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# Using water activity measurements to evaluate rheological consistency and structure strength of sludge

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## HIGHLIGHTS

- In this paper, we establish a link between sludge rheological properties and water activity.
- We show that $G'$ and $G''$ increase with solid concentration while water activity decreases.
- We show that $G'$ and $G''$ decrease with ageing time while water activity increase.
- We define water activity as an easy to use parameter to evaluate network strength of sludge.

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## ABSTRACT

In this study, an original method of characterization of water/solid matter interactions in sewage sludge has been developed, based on both rheological characteristics and thermodynamic water activity determinations, in order to check whether a link could be made with water activity and its mechanical properties. The effect of solid matter content, flocculation and ageing time on water activity and rheological parameters has been investigated. Through this study, we showed that rheological parameters ($G'$ and $G''$) of both raw and flocculated sewage sludge at optimal dose of polymer increase with solids concentration following a power-law, whereas in the same way water activity decreases following an exponential relationship (Arrhenius dependence). A slight increase of water activity values and those of $G'$ and $G''$ moduli with added polymer was also highlighted. On the other hand, we have shown that during ageing, the rheological parameters $G'$ and $G''$ decreased upon increasing the ageing time whereas in the same way, water activity increases. Rheological parameters clearly evidenced thermodynamic water activity dependence, regarding solid concentration and ageing time, with a decrease of $G'$ and $G''$ upon increasing water activity. Thus, water/solid matter interactions were supposed to intervene importantly in the rheology of sewage sludge. From all these results, it appears clearly that water activity measurements can be regarded as an effective and easy tool for evaluating the state of structuration of sewage sludge and for predicting its structural, textural and mechanical properties during dewatering and ageing.



## 1. Introduction

Nowadays sludge management is of major concern due to environmental pressure and economic considerations. Optimizing treatment process is a great challenge and an accurate estimation of sludge rheological properties and sludge structure strength is required for the design of pumping systems or for the control of the dewatering steps [1]. Slatter [2–5] has also consistently shown that rheology plays a fundamentally important role in analyzing the hydrodynamic behavior of the sludge, as it flows in pipes and reactors. However, several papers underlined that consistency and strength of the structure are not directly linked to solid content [6–9]: the qualitative composition of sludge is of much greater importance than quantitative composition (i.e. basic solid content). This is related to the presence of extracellular polymeric substances (EPSs) of different nature, depending on the type of sludge and the influence of sludge treatment. EPSs are highly charged polymers that interact with water in a similar way as gels [10–13], influencing the strength of the structure regarding the nature of interactions between solids and water which can be balanced between steric and electrostatic [35].

As a consequence, great attention has been paid to water repartition within sludge. For example, extensive studies, largely reviewed by Vaxelaire and Cézac [14], have been performed to estimated water states within activated sludge by using several techniques such as: differential scanning calorimetry (DSC) [15], dilatometry [16,17], drying [16], combined thermal gravimetry analysis and differential thermal analysis [18], nuclear magnetic resonance spectroscopy [19], centrifugal settling [20] and

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desorption isotherms [21]. Based on these studies, four categories of water in sludge were classically distinguished:

- Free water, which represents the largest part of the sludge. Thermodynamically, it behave as pure water which is not associated with solid particles and including void water not affected by capillary forces [22].
- Interstitial water, which is trapped inside crevices and interstitial spaces of flocs and organisms [17,22,23].
- Surface (or vicinal) water, which held onto the surface of solid particles by adsorption and adhesion [17,22,23].
- Bound (or hydration) water [17,22,23].

Besides this classification and considering the impact of the polymeric matrix which constitutes activated sludge, Mikkelsen and Keiding [12] have introduced the term "water-holding" to cover both surface bound water and osmotic water as well as water trapped within the polymer network. Thus, sewage sludge can be seen as soft matter and improving knowledge on water/solid matter interactions is one of the clues to improve sludge rheology, especially network strength which plays a fundamental work in conditioning and dewatering [36]. In addition, as rheological properties of soft matter are strongly related to interactions among particles dispersed in medium [24,25], it is not surprising that the state of water could appear as a relevant parameter to evaluate sludge consistency and structure strength.

One can easily relate the way water is linked to solid matter to its fugacity. Fugacity is the escaping tendency of a substance and the activity of species is defined as the ratio between the fugacity $f$ and the escaping tendency of a pure material, $f_0$ [37]: $A = f/f_0$. When dealing with water, a $w$ subscript is designated for the substance: $A_w = f/f_0$. $A_w$ is defined as the activity of water, or the escaping tendency of water in the considered system divided by the escaping tendency of pure water (with no radius of curvature). As the state of water in suspensions arises from interactions between water molecules and solid matter in the medium, coupling water activity with rheological properties will thus be an attempt to obtain information on the effects of water/solid mater interactions on the rheological properties of sewage sludge and the strength of its structure.

Thus, this work focuses on the characterization of the water/solid matter interactions, based on water activity measurement and on the sludge rheological properties, in order to check whether a link could be made with sludge structure. By focusing on the impact of solid concentration, conditioning and ageing time, we show that rheological properties and water activities can be connected together and that water activity may be used as a quick and easy-to-use tool to evaluate strength of sludge structure.

## 2. Materials and methods

### 2.1. Materials

Activated sludge was sampled at the Varennes-sur-Allier waste water treatment plant (Allier, France). The samples had a pH between 6.8 and 7.2, conductivity between 1105 and 1240 μS cm$^{-1}$ and dry matter content between 1.22% w/w and 1.35% w/w. After sampling, sludge was gently concentrated to 1.9% w/w by decantation and stored at 4 °C for maximum 3 days, to reduce temporal variability effect of biochemical composition change (fermentation).

As described later, for experiments, sludge was thickened with and without conditioning.

For sludge conditioning, structured cationic polyacrylamide in emulsion form (polymer FLOPAM EM 840 from SNF SAS product range, Andrézieux, France) was used. This conditioning polymer was also sampled at the Varennes-sur-Allier waste water treatment plant. Polymer solutions (47.53% w/w) were diluted at 0.63% w/w in deionized water under gentle stirring at room temperature for 6 h, according to instructions given by the manufacturer (SNF SAS, Andrézieux, France). Thus, polymer solutions were prepared at least 24 h prior to application as described by Saveyn et al. [26].

### 2.2. Sludge conditioning

A Stuart Scientific Jar Test device was used. The procedure was as follows: 200 rpm during 2 min for intense mixing of the polymer into the sludge, followed by a 8 min slow stirring period at 30 rpm to promote floc growth. Half a liter of sludge (1.43% w/w) was mixed with different doses of polymer. The optimum dose of polymer was determined with capillary suction time (CST) procedure, using a Triton Electronics 304 M CST meter. Each measurement was performed five times.

In this work, sludge was flocculated at the defined optimum dose of polymer. After conditioning, the samples were drained with a coffee filter during 4 h.

### 2.3. Thickening and ageing tests

Sludge was divided into several identical samples, then thickened in a laboratory ultra-centrifuge, at a fixed temperature and fixed duration but at different velocities corresponding to centrifugal accelerations ranging from 4000 to 12,000*g* for the raw sludge and from 2000 to 14,000*g* for the flocculated sludge. The operating conditions are the following:

- time of centrifugation: 5 min,
- temperature: 20 °C,
- bowl: designed for six tilted tubes of unit volume of 40 ml.

After centrifugation, solids concentrations were determined by weighting the dry residue after drying at 105 °C for 24 h. The corresponding solids concentrations of the thickened samples are reported in Table 1.

After thickening, sludge was stored at 4 °C to limit fermentation. Before experiments, sample was first acclimatized for about 40 min in a water bath at 20 °C. Another separate sample (at 10.5% solids) was stored at room temperature (20 °C) during 16 days to focus on the impact of ageing.

Note that centrifuge may alter sludge structure and modify rheological and water activity parameters but same considerations will be made with mechanical dewatering or soft drying. Thus, in

**Table 1**
Solids concentrations of raw and flocculated sludge after thickening at different centrifugal accelerations.

| Centrifugal accelerations (g) | Solids concentrations (%, w/w) | |
|---|---|---|
| | Raw sludge | Flocculated sludge |
| 2000 | 1.9 | 8.78 |
| 3000 | – | 8.81 |
| 4000 | 3.2 | 10.28 |
| 5000 | 5.28 | 10.48 |
| 6000 | 6.28 | 11.56 |
| 7000 | 6.58 | – |
| 8000 | – | 13.16 |
| 9000 | 9.60 | 14.00 |
| 10,000 | 10.71 | – |
| 12,000 | 12.78 | 15.44 |
| 13,000 | – | 15.60 |
| 14,000 | – | 15.82 |