of sludge [2]. In addition, sludge physical together with biological properties heavily affect also the solid-liquid (i.e. sludgetreated water) separation process [3], which occurs in final clarifiers and whose efficiency influences the overall plant performance.

One of the most important sludge physical characteristics is the rheological behavior, which is capable to interfere with reactors hydrodynamics, oxygen transfer (i.e. design of aeration systems), and sludge pumping (i.e. recycle flows), transportation (i.e. pressure losses in

pipes), and conditioning/dewatering [4-6] with evident consequences on the operating costs [7]. As well known, rheology describes the deformation of a body under the influence of stress and the determination of shear stress  $\tau$  as a function of shear rate  $\dot{\gamma}$  enables to characterize the flow behavior of a fluid. Sewage sludge rheology has been extensively investigated in recent years [8] and, thus, AS (as well as granular, anaerobic, and membrane bioreactor sludge) is classified as a non-Newtonian fluid, which means that shear stress is not linearly related to shear rate. From a structural point-ofview at high shear rate the motion of the particles prevents the particle-particle bonding and leads to a more dispersed suspension behaving more like a fluid. On the contrary at lower shear rates, individual particles can aggregate in clusters being able to form a rigid network and the slurry can be considered as a solid [9]. Viscosity (shear stress/shear rate ratio  $\mu$ ) is the main characteristic summarizing flow behavior: The greater the viscosity, the more viscous and less flowable is the fluid, which means that the molecules in higher viscous liquids are more strongly bound to each other, and thus less freely moveable [8].

Different factors can affect the evolution of AS viscosity: dry matter content, a "non-specific" parameter described by Total Suspended Solids (TSS) concentra-

tion, and also several characteristics related to sludge composition such as the surface charge, the bulk solution ionic strength, the extracellular polymeric substances (EPS) content, ... [8, 10, 11]. As a consequence, sludge rheological properties being dependent on its nature are able to provide clues to its physical (e.g. solid content) and biological (e.g. floc structure and abundance of filamentous bacteria [11, 12]) state. The biological state is directly linked to the surrounding environment, e.g. waste water composition, operational conditions, ... [13]. Specifically, filamentous micro-organisms form a part of the bacterial population that plays a predominant role in AS community serving as "backbones" for flocs and then favor its growth. Nevertheless, their over-proliferation is recognized as being the main factor inducing poor ability of sludge to settle and negatively affecting the purification process [12].

However, despite their recognized potentiality, rheological data have not yet become usual parameters for WWTPs (Waste Water Treatment Plants), since their significance (and comparability) strongly depends beyond sludge nature on rheometer type, measurement device, and experimental procedures (i.e. the measurement protocol). For these reasons, the present study proposed the evaluation of AS rheological properties by means of a well-established measurement protocol according to Ratkovich et al. [8]. Moreover, some of the major influential variables on rheological characteristics were analysed with the twofold aim to link the viscosity evolution to different (both physical and biological) "routine" parameters and allow its forecast also without a direct measure. The factors taken into account were: i) dry matter concentration (starting from 1 g/L corresponding to the concentration in biological reactors up to 60 g/L after a post-thickening stage) to provide a "data-base" of flow characteristics during the whole sludge route from its generation to the outgoing from WWTP and ii) several specific characteristics related to sludge microbiological composition, i.e. the floc structure and the presence of filamentous bacteria, which were used as key parameters to compare sludges coming from different WWTPs.

### 2 MATERIALS AND METHODS

## 2.1 SLUDGE SAMPLING AND PREPARATION

Analysed sludges were taken from the biological reactor of 2 WWTPs located in Northern Italy. The first plant (#1 WWTP, design size 400,000 p.e.) treats domestic and industrial wastewater and the process scheme includes equalization/homogenization, pre-denitrification, oxidation-nitrification, and secondary settling.

Dry solids		Viscosity					
concentration	Rheometer adapter/spindle	@ 100 s <sup>-1</sup>	K	m	$RMSD_p$	H	RH
(%)		[mPa·s]	[mPa·s <sup>m</sup> ]	[-]	[%]	Pa/s	%
#1 WWTP							
0.1 %	ULA	1.4	0.04	1.71	9.84	1.0	1.8%
0.4 %		2.5	0.40	1.33	6.51	4.9	4.7%
1.6 %	SSA / SC4-18	18.2	360	0.33	9.43	41.8	5.7%
1.8 %		26.7	465	0.35	4.13	48.1	7.0%
2.0 %		47.3	649	0.29	4.31	38.6	7.8%
2.1 %	SSA / SC4-34	76.9	993	0.44	18.0	5.4	2.0%
2.3 %		77.0	822	0.51	18.4	13.4	4.7%
3.1 %		153.5	1,308	0.57	14.3	66.6	11.1%
3.3 %		181.3	129	n.a.	7.21	70.9	20.7%
3.6 %		179.3	900	0.68	13.8	64.4	10.2%
3.7 %	SSA / SC4-25	141.7	1,702	0.52	6.21	64.6	9.7%
4.3 %		264.2	4,962	0.44	3.03	280.7	21.2%
4.5 %		272.7	7,151	0.33	6.87	114.7	7.7%
5.3 %		469.5	7,005	0.44	2.52	250.5	12.1%
5.8 %		1,090	12,359	0.56	2.06	445.8	18.1%
#2 WWTP							
0.3 %	ULA	2.1	0.21	1.43	7.64	2.8	4.3%
1.3 %	SSA / SC4-18	7.2	7.46	0.81	7.57	13.7	6.9%
2.6 %	SSA / SC4-34	76.3	955	0.47	16.6	38.3	12.9%
3.8 %	SSA / SC4-16	192.6	1,390	0.49	9.89	16.1	2.8%
4.2 %		280.4	1,770	0.53	7.41	19.5	2.2%
4.6 %	SSA / SC4-25	549.5	2,706	0.63	6.89	51.9	3.7%
4.9 %		552.1	2,093	0.65	8.83	139.7	13.8%

Table 1: List of tested TSS concentration, together with employed adapters/spindles. Main rheological findings are summarized: viscosity measurement at 100 s<sup>-1</sup>, Herschel-Bulkley parameters K and m together with goodness of fit as RMSD<sub>p</sub>, and hysteresis loop values (ULA = Ultra Low Adapter, SSA = Small Sample Adapter, n.a. = not available).

The sludge treatment line consists of: dynamic thickening, anaerobic digestion and mechanical dewatering. The second plant (#2 WWTP, design size 200,000 p.e.) handles mainly domestic discharges by means of a CAS (Conventional Activated Sludge) system with biological nitrogen removal. The sludge treatment line consists of: dynamic thickening and mechanical dewatering. Sludge samples were thickened by means of a laboratory centrifugation system (ALC International) in order to explore the rheological behavior at different TSS content: Tested concentrations are listed in Table 1 clearly displaying that the range varied from 0.1% (i.e. AS in biological reactor) to 5.8% (e.g. a digested post-thickened sludge).

### 2.2 RHEOLOGICAL MEASUREMENTS

A rotational rheometer (Ultra Programmable Rheometer LV-DV III+, Brookfield, Middleboro, USA) was employed for the measurements; its main advantages/ drawbacks are summarized in [8]. The principle of operation of LV-DV III+ rheometer is to drive a spindle (immersed in the test fluid) through a calibrated spring. The viscous drag of fluid against the spindle is measured by the spring deflection, evaluated by a rotary transducer and depending on the rotational speed of spindle, the size and shape of the spindle, the adapter, and the full scale torque of the calibrated spring (equal to 0.0673 mNm for LV-DV III+ model). Different spindles/adapters were used depending on dry solids concentration in sludge samples (see Table 1). Viscosity data acquisition and analyses were carried out using Rheocalc V3.1 software (comprising Brookfield Engineering Advanced Viscometer Instruction Set (BEAVIS), which allows the creation of programs for the control

of connected instrumentation and data elaboration). As a preliminary step, instrumental calibration was carried out by measuring several standard solutions (General Purpose Silicone Fluids) provided from the manufacturer, in particular four suspensions with the following expected viscosity: 5, 50, 500, and 5000 mPas.

## 2.3 EXPERIMENTAL PROTOCOL AND DATA ELABORATION

As the experimental procedure is crucial for the reliability and comparability of results the protocol employed in tests is hereafter summarized (according to [8]):

- all the experiments were conducted with a "shear rate controlled" measurement procedure, increasing up to the maximum supported by rheometer (i.e. 250 rpm). This value corresponds to different shear rate ramps, depending on adapter/spindle used to perform the analysis: e.g. from o to ≈ 300 s<sup>-1</sup> with a reading step of ≈ 12.5 s<sup>-1</sup> for the most diluted samples or from o to ≈ 60 s<sup>-1</sup> with a reading step of ≈ 1 s<sup>-1</sup> for the most concentrated ones
- each step lasted for 1 min (in order to guarantee reading stabilization)
- temperature of tests was set at 20 °C by means of a cryothermostat
- each test was performed with an upload/download cycle, in order to detect the possible creation of a hysteresis loop
- instrumental uncertainty was computed for each viscosity reading, according to the spindle/shear rate combination

Values of shear stress were plotted versus values of shear rate and interpolated by means of the Herschel-Bulkley model as presented in Equation 1 [3, 4, 8, 12, 14]:

$$\tau = \tau_o + K \dot{\gamma}^m \tag{1}$$

where K is the consistency index, m the flow index, and  $\tau_o$  the yield stress. The rheograms allow to compute the "apparent" viscosity, i.e. shear stress/shear rate ratio for each shear rate value. Finally, in order to test the repeatability of viscosity measurements a statistical analysis was carried out on several replicates (a total of 100) on the same sample (0.4 % TSS concentration of #1 WWTP) and data were elaborated with SPSS 19.0 statistics program (SPSS for Windows, Chicago, USA).

# 2.4 MICROBIOLOGICAL CHARACTERIZATION OF SLUDGE

The microscopic examination and image capture of sludge were performed with an optical microscope

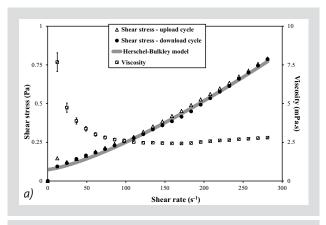
(Zeiss Axiolab). This device was used at a 400 and 1000 x magnification and allowed to assess floc size and the relative amount of filamentous bacteria in the different samples, according to Jenkins scale [15].

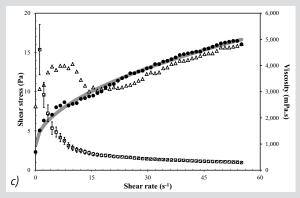
### 3 RESULTS

### 3.1 RHEOLOGICAL MEASUREMENTS

First of all, measurements carried out on standard solution evidenced that rheometer was perfectly calibrated, the approximation errors being lower than 2 % for each suspension (data not shown). Once instrumental reliability has been guaranteed, rheological tests were performed and findings are shown in Figure 1. In particular, the rheograms obtained for four samples of #1 WWTP are displayed (0.4, 1.8, 3.7, and 5.3 % in Figure 1a, b, c, and d), chosen in order to cover all the range of investigated TSS concentrations. Rheograms revealed the (expected) non-Newtonian behavior of AS, due to the nonlinearity of  $\tau/\dot{\gamma}$  curves, and were modelled according to the Herschel-Bulkley approach (solid line in Figures 1). Consequently, the viscosity cannot be assumed as a constant, varying as a function of shear rate (as displayed in Figures 1 as well based on the download cycle). For this reason, the selection of a fixed shear rate (i.e. ≈ 100 s<sup>-1</sup>) was needed for the comparison among different solid content: the related viscosity measurements (or predict by the model for the most concentrated samples unable to reach such a shear rate) are reported in Table 1.

In particular, the Herschel-Bulkley model fits the sample data quite well: The values of the Root Mean Square Deviation (RMSD), which represents a performance indicator for the 3-parameters model [8] were less than 10% except for those sludge samples processed with a specific spindle (SC4-34), which can then be considered unsuitable for the sludge. K and m values were determined through the download cycle rheograms and are summarized in Table 1, which shows a good agreement with available literature data [3, 4, 8, 12, 14, 16]. On the contrary, the numerical values of yield stress were not taken into account, as it strongly depends on the selected shear rate range, so it was only qualitatively considered in the model, being sludge with high solids concentration characterized by stress resistance during rest period [17]. For samples with high solids content (e.g. Figure 1c and d), the shear stress recorded elevated values at low shear rates (up to 20 s<sup>-1</sup>) probably due to the settling of suspended flocs. This hypothesis is further enhanced by the consistency of the download cycle rheogram in which particles are already suspended and any sedimentation mechanism





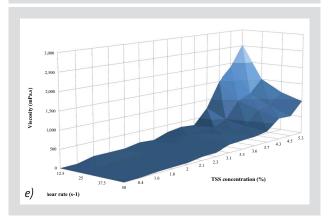
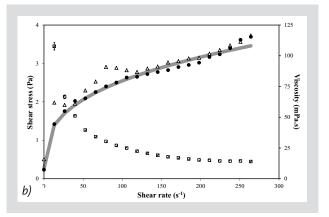
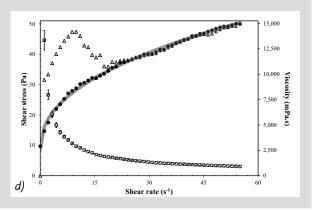


Figure 1: Examples of rheogram obtained for different sludge concentrations (#1 WWTP): 0.4 % a), 1.8 % b), 3.7 % c), 5.3 % d). Both shear stress/shear rate (primary axis) and viscosity/shear rate patterns (secondary axis, error bars representing instrumental uncertainty) are plotted. e) 3D chart reporting viscosity as a function of both TSS concentration and shear rate for #1 WWTP samples.

does not occur. The hysteresis loop observed at high sludge solid concentration is typical from a viscoelastic fluid, whose thixotropic parameters where described according to [18]: The hysteresis area H and hysteresis area as the percentage of the total area beneath the decreasing shear rate curve RH. The experimental values of H and RH, shown in Table 1 evidenced how the presence of solids imparts viscoelastic behavior to sludge: Only at higher TSS concentration a noticeable thixotropic pattern (with H > 100 Pa/s and RH > 10%) was recorded as a consequence of the physical energy needed to overcome the viscous resistance to internal structures breakdown.





Finally, a strong dependency of rheological properties on dry solids concentration can be highlighted (and in detail discussed in Section 3.3). Figure 1e reports viscosity values for #1 WWTP samples in a 3D chart as a function of both TSS content and shear rate, which affect the rheological properties of sludge in an opposite way, as also reported in general literature models [8]: The viscosity is directly proportional to solid content of sludge, on the contrary it decreases at increasing shear rates.

## 3.2 STATISTICAL ANALYSIS

A statistical analysis of viscosity was then performed on data obtained from n=100 replicates (of a 4 gTSS/L sample) and presented in Figure 2. As displayed by the histogram in Figure 2, viscosity data display a normal distribution: A Shapiro-Wilk normality test was performed with p-value of 0.1291. The standard deviation highlights a low level of variability (sd=0.014 mPas, mean=2.50 mPas, leading to a CV < 0.6%). A 99 % confidence interval for the mean was then computed (2.496, 2.505), which indicates an extremely small amount of variability. As a result the mean is a robust and reliable measure.

## 3.3 EFFECT OF SOLID CONTENT AND (MICRO)BIO-LOGICAL CHARACTERISTICS ON SLUDGE RHEOLOGY

The leading factor affecting AS rheological behavior is solid content [8]: For this reason, values obtained from experimental measurements were then correlated to dry solids concentration in sludge samples (Figure 3). As

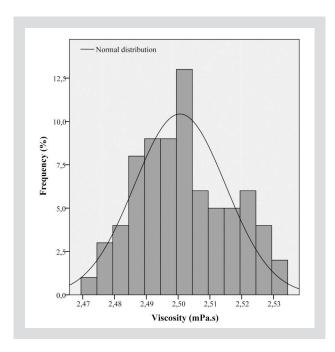


Figure 2: Statistical analysis, reporting frequencies diagram of viscosity measurement on a 4 qTSS/L sample.

expected [19] the relationship between viscosity and TSS was accurately represented by means of a power law as demonstrated by the high values of R2. Similarly, Herschel-Bulkley parameters (K and m) showed the same strong dependence on TSS: An increase of solids concentration was responsible for an increase of consistency index and on the contrary for a reduction of the flow index meaning a switch from dilatant (m > 1) to pseudoplastic (m < 1) fluid (Figure 4). Moreover, as a further outcome, both tested sludges displayed very similar behaviors: the parameters of  $\mu$ /TSS power law were comparable to those reported in the available literature, in particular close to the highest values. Several works [3, 8, 9, 12, 20, 21] were taken into account to generate the "literature review" pattern represented in Figure 3 together with the maximum and the minimum: Its high variability (filled area) is a consequence of viscosity measurement "sensitivity" (to the use of different devices, protocols, ...) as reported in the Introduction (Section 1).

The reason of this trend was successfully sought in sludge biological characteristics. From the macroscopic point of view, the Sludge Volume Index (SVI, an empirical indication of sludge settleability in the final clarifier) assumed alike values equal to 325 and 275 mL/g for #1 and #2 WWTP, respectively. Such values represent a straight warning towards possible critical situations (e.g. the onset of bulking phenomena). Likewise, the microscopic observation of AS revealed that both the plants were characterized by middle dimension flocs  $(150 - 500 \mu m)$  with a high level of filamentous bacteria. These features directly affect the physical behavior of sludge at the macroscopic scale, since filamentous bacteria colonize each floc (which is the morphological and functional unit of activated sludge process itself) and form bridges among flocs. Within this context, detected filament types were M. parvicella (Type 021N, Type

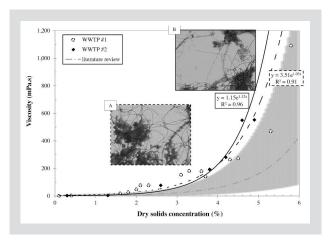
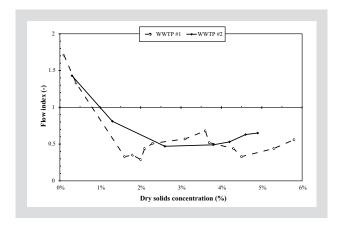


Figure 3: Viscosity increase as a function of dry solids concentration for two tested sludges, with details of floc observation (Gram stain, 1000 x magnification), and comparison with literature data (filled area representing its variability). Open circles, dotted line, and picture a: #1 WWTP. Filled square, solid line, and picture b: #2 WWTP.

oo41) for #1 WWTP and *M. parvicella* (Type 0092, Type 0914) for #2 WWTP. The dominant filament *M. parvicella* synthesizes oils, thus worsening sludge settleability. Also *Zooglea sp.*, which is strictly related to EPS production and the main onset of the so-called viscous bulking consisting in increased volume and tendency to flotation was detected in both plants. Within the Figure 3 two typical pictures captured during floc observation are reported displaying *M. parvicella* detected with Gram stain at 1000 x magnification.

### 4 CONCLUSION

In this work, the main rheological properties of AS were measured at different concentrations, thus simulating different stages of WWTPs, i.e. from biological reactor up to the digested sludge. A strict and defined protocol was carried out involving both the experimental measurement and the data processing: This is a key point for tests consistency and data comparability. Milestones for the rheological analysis were the determination of the rheogram (both upload and download cycle) together with the hysteresis area and the calculation of the viscosity at a set shear rate. As result, two sludges showed a quite similar rheological trend: i) both evidenced a non-Newtonian behavior, perfectly fitting with Herschel-Bulkley model, ii) a clear hysteresis loop was determined, as wider as higher the solid concentration, and iii) viscosity was determined at 100 s<sup>-1</sup> shear rate with values of a few mPas for AS in the biological reactor up to hundreds of mPas for a digested sludge. In conclusion, the solid content was the driving factor affecting rheological characteristics. In particular, the viscosity increased with dry solids concentration in sludge according to an exponential law which means a heavy boost of viscosity along the sludge treatment line. The viscosity was used as a tool to provide information also on the microbiological characteristics of AS and specifically to qualita-



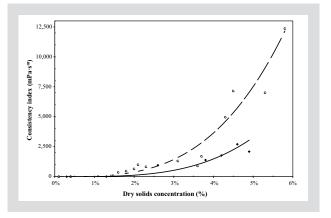


Figure 4: Flow index reduction (left) and consistency index increase (right) as a function of dry solids concentration of sludge.

tively foresee the amount of filamentous bacteria. Both tested sludges were characterized by elevated viscosity values, which corresponded to an over-proliferation of filamentous microorganism. Finally, as general outcome, this research evidenced that viscosity is a useful parameter whenever assessing CAS systems, and should be measured in order to properly design plant features (such as reactors, pipes, ...) and to obtain information on sludge settleability properties together with the traditional microbiological analysis of AS flocs.

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