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# EVOLUTION OF THE PROPERTIES OF A MUNICIPAL SEWAGE SLUDGE DURING FLOCCULATION AND CENTRIFUGATION

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## Abstract

Regardless the valorization or disposal route, a municipal sewage sludge must undergo transformations to reach the final desired properties. The knowledge of the properties of a sludge during its treatment is thus of prime importance for a better control of the final properties. The present study concerns the characterization of two aerated sewage sludges, sampled in an urban wastewater treatment plant before and after anaerobic digestion, during two essential steps: flocculation and centrifugation. The influence on the properties of polymer concentration and dry solid content is more particularly investigated. Several physico-chemical properties are used to monitor the structure evolution of the sludges: particle size distribution (Mastersizer 2000, Malvern instrument), zeta potential (Zetamètre nano S, Malvern) and rheological properties (RS150 Rheostress, Haake). Finally, the same optimal flocculant concentration is identified with the three techniques for both sludges. Moreover, the rheological properties of the sludges during the centrifugation is measured by using oscillatory mode for solid concentrations from 8 to more than 25 % of dry matter.

**Keywords:** sewage sludge, flocculation, centrifugation, rheology, zeta potential, particle size distribution.

## INTRODUCTION

Sewage sludge handling is becoming more and more problematic: its direct valorization as a cheap fertilizer is being limited and its production will increase with the generalization of wastewater treatment plants in European cities. In this context, sludge drying is essential in order to reduce handling and storage operations, but also as a pretreatment step before its potential energetic valorization. Besides a uniform treatment, the flexibility of the process, in order to adapt the final solid content of the dried sludge to the demand, will be a major requirement of any sludge drying system in the near future. Presently, this flexibility does not exist: one third of the dryers in operation are designed to reach a dry solid content of 65%, whereas in the other two thirds, the dry solid content exceeds 90%. Consequently, most of the time, the sludge is too dried and the energy consumption of the process is much higher than needed, particularly in paddle dryer technologies were excessive drying is often noted.

Regardless the valorization or disposal route, a municipal sewage sludge must undergo transformations to reach the final desired properties. The knowledge of the properties of a sludge during its treatment is thus of prime importance for a better control of the final properties. In the literature, sludge rheological behavior has been widely studied to provide information on the hydrodynamic behavior of sludge flow [1-3]. Nevertheless, most of these studies are focused on the study of sludges at low dry solids contents for the implementation of processes as bioreactors, digester or dewatering stage, which implied dry solids contents lower than 10%. Only few studies deal with the characterization of the rheological properties of sludges at higher solids content (>10%) in the fields such as landfill application [4,5] or drying application [6,7]

Rheological characterization can be performed by using two kinds of measurements: flow shear and dynamic measurements. Most of the studies used flow measurements as they are the simplest to carry out and to interpret. Nevertheless, as the solids content increases, this kind of measurements becomes difficult to perform due to apparition of phenomena such as wall slip [4]. In general, dynamic measurements are used to complete information obtained from flow measurements [1]. Indeed, flow measurements provides information about viscous and viscoplastic sludge properties and dynamic oscillatory measurements lead to structural properties of sludge [5]. However, it has to be noticed that several authors observed that rheological characteristics such as the yields stress determined by dynamic and flow measurements methods are of the same order of magnitude [5-8].

The present study concerns the characterization of two sewage sludges during flocculation and centrifugation stages. These two sludges were sampled in an urban wastewater treatment plant (WWTP) before and after anaerobic digestion stage. The influence of polymer flocculent concentration and dry solids content were investigated on several properties: particle size distribution, zeta potential and rheological properties.

## MATERIALS AND METHODS

### Sludge samples and flocculants used

The sewage sludge was sampled in the WWTP of Albi city (France), designed for a capacity of 91000 population equivalent (PE) but operating at 60000 PE. It implements a conventional extended aeration step, a nitrification/denitrification and a biological phosphorus removal. The aerated sludge was sampled at the outlet of the aeration treatment, whereas the digested sludge was sampled after the anaerobic digestion stage. After sampling, the total solids content (TS), total volatile solids content (TVS) and total fixed solids content (TFS) were determined (table 1). TS describes the remaining residue after drying of the wet sample at 105 °C, TVS those solids that can be volatilized and burned off when total solids are ignited at 550 °C and TFS the residue that remains after this ignition. In the present study, analyses were carried out with an oven (Heraeus, Hanau, Germany). TVS and TFS are expressed in weight percent on dry basis while TS is usually expressed as a weight percent of the moist sludge. The relative standard deviations (RSD) have been estimated on the basis of two different experiments.

*Table 1: Total solids content (TS), total volatile solids content (TVS) and total fixed solids content (TFS) of the raw sludges.*

| | TS (%) | TVS (%) | TFS(%) |
|---|---|---|---|
| Aerated sludge | 6.2 ± 0.6 | 73.2 ± 1.6 | 26.8 ± 1.5 |
| Digested sludge | 3.5 ± 0.2 | 64.1 ± 1.9 | 35.9 ± 1.9 |

Two cationic emulsions were supplied by the Albi WWTP and were used for the flocculation stage: the BASF Zetag® 8868 with the aerated sludge and the BASF Zetag® 9018 for the digested sludge. Before using, the two commercial polymers were diluted in demineralized water at different concentrations varying from 3 to 6 g/L.

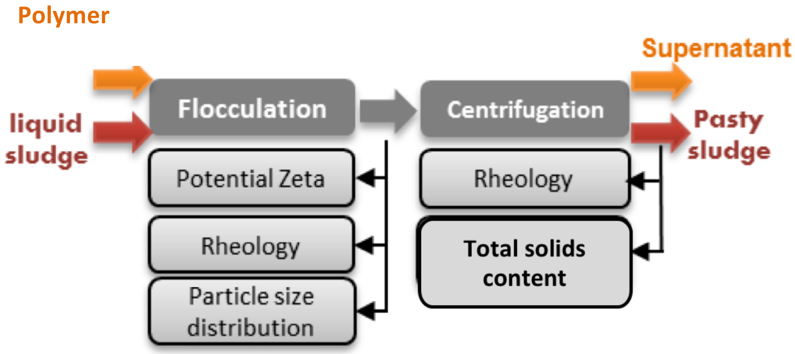


Figure 1: Experimental procedure

### Experimental procedure

The global experimental procedure is depicted on figure 1.
To investigate the influence of the flocculation stage, a jar test method was performed. The protocol used, depicted in figure 2, was adapted from the method developed by Dihang for the flocculation of clay suspensions [9].
For the study of the effect of the dry solids content, each sludge was flocculated at its optimum polymer concentration, previously determined, and then thickened in laboratory centrifuges at different velocities (3100-10000g) and durations (15 min-3 h). The supernatants was then discarded and the rheological characterization was performed before the determination of the dry solid content for each sample (figure 1).

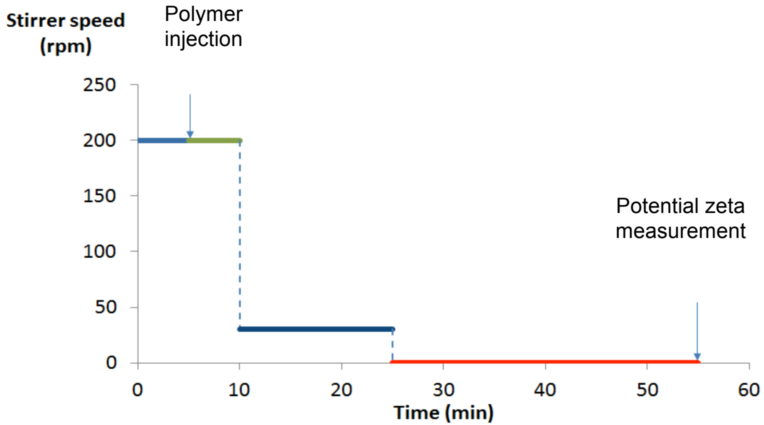


Figure 2: Jar test protocol

## Particle size distribution

Particles size distribution (PSD) of the raw sludge is determined by means of a Mastersizer 2000 particle size analyser (Malvern Instrument Ltd., Worcestershire, United Kingdom). This laser diffraction system uses the Mie theory to characterize the particles over a wide size range, from 0.2 to 2000 µm. Water is used as dispersion media (100 mL).
At first, the background noise is measured for water before the sludge introduction ($n_{refraction}$=1.33). This reference state corresponds to a null obscuration. Then, the sludge is quickly added in the measuring cell and the analysis starts as soon as the obscuration stabilizes around 20%, guaranteeing a good dispersion of the product. The result is considered for a sample when the variation between consecutive measurements of the De Brouckere mean diameter, $D_{[4,3]}$ defined by Eq. (1), is lower than 5%.
It should be noted that, to applied the Mie theory, the refraction and absorption indexes of sludge have been set by default (refraction=1.52 and absorption=0.1). The PSD is determined in volume and a sludge aggregate, which is likely to be irregular in shape and non-spherical, is substituted by an imaginary sphere having the same volume. Each sample was tested in duplicate and the average value was used in this study.

$$D_{[4,3]} = \frac{\sum D_i^4 n_i}{\sum D_i^3 n_i} \tag{1}$$

## Viscoelastic properties

A RS150 Rheostress rheometer (Haake, Karlsruhe, Germany) was used in oscillatory mode to characterize the sludge physical structure. In order to avoid wall slip, parallel-plate geometry (35 mm diameter and 2 mm gap) with rough surfaces was selected for these analyses [4]. During measurements, the product temperature was kept at 20 °C by means of a Peltier element connected to a thermal bath. The rheometer is coupled to a RheoWin software (Haake, Karlsruhe, Germany), which allows to obtain rheograms and data analysis.

Rheological characterizations were performed by means of dynamic measurements, which allow to determine the storage modulus G', the loss modulus G", and accordingly the complex modulus G*, and the loss angle δ. A strain sweep from 0.001 to 1 is applied at a constant pulsation ω of 6.2 rad.s$^{-1}$ for the evaluation of the linear viscoelastic behavior range, in which the material modulus G' and G'' do not depend on the magnitude of the deforming strain. All the experiments were made in duplicate, the sample being discarded after each measurement.

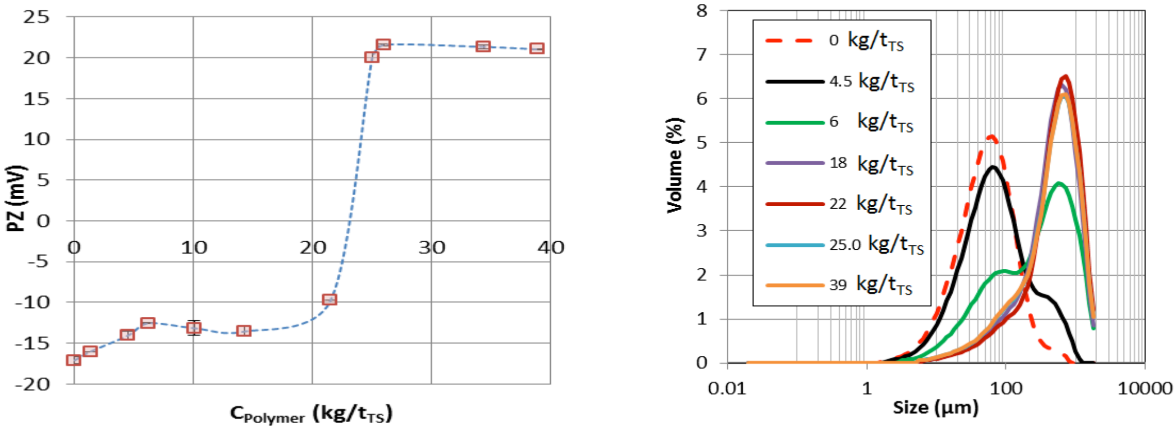
### Zeta potential

Zeta potential was determined using a Zetasizer Nano ZS (Malvern Instrument Ltd., Worcestershire, United Kingdom) at 25 °C. Measurements were made in a standard disposable cuvettes Malvern dip cell (0.7 to 1.5 mL). For each sample, the average of two runs was considered.

## RESULTS AND DISCUSSION

### Flocculation

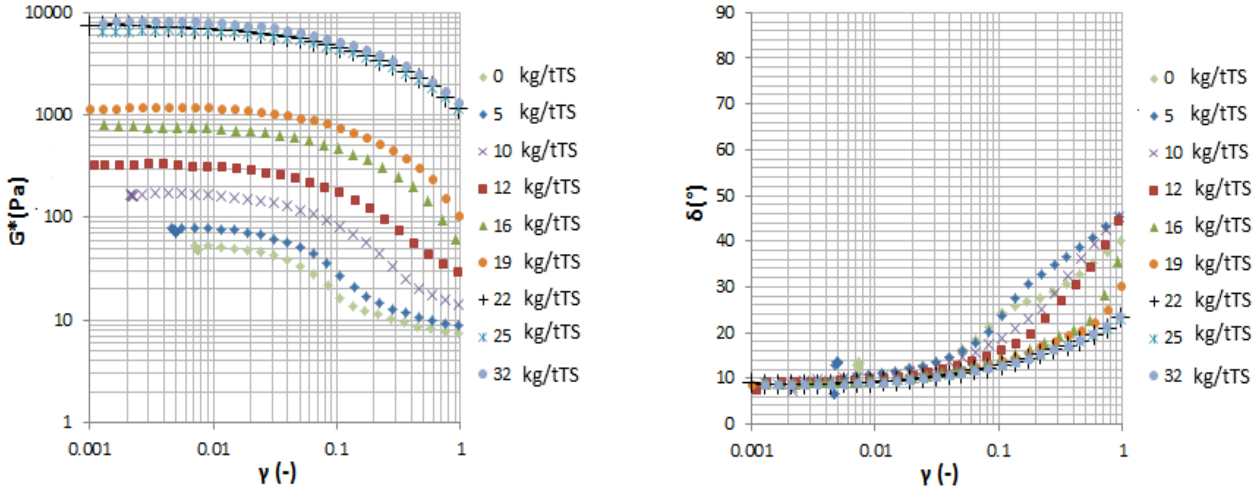
Figure 3-a shows the evolution of the zeta potential as a function of the polymer concentration for the digested sludge. The zeta potential of raw sludge is measured at -17 mV. At low values, as the polymer concentration increases, the zeta potential increases slightly to -10 mV for a polymer concentration equal to 21.5 kg/$t_{TS}$. Above this value, the polymer concentration exhibits a marked effect on the zeta potential with a sharp charge reversal from -10 mV to 20 mV. The concentration at which the zeta potential is equal to zero, called the isoelectrical point, is about 22.5 kg/$t_{TS}$. Beyond this concentration, the addition of polymer no longer changes the zeta potential. These observations are consistent with the PSD shown in figure 3-b. Indeed, the increase of the polymer concentration shifts the PSD toward bigger particles diameters, highlighting the formation of flocs, aggregates and sludge structure. In comparison with the raw sludge, the De Brouckere Diameter is multiplied by 7. As for the zeta potential, the PSD does not change significantly at higher polymer concentrations.



(a) (b)

Figure 3: Evolution of zeta potential (a) and PSD (b) as a function of the polymer concentration

The rheogram of the complex modulus G* and the loss angle δ as a function of strain are presented in figure 4 at different polymer concentrations. Each curve can be divided in two parts. In the first one, called linear viscoelastic region, the rheological properties are independent of the strain until a critical strain value. In this region, regardless of the polymer concentration, the loss angle δ is always close to 9°, what indicates the majority contribution of the elastic modulus G' in the complex modulus G* (~98 %). It shows that, below the critical strain, sludge samples behave like a solid. In the region above the critical strain, a decrease of the complex modulus G* and an increase of the loss angle δ are observed with the strain increase. In this region, the sludge behaviour is nonlinear and depends on the strain applied, highlighting the structural evolution of the sample. Several authors attribute this change to a transition of the sludge from a solid-like to a liquid-like behaviour, typical of pasty materials [5, 10].



(a) (b)

Figure 4: Complex modulus G* (a) and the loss angle δ (b) as a function of strain at different flocculant concentrations

The increase of the complex modulus G* indicates the increase of the sludge structure with the increasing concentration of the polymer (figure 4). These results indicate that the use of a polymer induces a significant increase of the overall shear resistance of the sludge [8, 11]. Above a polymer concentration of 21.5 kg/$t_{TS}$, all the curves are superimposed, indicating that additional polymer has no more influence on the sludge viscoelastic properties and thus on its structure.

A summary of the experimental characterisations of the digested sludge is presented on figure 5. The represented complex modulus G* value corresponds to the one in the linear viscoelastic region. Finally, it highlights that zeta potential, PSD and rheological characterization are three complementary techniques to follow the evolution of the sludge during the flocculation step. At low concentrations, the addition of cationic polymer induces the neutralization of negatively charged particles and their attachment on the polymeric chains. This step is also associated to the increase of the zeta potential, the PSD and the viscoelastic properties of the sludge. Once the optimal polymer concentration is exceeded, a sharp inversion of the zeta potential occurs from negative to positive values, while the viscoelastic properties and the De Brouckere diameter do not change significantly, indicating that addition of polymer has no further effect on sludge properties.

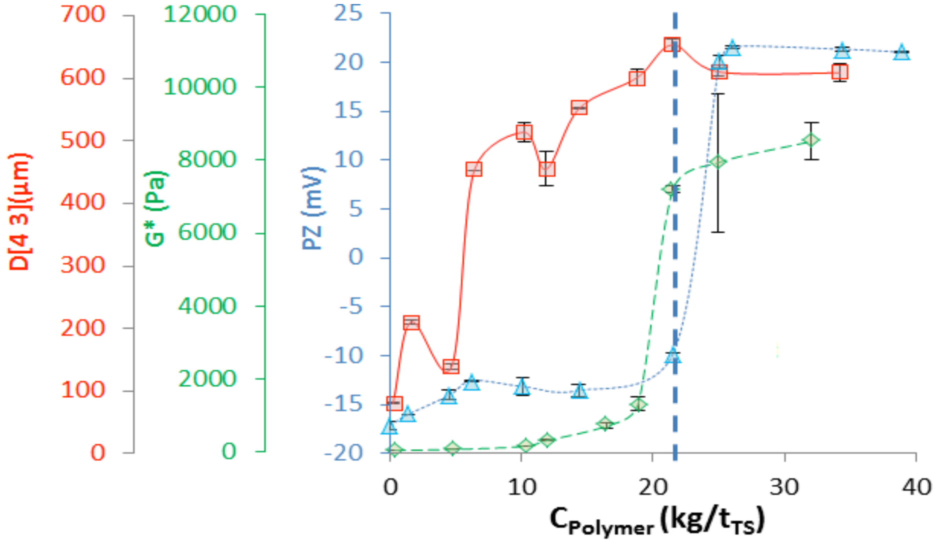
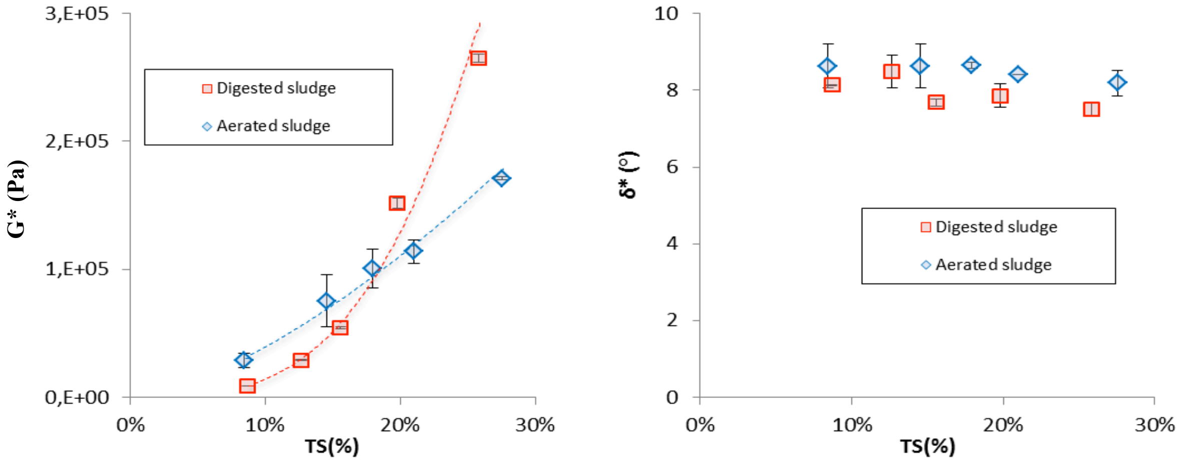


Figure 5: Summary of the experimental characterizations of the digested sludge

The results are not presented in this document for the aerated sludge and the BASF Zetag® 8868 polymer, but it should be noted that similar observations were obtained. Finally, the optimal polymer concentrations were identified at 21.5 and 24 kg/$t_{TS}$ for the digested and the aerated sludge respectively.

## Centrifugation

Many authors have examined the effect of solids concentration on sludge rheological properties by using flow shear measurements [2]. Generally, the evolution of sludge rheological properties as function of the solids content can be represented by either an exponential or a power law. Figure 6 depicts the evolution of the complex modulus G* and the loss angle δ as a function of the total solids content for the aerated and digested sludges. The loss angle δ is constant whatever the dry solids content, at about 9° for both sludges, what means both sludges exhibit the same viscoelastic behaviour (Figure 6-b). Moreover, the complex modulus G* increases with the increase of the total solids content, indicating a more rigid network (Figure 6-a). These observations are consistent with previous ones from the literature [5, 7, 12].



(a) (b)

Figure 6: Rheological parameters evolution as a function of the total solids content for the aerated and digested sludges

The increase of the rheological parameters G', G" and G* with the increase of the total solids content follows a power law which can be described by the Equation 2.

$$G = B * TS^a \tag{2}$$

The correlation coefficient, $r^2$, and the parameters of Eq. (2) for the aerated and digested sludges are listed in the table 2. For both sludges, the elastic modulus G' is still the majority component of the complex modulus G* whatever the solids content, confirming the strong predominance of the solid-like behaviour. The slope of the curves, a, for the digested sludge is close to the one obtained by Agoda-Tandjawa et al. for the dewatering of an activated sludge with and without a flocculation stage [12].

*Table 2: Parameters of Equation 2*

| | Aerated sludge | | | Digested sludge | | |
|---|---|---|---|---|---|---|
| | G' | G" | G* | G' | G" | G* |
| B | 1238.2 | 206.4 | 1243.1 | 8.4 | 1.49 | 8.47 |
| a | 1.50 | 1.46 | 1.50 | 3.22 | 3.13 | 3.22 |
| $r^2$ | 0.99 | 0.98 | 0.98 | 0.99 | 0.99 | 0.99 |

These results show that, for the same total solids content above 20 %, the digested sludge structure is stronger than the aerated sludge one. From this observation, it could be conclude that the flow of the digested sludge will be more difficult.

## CONCLUSION

This study focuses on the properties evolution during flocculation and centrifugation stages of two sludges, sampled in an urban wastewater treatment plant before and after anaerobic digestion. The properties of the sludge are characterized by three different techniques: a zetasizer, a particle size analyser and a rheometer.

According to the results obtained, zeta potential, particles size distribution and viscoelastic properties can be used to monitor the flocculation stage of a sludge with a cationic polymer. This allows to highlight the influence of the polymer type on the created structure and thus on the viscoelastic properties of both sludges.

The mechanical dewatering stage indicates that the evolution of the viscoelastic properties as a function of the total solids content can be described by a power law in the case of the two sludges.

One limit of this study concerns the conditions of samples dewatering. Indeed, it wasn't possible to reach a solids concentration higher than 27%. Therefore, it would be interesting to complete this work with another dewatering techniques, such as for instance filters press, to reach higher solids content typically encountered in drying processes. Moreover, this work could be completed with the study of the influence of the temperature on the sludge structure and viscoelastic properties.

## NOMENCLATURE

| | | |
|---|---|---|
| PSD | Particle size distribution | |
| WWTP | Waste water treatment plant | |
| $C_{polymer}$ | Flocculant concentration | ($kg/t_{TS}$) |
| D[4.3] | De Brouckere diameter | (µm) |
| G' | Storage modulus | (Pa) |
| G'' | Loss modulus | (Pa) |
| G* | Complex modulus | (Pa) |
| PZ | Zeta potential | (mV) |
| TS | Total solids content | (%) |
| TVS | Total volatile solids content | (%) |
| TFS | Total fixed solids content | (%) |
| t | Time | (s) |

### Greek letters

| | | |
|---|---|---|
| $\gamma$ | Strain | (-) |
| $\omega$ | Angular pulsation | ($rad.s^{-1}$) |

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