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Variations of internal structure and moisture distribution in activated sludge with stratified extracellular polymeric substances extraction

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ABSTRACT

The floc rupture and fractal structure during the ultrasonic pretreatment, rheological parameters as well as bound water content of activated sludge before/after extracellular polymeric substances (EPS) extraction was investigated to examine the variations of internal structure and moisture distribution in activated sludge with stratified EPS extraction. It was observed that the floc disruption was dominated by the model of large-scale fragmentation initially and by smaller-scale surface erosion subsequently for activated sludge before/after EPS extraction. Activated sludge with tightly bound EPS (TB-EPS) showed greater mass fractal dimension (D_f) and rheological parameters hysteresis loop area, limiting viscosity (η_∞), yield stress (τ_y), energy of cohesion of network structure (E_c) and shear modulus (G) than the other sludge samples, indicating that activated sludge after loosely bound EPS (LB-EPS) extraction exhibited a denser and stronger network structure. A water classification is also proposed: free water (bulk water and some portion of interstitial water) and bound water (osmotic, surface and intracellular water). The osmotic water (interstitial-bound water) in the LB-EPS fraction (4.3% of the total water content) exhibited much more than that in the TB-EPS (0.2% of the total water content). Bound water (surface and intracellular water) in the TB-EPS and Pellet comes to be the subject to be treated after mechanical dewatering.

1. Introduction

Activated sludge process has been the standard method for wastewater treatment with the production of large amounts of activated sludge (Kim et al., 2013). The activated sludge dewatering is crucial for reducing the volume of the activated sludge, decreasing the subsequent treatment and disposal operations (Li and Ganczarczyk, 1990). The activated sludge is formed by a connected structure of biological micro-flocs, which are composed primarily of three-dimensional, gel-like, highly hydrated extracellular polymeric substances (EPS) (Sheng et al., 2010). The EPS could be divided into slime and bound EPS (Yu et al., 2009). Bound EPS is associated with flocs closely while slime is weakly bound to cells and has no direct contact with the cell. Bound EPS exhibits a double-layered EPS structure, composed of loosely and tightly bound EPS (LB- and TB-EPS) (Wingender et al., 1999; Li and Yang, 2007). The LB-EPS may function as the primary surface for cell attachment and flocculation (Li and Yang, 2007), while the TB-EPS has a specific shape and is bound tightly and stably to the cell surface (Wingender et al., 1999). The EPS is believed to mainly determine the structural and functional integrity of flocs (Park and Novak, 2007; Kong et al., 2015; Niu et al., 2016; Bao et al., 2016) and plays an important role in sludge dewatering (Yu et al., 2008).

Disintegration is a common pretreatment method for sludge, which can destroy the sludge floc structure and releases the cell and EPS contents into the liquid phase (Niu et al., 2016). To the best of our knowledge, ultrasound application has been extensively used for disintegrating sludge (Luostarinen et al., 2011; Park et al., 2013; Liu et al., 2015; Ren et al., 2015) and thereby improving dewaterability (Dewil et al., 2006; Khanal et al., 2007; Shao et al., 2010). Neyens et al. (2004) addressed that the sludge disintegration was due to the disruption of the sludge EPS during the ultrasonic pretreatment. The effects of ultrasound on physical-chemical characteristics (e.g. settleability, floc size, nitrate nitrogen content) of activated sludge (Feng et al., 2009) have also been reported. It is of great interest to see the rupture models and variations in the internal structure of activated sludge with stratified EPS extraction during ultrasonic treatment, so as to find the significance of EPS on the floc structure.

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Aside from the micro-structure characterization of activated sludge during ultrasonic treatment, rheological studies can also provide complementary information on the internal structure in a macro-level and dewaterability of sludge (Yen et al., 2002; Yan et al., 2013; Chen and Wang, 2015). Activated sludge suspensions are described as non-Newtonian fluids, whose flow properties can be measured using rheology (Moeller and Torres, 1997). Influence of EPS contents on the rheology of sludge flocs (Viñarta et al., 2006; Mori et al., 2008) has been performed. Some network properties obtained from some rheological tests at a certain TSS content (Yuan and Wang, 2013) and gel-like properties obtained from the strain amplitude sweep test (Yuan et al., 2014) of activated sludge before/after EPS extraction has been performed as well. Yet, the variation of the cohesion energy or the rigidity of the sludge work with stratified EPS extraction at various TSS concentrations has not been discussed.

In addition to the influence of the network structure properties on the activated sludge dewatering process (Jorand et al., 1995; Liao et al., 2002; Vahedi and Gorczyca, 2011), large water content of the activated sludge hinders the activated sludge dewatering process (Li and Ganczarczyk, 1990). The representation of the moisture distribution of the activated sludge has always been considered to be essential for the examination of dewatering problems (Vaxelaire and Cézac, 2004; Sato et al., 1982; Katsiris and Kouzeli-Katsiri, 1987; Lee and Lee, 1995). The simplest way of classification is to divide the water into two categories: "free water" and "bound water" (Tsang and Vesilind, 1990; Vaxelaire and Cézac, 2004). Particularly, the bound water content (BWC) is the key portion that dramatically influences the sludge dewatering (Mikkelsen and Keiding, 2002). Some researchers found that the reduction in the BWC could result in the improvement of the dewaterability of activated sludge (Sato et al., 1982; Katsiris and Kouzeli-Katsiri, 1987). Thus locating the bound water and studying the moisture distribution in the activated sludge structure has important significances for sludge dewatering performance.

Therefore, the main objective was to explore the rupture models, the variations in the floc structure during the ultrasonic pretreatment, the internal structure of activated sludge before/after EPS extraction at various TSS contents and the specific location of the bound water. Additionally, conceptual sludge models were also proposed to elucidate results of rheological testing and the moisture distribution in the activated sludge structure. These results obtained will provide further information on the effects of EPS on the internal structure and dewaterability of activated sludge.

2. Materials and methods

2.1. Characteristics of activated sludge

Activated sludge samples were collected from a municipal wastewater treatment plant in Beijing, China. Sludge samples were transferred to the laboratory within 2 h and immediately passed through a 1.2 mm sieve. Filtered samples were subsequently stored at 4 °C. Table 1 lists the main characteristics of activated sludge. The total suspended solids (TSS g l^{-1}) and volatile suspended solids (VSS g l^{-1}) were assessed from the weight loss of suspended sludge samples dried at specific temperatures and durations according to the standard method (APHA, 2005). All tests were carried out within a week and in triplicate using chemicals of analytical grade.

2.2. Stratification of EPS

The different EPS layers: the slime, LB- and TB-EPS of activated sludge was extracted by centrifugation and ultrasound method (Yu et al., 2008). Slime was obtained from the supernatant after low-speed centrifugation. LB- and TB-EPS were dissolved in a buffer solution (pH 7) containing Na_3PO_4, NaH_2PO_4, NaCl, and KCl at a molar ratio of 2:4:9:1. Conductivities of buffers were adjusted with distilled water to match those of the filtrated sludge samples presented in Table 1. In brief, the filtered sludge samples were initially centrifuged at 2000*g* for 15 min. The supernatant was collected as the slime. Subsequently, the settled sludge samples were re-suspended to their original volume with the aforementioned buffer solution (activated sludge after slime extraction). Next, the suspensions were subjected to centrifugation at 5000*g* for another 15 min with the bulk solution and solid phase obtained separately. The organic matter in the supernatant comprised the LB-EPS. The collected sediments were re-suspended again with the aforementioned buffer solution to the predetermined volumes (activated sludge after LB-EPS extraction) and then exposed to ultrasound at 20 kHz and 480 W for 10 min. The extracted solutions were centrifuged at 20,000*g* for 20 min. The organic matter in the bulk solution was the TB-EPS, while the residues re-suspended again with the aforementioned buffer solution to the original volumes were the pellet (activated sludge after TB-EPS extraction).

2.3. Ultrasonic pretreatment

100 ml sludge suspensions were sonicated with an ultrasound generator equipped with a probe transducer (CPX130, Vernon Hills, illinois USA), using 20 kHz (frequency) and 37 W (power output). Sludge samples were placed in a round-bottomed tube with the probe placed at the middle of the sample, which was at a level of about 2 cm above the tube bottom. The tube containing the sample was kept in crushed ice for 15 min before and throughout treatment. During the ultrasonic pretreatment, sludge samples were withdrawn at different times ranging from 30 to 600 s for the analysis of particle size and mass fractal dimension (D_f), which was determined using a Mastersizer 2000 instrument (Malvern, UK. Wu et al., 2002). The tests were performed in triplicate.

2.4. Rheological testing

Rheological measurements of activated sludge before/after EPS extraction under 5 TSS concentrations were conducted by a rheometer (Physica MCR 300, Anton Paar, Austria) in conjunction with US 200 software. The temperature was kept constant at 25 °C by a Peltier control. A PP 50 plate and plate sensor with the diameter of 49.94 mm and with a gap of 2.0 mm were employed. To obtain different TSS or VSS concentrations from sludge samples, raw and sludge samples resuspended after EPS extraction were first centrifuged at 1000*g* for 10 min, after which the bulk solution and solid phase obtained separately. The collected sediments were subsequently diluted with the supernatant. The VSS/TSS ratios of around 75% were observed for all sludge samples. Two rheological test modes including the controlled shear rate (CSR) test (Yuan and

Table 1
Characteristics of activated sludge.

pH	Conductivity (mS cm^{-1})	TSS (g l^{-1})	VSS (g l^{-1})	VSS/TSS (%)	COD (mg l^{-1})	SCOD (g l^{-1})	Zeta potential (mV)	Particle size (μm)
6.73 ± 0.04	1.46 ± 0.01	8.86 ± 0.30	6.13 ± 0.21	69.15 ± 0.03	6360.0 ± 329.5	68.6 ± 5.8	−16.9 ± 2.3	88.31 ± 1.08