Chemical Engineering Journal 255 (2014) 14–22

Contents lists available at ScienceDirect

Chemical Engineering Journal

journal homepage: www.elsevier.com/locate/cej

Effect of ultrasound, thermal and alkali treatments on the rheological profile and water distribution of waste activated sludge

Maria Ruiz-Hernando, Francesc-Xavier Simón, Jordi Labanda *, Joan Llorens

Department of Chemical Engineering, University of Barcelona, C/Martí i Franquès 1, 6th Floor, 08028 Barcelona, Spain

HIGHLIGHTS

- Waste activated sludge was subjected to ultrasound, thermal and alkali treatments.
- The treatments reduced the elasticity and zero shear rate viscosity of the sludge.
- The treatments reduced the steady state viscosity and thixotropy of the sludge.
- The bound water content in the sludge increased when treatment intensity increased.
- Despite increasing bound water, the treatments were able to improve sludge dewatering.

ARTICLE INFO

Article history:
Received 9 April 2014
Received in revised form 6 June 2014
Accepted 10 June 2014
Available online 18 June 2014

Keywords:
Waste activated sludge
Rheology
Bound water
Ultrasound treatment
Thermal treatment
Alkali treatment

ABSTRACT

Waste activated sludge is difficult to dewater, which hinders handling operations, such as pumping, transport and disposal. An accurate measurement of the distribution of water in sludge may help identify dewatering problems. Bound water, which is a gross estimate of several states of water including vicinal water, water of hydration and some fraction of interstitial water, is considered one of the primary limiting factors affecting sludge dewatering. In the present study, waste activated sludge was subjected to ultrasound, thermal and alkali treatments based on the assumption that these treatments can partially disintegrate sludge by disrupting flocs and cells and releasing interstitial water and organic compounds in the liquid phase, thus improving sludge dewatering. The three aforementioned treatments resulted in the reduction of the steady state viscosity and the hysteresis area of the sludge, and this reduction was more significant as the treatment intensity was increased. Likewise, the treatments also reduced the sludge elasticity and zero shear rate viscosity. Conversely, the bound water content increased when the treatment intensities increased because the organic matter released after sludge floc disruption created extra surfaces for water binding. Nevertheless, the centrifugation tests revealed that the three conditions of the thermal treatment (60, 80 and 90 °C) and the higher intensities of ultrasound (27,000 kJ/kg TS) and alkali (157 g NaOH/kg TS) treatments improved sludge dewatering by releasing the interstitial water.

1. Introduction

Waste activated sludge (WAS) is characterised by its high water content and its difficulty to dewater, which hinder handling operations, such as pumping, transport and disposal. Thus, knowing how water is distributed within the WAS is essential to examine dewatering problems [1]. Vaxelaire and Cézac [2] reported that the behaviour of a molecule of water during the dewatering process is widely dependent on its proximity to the solid. Usually, two primary types of water are identified: the free (or bulk) water, which is not influenced by the solid particles and can be easily removed, and the bound water, whose properties are modified due to the presence of particles. Thus, bound water is generally considered a gross estimate of several states of water including vicinal (or surface) water, water of hydration and some fraction of interstitial water [1]. The vicinal water is tightly held on the surface of particles by adsorption and adhesion and can only be removed by changing the surface quantity able to adhere water. The water of hydration is chemically bound to the particles and can only be removed by thermal dehydration. Conversely, the interstitial water, which is the water trapped in interstitial spaces of flocs and microorganisms,

---

* Corresponding author. Tel.: +34 934031334; fax: +34 934021291.
E-mail address: jlabanda@ub.edu (J. Labanda).

http://dx.doi.org/10.1016/j.cej.2014.06.036
1385-8947/

can be freed if the sludge floc is broken up and the microbial cells are disrupted. Vesilind [1] reported that the effect of particles on interstitial water is unclear and suggested that some part of the interstitial water may not physically behave as free water because vicinal water can also be located within the microbial cells as long as it is associated with the surface of the particle. There are various methods for measuring the bound water based on the principle that free water, but not bound water, has the same thermodynamic properties of pure water [3–9]. Among these methods, the differential scanning calorimetry (DSC) test is a suitable technique for measuring bound water by permitting a direct thermal analysis of phase changes of free water.

The low dewaterability of the WAS, which is primarily formed by microbial cells, can be improved by applying treatments, such as ultrasound, thermal or alkali. These treatments can partially disintegrate the WAS by disrupting flocs and cells and solubilising the extracellular polymeric substances (EPSs), thus releasing part of the interstitial water trapped in sludge flocs [10–12]. Accordingly, these treatments affect the viscosity and the filterability of sludge [11] because of the reorganisation of the sludge flocs with the EPSs and free water molecules. Rheology, which is the study of stress–strain relationships of elastic plastic and viscous materials, is a useful tool for the characterisation of WAS and to control sludge treatment processes for dewatering [13]. It is also useful for evaluating the pressure drops in pipes for sludge transportation by pumps [14–16]. Rheological measurements performed within the linear viscoelastic region, far away from the normal flow conditions, can be used to describe the internal structure of WAS because the applied stress does not change the internal structure of materials. Conversely, when the stress changes the structure of the material, the measurements are performed within the non-linear viscoelastic region, and viscosity is dependent on the applied stress (non-Newtonian behaviour). Under normal flow conditions, WAS usually behaves as a non-Newtonian pseudo-plastic fluid [17], which indicates that the viscosity decreases with the applied shear rate. The Ostwald–de Waele model is commonly used to represent the non-Newtonian behaviour of sludge because provides good fitting [18]. Other models, such as the Herschel–Bulkley model or the Bingham model also provide good fits. In contrast to the Ostwald–de Waele equation, these models are characterised by the presence of yield stress, below which the sample does not flow. However, one fundamental problem with the concept of yield stress is the difficulty in determining the true yield stress due to the method [19] and shear banding at low shear rates [20]. WAS is also thixotropic, which means that the viscosity is time dependent. The hysteresis loop area is a common method for measuring thixotropy [21], which consists of measuring the area enclosed between the up- and down-curve in a plot of shear stress vs. shear rate when shear rate is linearly increased and decreased over time. However, the hysteresis area is a relative measurement of the thixotropy because it depends on the design parameters of the test, such as the elapsed time and the maximum shear rate [22]. Nevertheless, when the same test is performed, the hysteresis area is very useful in determining the variation of the thixotropy.

The aim of the present study was to compare the effect of the ultrasound, thermal and alkali treatments on WAS rheology, water distribution and dewatering. First, the rheological behaviour of the WAS was analysed within the linear viscoelastic region by the creep assay and within the non-linear viscoelastic region using the hysteresis loop and shear rate step assays. Second, the bound water content of untreated and treated sludge was quantified by DSC. In addition, the effect of the treatments on sludge-water separation was evaluated by directly measuring the water removed by centrifugation.

2. Materials and methods

2.1. Waste activated sludge samples

The WAS samples used in this study were taken from a municipal wastewater treatment plant (WWTP) near Barcelona (Spain). At the WWTP, the WAS was thickened after leaving the secondary tank by centrifugation. Prior to centrifugation, a cationic polyelectrolyte (about 1 kg/ton dry sludge) was added in order to enhance sludge dewaterability. The total solid (TS) content was quantified in the laboratory following a standard method 2540 G [23]. The sludge was stored at 4 °C to minimise the bacterial activity until it was used.

2.2. Treatments conditions

Ultrasound, thermal and alkali treatments were studied. The ultrasonic apparatus consisted of a HD2070 Sonopuls Bandelin Ultrasonic Homogeniser equipped with a MS 73 titanium microtip probe (Bandelin, Berlin, Germany; 20 kHz). The ultrasonication power was fixed at 70 W, and the exposure times were changed to provide different specific energies (E_S): 5000, 11,000 and 27,000 kJ/kg TS. Higher E_S were not considered because little difference in viscosity was detected in a previous paper between 27,000 (72.5%) and 33,000 kJ/kg TS (72.7%) at a shear rate of 30 s^{-1} [24]. The E_S applied to the sludge was calculated as:

$$E_S = \frac{P \cdot t}{V \cdot \mathrm{TS}} \qquad (1)$$

where P is the ultrasonic power, t is the application time, V is the sample volume and TS is the concentration of total solids. To minimise the increment of sludge temperature due to the thermal effect of the cavitation phenomenon, the beaker containing the samples was submerged in an ice bath. Therefore, the temperature of the sludge samples after ultrasonication never exceeded room temperature.

The thermal hydrolysis was performed in a closed tank (Autoclave Engineers) at 60, 80 and 90 °C. Temperatures above 100 °C were not examined to avoid water loss by evaporation, which would interfere with sludge rheology. A heating jacket was placed on the outside of the tank to provide heat to the sludge sample (115 ml) contained inside the tank. The treatment lasted until the temperature reached the desire value (approximately 30 min) and was then maintained for 30 more min. The resulting E_S were: 11,000, 15,000 and 18,000 kJ/kg TS. The WAS was mechanically agitated during the thermal treatment to ensure the temperature homogeneity of the sample.

The alkali conditioning was conducted at room temperature (25 °C) by adding different concentrations of NaOH (35.3, 70.6 and 157 g NaOH/kg TS); the samples were subsequently neutralised with HCl in the pH range of 6.5–7.5 after 24 h. The pH values (±confidence interval 95%) of these concentrations just after adding the NaOH were 9.9 ± 0.9, 11.2 ± 0.6 and 12.5 ± 0.2, and after 24 h (before neutralisation) were 6.9 ± 0.2, 9.1 ± 0.7 and 12.3 ± 0.2, respectively. Furthermore, the effect of the neutralisation at a higher pH range (8–12) was also tested.

2.3. Rheological tests

The rheometer used in this study was a Haake RS300 control stress rheometer (Germany) equipped with HAAKE Rheowin Software. Measurements were conducted at 22 ± 0.1 °C. The rheological characterisation was performed at a constant TS (45.9 g/L) because the rheological properties of sludge are highly conditioned by this parameter [25–27].