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Physicochemical properties of sewage sludge disintegrated with high pressure homogenization

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ABSTRACT

High pressure homogenization (HPH) pretreatment can improve the sludge anaerobic digestion; however, the physical and chemical properties of homogenized sludge are completely unclear. The effect of HPH treatment on the physicochemical properties of homogenized sludge was studied. The disintegration degree and soluble chemical oxygen demand increased with the increment of homogeneous pressure and cycle, showing effective organic release from sludge solid to liquid after HPH treatment. Besides the organic, volatile fatty acid and NH_4^+-N were also released. The HPH treatment significantly disrupted sludge flocs, and the median sludge size starkly decreased from 48.26 to a range of 9.375–15.54 μm. The increase of homogenization pressure and cycle led to an insignificant change of sludge particle size, but the distribution of sludge particle size obviously changed and the volume of small particles obviously increased. Zeta potential of sludge liquid obviously decreased and apparent viscosity significantly increased after HPH treatment; however, the homogenization pressure and cycle displayed insignificant influence on them. Surface tension of sludge liquid decreased with the increase of homogenization pressure and cycle. The change of sludge physiochemical properties may be mainly attributed to the extracellular polymeric substances release from sludge flocs. The results may provide deeper insights why the sludge anaerobic digestion can be effectively improved by HPH pretreatment.

Introduction

At the beginning of 20th century, activated sludge method began to be applied in wastewater treatment, and now it has become one of the most widely used techniques around the world. The activated sludge processes produce a large amount of excess sludge, which may cause a serious of environmental and health issues. Cost of sludge treatment and disposal accounts for about half or even up to 60% of the total cost in wastewater treatment plants (WWTPs) (Luo et al., 2013). To minimize and utilize the excess sludge is becoming the most important and challenging problems in the field of wastewater treatment (Zhang et al., 2009). Various pretreatment techniques have been studied in laboratory, pilot and also in full-scale for sludge reduction, which mainly include physical, chemical and mechanical methods (Tiehm et al., 1997; Rai and Rao, 2009; Kim et al., 2010; Braguglia et al., 2012).

As a well-known mechanical method for cell disruption, high pressure homogenization (HPH) treatment was mainly used for the food stabilization and dairy emulsions (Paquin, 1999). Recently, several studies have been reported that HPH was applied to sludge disintegration for improving anaerobic sludge digestion (Onyeche et al., 2003; Rai and Rao, 2009; Fang et al., 2014). When the sludge is pumped through homogenization valve, the pressure increases and then is immediately released as the sludge passes through (Floury et al., 2004). Several different stresses, such as large pressure drop, highly focused turbulent eddies and strong shearing forces, work for sludge disintegration. In addition, pressure energy can be converted into heat and further be transmitted to the fluid, raising the fluid temperature (Thiebaud et al., 2003). The effectiveness of HPH is affected by several factors, such as homogenization pressure, number of homogenization cycles, and total solid content (TS), which has been investigated extensively. The increase of homogenization pressure or homogenization cycle significantly

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improved the sludge disintegration, while the increase of TS lowered the sludge disintegration efficiency (Zhang et al., 2012b). HPH treatment as a disintegration method has several advantages, for example, no obviously chemical changes, high disruption efficiency and simple operation (Barjenbruch and Kopplow, 2003).

Most researchers focused on the organic conversion between sludge solid and liquid phases with a HPH pretreatment. Camacho et al. (2002) reported that the applied shear forces by HPH led to a progressive cell break up and 90% total chemical oxygen demand (TCOD) could be maximally released from sludge particulate fraction. Zhang et al. (2013) showed that both total suspended solid (TSS) and volatile suspended solid (VSS) decreased with increasing the homogenization pressure and cycle. However, there are few literatures on the change of sludge physical and chemical properties during homogeneous process, which may influence the further sludge treatment and utilization. The objective of this study is to investigate the effect of HPH on physicochemical properties of disintegrated sludge. Besides the organic release, the release of volatile fatty acid (VFA) and NH_4^+-N after HPH treatment was analyzed. The effect of HPH treatment on floc structure, particle distribution and pH of sludge, and Zeta potential, apparent viscosity, surface tension of sludge liquid was investigated. The results are expected to provide deeper insights why the sludge anaerobic digestion can be effectively improved by HPH pretreatment.

Materials and methods

Sludge characterization

Sludge used in this study was collected from the secondary sedimentation tank of a municipal WWTP with an anaerobic-anoxic-oxic process in Beijing, and was stored at 4 °C for later use. The sludge had an average pH value of 7.1, an average TCOD of 8425 mg/L, an average soluble chemical oxygen demand (SCOD) of 192 mg/L and an average TSS of 6540 mg/L.

High pressure homogenization treatment

A high pressure homogenizer (JJ-30, Shengtong Inc., China) with a working pressure range of 0–100 MPa was used in this study for sludge disintegration. The sludge sample was firstly pumped through the homogenization valve by an air driven positive displacement pump, and the homogenization pressure was fixed at a certain value through adjusting the homogenization valve. When the sludge sample went through a convergent section of the homogenization valve, the sludge stream was immediately accelerated to 1000–1500 m/s. Then the high-velocity sludge stream impinged on an impact ring and the pressure suddenly dropped from high pressure to low pressure, and the sludge was disrupted and liquefied due to the combined action of pressure gradient, cavitation, turbulence, impingement, shear stresses, and extensional shear. Finally, the sludge was forced out of the homogenization valve with a lower velocity (Zhang et al., 2012b). The homogenization treatment was carried out at a given pressure (0–60 MPa) for one to three cycles. After HPH treatment, physicochemical properties of the homogenized sludge were measured.

Analytical methods

The sludge pH was obtained by pH meter (PHS–3C, Leici Co., China). The sludge particle size analysis was carried out through Malvern Mastersizer 2000 (Malvern, UK). The sludge TSS were measured based on APHA standard method (Eaton et al., 2005).

The sludge samples were centrifuged at 5000 r/min for 20 min, then the supernatant was immediately filtered through a cellulose membrane with a pore size of 0.45 μm, and the filtrate was used for analyzing SCOD, NH_4^+-N, VFA, Zeta potential, apparent viscosity and surface tension. The chemical oxygen demand (COD) of filtrate was measured by potassium dichromate method according to APHA standard method (Eaton et al., 2005), and was defined as SCOD. The NH_4^+-N of filtrate was measured by Nessler's reagent spectrometry by Multiparameter Bench photometer (LH5B-3(B), Lianhua Instruments Inc., China). The VFA of filtrate was determined by a gas chromatograph 3710 (Agilent, USA) equipped with a flame ionization detector and analytical column CPWAX52CB (30 m × 0.32 mm × 0.25 mm). The Zeta potential of filtrate was filled in capillary cell, and monitored by a Malvern Zetasizer Nano (Malvern, UK) with the refractive index value of 1.450 and the absorption of 0.001. The apparent viscosity was measured by a rotating torque cylinder at a temperature of about 20 °C, which was performed at a shear rate of 60/s to keep the sludge sample in suspension for 5 min. The surface tension was measured by a SITA pro line t15 tensiometer (SITA, Germany) with the bubble lifetime of 250 ms, and the value was the average of three measurements. The efficiency of sludge disintegration was evaluated by disintegration degree (DD_{COD}), which was calculated as Eq. (1) (Bougrier et al., 2005):

$$DD_{COD}(\%) = \frac{SCOD - SCOD_0}{TCOD - SCOD_0} \times 100\% \quad (1)$$

where $SCOD_0$ is the SCOD of sewage sludge before treatment.

The sludge flocs were photoed with a microphotograph (OLYMPUS, BX53) and analyzed by Image Pro Plus 6.1. Percentage of floc area (A_f) in photo was calculated as Eq. (2) (Costa et al., 2013).

$$A_f = \frac{100\sum_{i=1}^{n_{abi}} A_i}{TA}\% \quad (2)$$

where $\sum_{i=1}^{n_{abi}} A_i$ is the total area of all sludge flocs, and TA is the glass area.

The experiments were carried out with three replicates. T-test was used to evaluate significance analysis for experimental data.

Results and discussion

Organic matter release

The sludge can be disintegrated by homogenizer. The variations of SCOD and sludge DD_{COD} under different homogenization pressures and cycles are shown in Fig. 1. The SCOD increased with the increase of homogenization pressure from 0 to 60 MPa with a single homogenization cycle. When the homogenization pressure was 60 MPa with one homogenization cycle, the DD_{COD} reached 26.55%. The results demonstrated that more sludge organic matters were released from solid phase into liquid phase at higher homogenization pressure. In addition, multiple-cycle process was more effective for sludge disintegration than the single homogenization cycle. As homogenization cycle increases from one to three at a homogenization pressure of 60 MPa, the SCOD and DD_{COD} reached 3310 mg/L and 37.87%, which was 39.19% and 11.32% higher than that of the single homogenization, respectively. That may be attributed to the release of extracellular polymeric substances (EPS) due to floc disintegration or even the release of cell components because of cell lysis (Zhang et al., 2007). Other authors reported similar results that soluble organic release increased with increasing the homogenization pressure and cycle (Nah et al., 2000; Camacho et al., 2002). The organic release will improve further sludge anaerobic digestion.

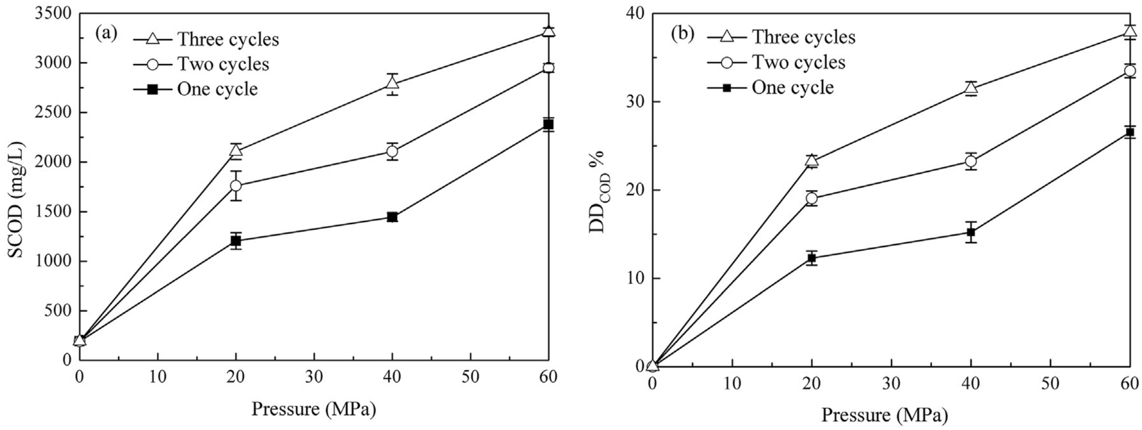

Fig. 1. Effect of HPH treatment on sludge disintegration: (a) SCOD and (b) DD_{COD}.

The homogenization pressure and homogenization cycle led to an obvious change of disintegrated sludge, as shown in Table 1, the VSS significantly decreased with the increase of homogenization pressure and cycles, showing that the organic were released from the sludge solids to liquid phase. The VSS decreased by about 59% after a homogenization treatment at a pressure of 60 MPa with three cycles, compared with raw sludge. VFA in disintegrated sludge significantly increased after homogenization treatment, and increased by approximately 593.1% at a homogenization pressure of 60 MPa with three cycles. The increased VFA could serve as the substrate and benefit to further sludge anaerobic digestion (Zhang et al., 2012a). Compared with raw sludge, the NH_4^+-N concentration in disintegrated sludge also obviously increased, which maybe resulted from mineralization of organic nitrogen. The sludge pH value showed a slightly reduction, which may be related with release of VFA.

Disruption of sludge flocs and cells

The microphotographs of floc structure for raw sludge and homogenized sludge at a homogenization pressure of 60 MPa are shown in Fig. 2. The HPH significantly changed the floc structure. The raw sludge displayed relatively tight flocs and microorganisms coated with EPS, while the homogenized sludge flocs became looser and their size decreased sharply, even the sludge flocs were thoroughly broken. The percentage of floc area increased from 69.49% to 82.43% after sludge homogenization.

The sludge particle size distribution is shown in Fig. 3. The sludge flocs consist of primary particles (~2.5 μm), microflocs (~13 μm) and porous flocs (~100 μm) (Jorand et al., 1995). The particle size distribution demonstrated that after sludge homogenization not only the porous flocs were destroyed, but also most of the microflocs were simultaneously destroyed, which led to an increase of the volume of primary particles. The median size ($d_{0.5}$) of raw sludge was about 48.26 μm and reduced to 15.54, 13.70 and 11.80 μm after homogenization treatment at 20, 40 and 60 MPa with one homogenization cycle, respectively. The reduction of particle size was starker than that with other treatment. For instance, Chu et al. (2001) reported an average particle size decrease from 99 μm to 22 μm after sonication treatment for 20 min at a sonication density of 0.33 W/ml and frequency of 20 kHz. A slight particle size $d_{0.5}$ decrease from 87.4 μm to 74.1 μm was reported with 10% amylase treatment for 28 h at 37 °C (Yu et al., 2013), and little particle size $d_{0.5}$ change from 55.2 to 52.5 μm was obtained after the sludge oxidation with $KMnO_4$ for 60 min (Wu et al., 2014). These results demonstrated the HPH disrupted the sludge flocs more thoroughly and the mechanisms of different sludge methods are different. When the homogenization cycle increased at a pressure of 60 MPa, the $d_{0.5}$ of homogenized sludge had insignificant change. The HPH pretreatment improved the sludge surface area, which may be more available for enzymatic reaction to improve the anaerobic digestion performances. However, the organic release is remarkable with an increase of homogenization pressure from 20 to 60 MPa with one cycle and with an increase of homogenization cycle from one to three at a pressure of 60 MPa (as shown in Fig. 1), which is different from the slight particle size reduction. Those organic matters might mainly come from sludge EPS solubilization, and the HPH treatment might not cause significant cell lysis.

Table 1
Changes of sludge properties before and after HPH pretreatment.

HPH		VSS (mg/L)	VFA (mg/L)	NH_4^+-N (mg/L)	pH
Pressure (MPa)	Cycle				
None	None	4675	72	19	7.31
20	1	4098	222	47	7.22
30	1	3780	276	50	7.14
40	1	3551	324	55	7.04
60	1	2811	343	63	6.95
60	2	2244	408	71	6.85
60	3	2032	499	74	6.78

Other physicochemical properties of homogenized sludge

Zeta potential of homogenized sludge liquid

Fig. 4 displays the effect of HPH treatment on Zeta potential of sludge liquid. The raw sludge liquid was negatively charged, and the corresponding Zeta potential was −6.95 mV, which was consistent with other reports (Liao et al., 2001). When the homogenization pressure was 20 MPa, the Zeta potential decreased to −12.93 mV, which enhanced the sludge stability. On the other hand, the Zeta potential only slightly changed from −12.95 to −13.90 mV with increasing the homogenization pressure and cycle. After sludge HPH, EPS, especially loosely bound EPS (LB-EPS) were solubilized, which increased negative charge in supernatant and changed Zeta potential. Wilén et al. (2003) found that the total EPS content and individual EPS component both enhanced the negative charge of sludge, and the effects of proteins and humic substances were significant. These results agreed with the release of organic matters (Fig. 1). However, Chu et al. (2001) observed that ultrasound had no obvious effect on the surface charges of sludge particles.

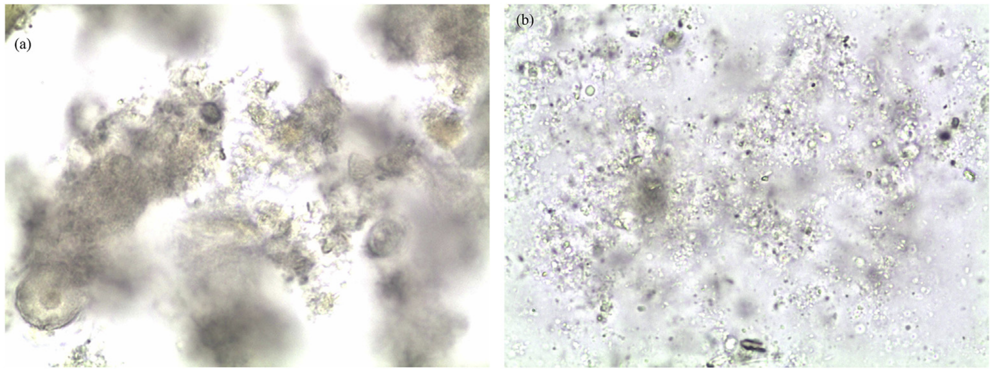

Fig. 2. Microscopes of sludge flocs of (a) raw sludge (400×) and (b) sludge treated at a homogenization pressure of 60 MPa with one cycle (400×).

Apparent viscosity of homogenized sludge liquid

As shown in Fig. 5, the apparent viscosity of sludge liquid was influenced by homogenization treatment. The apparent viscosity of raw sludge liquid was 1.16×10^{-3} Pa s. After the HPH treatment at a pressure homogenization of 20 MPa with one cycle, the apparent viscosity remarkably increased to about 1.47×10^{-3} Pa s, but there was no obvious difference between different homogenization pressures and cycles. Nagaoka et al. (1996) reported that higher EPS production caused an increase in the viscosity of mixed liquor in submerged membrane bioreactor. Moreover, Li and Yang (2007) reported that there was a positive correlation between the sludge viscosity and its LB-EPS content. The increased apparent viscosity can represent the EPS solubilization from sludge flocs.

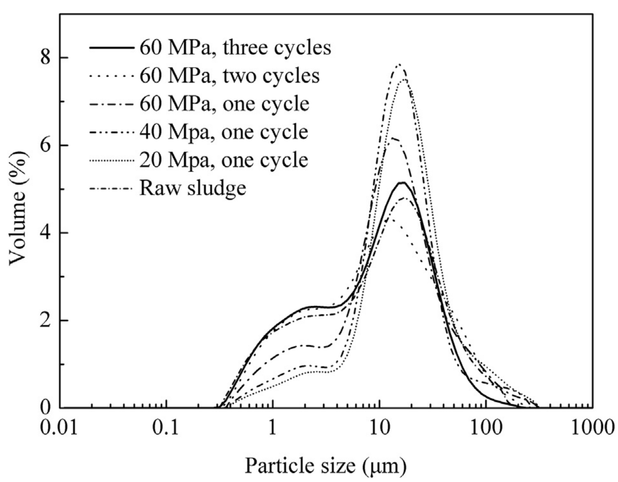

Fig. 3. Particle size distribution for raw sludge and sludge disintegrated with HPH.

Surface tension of homogenized sludge liquid

The surface tension change of sludge liquid with different homogenization pressures and cycles is shown in Fig. 6. The surface tension gradually decreased with the increase of homogenization pressure and cycle. After a homogenization treatment at a pressure of 60 MPa, the surface tension reduced from 70.91 to 60.67 mN/m with a reduction of 14.44%. When further increasing homogenization cycles from one to three cycles at a homogenization pressure of 60 MPa, the surface tension decrease reached 24.30%. The major components of EPS are usually carbohydrates, proteins and humic substances, which are naturally amphipathic molecule and could change the surface tension of solution (Sheng et al., 2010). After sludge HPH treatment, organic substances, such as carbohydrates and proteins, were released into liquid phase (Zhang et al., 2013), which led to the reduction of surface tension. These results were in agreement with the conclusion that the sludge SCOD concentration increased with the increase of homogenization pressure and cycle.

Conclusion

The effect of HPH treatment on physicochemical properties of sludge was elucidated. The sludge disintegration was improved

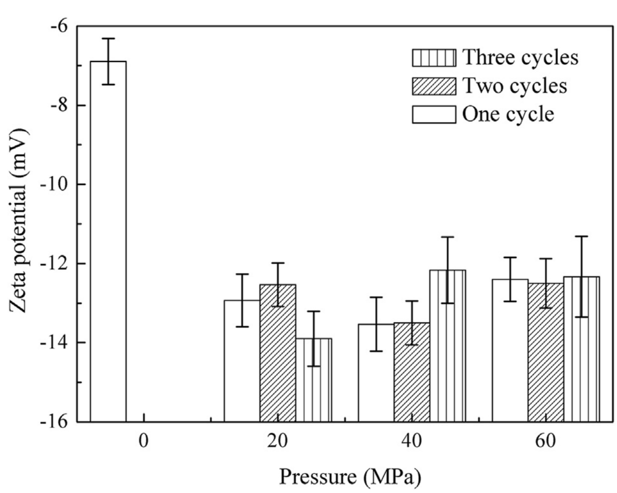

Fig. 4. Effect of HPH treatment on Zeta potentials of sludge liquid.

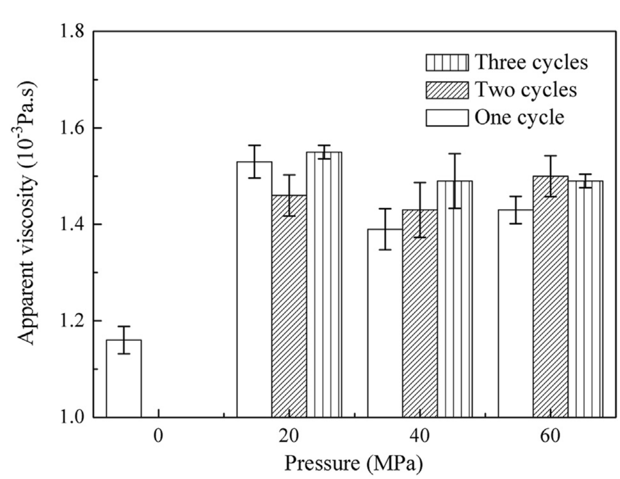

Fig. 5. Effect of HPH on apparent viscosity of sludge liquid.

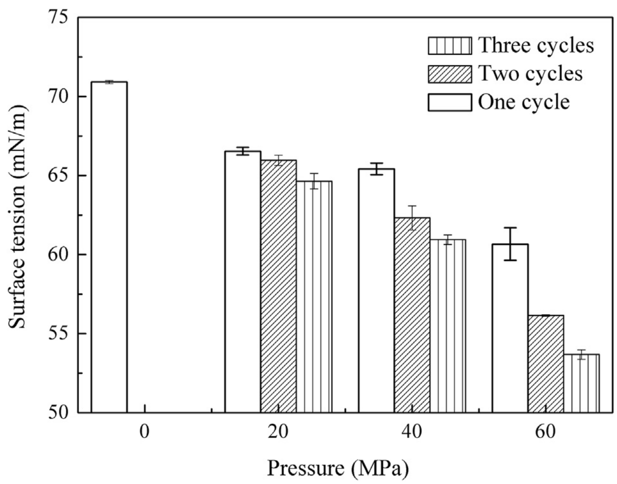

Fig. 6. Effect of HPH on surface tension of sludge liquid.

with the increment of homogeneous pressure and cycle. The SCOD and DD_{COD} reached 3310 mg/L and 37.87% at a homogenization pressure of 60 MPa with three cycles. Due to the sludge disintegration by HPH pretreatment, the median sludge size starkly decreased, but the homogenization pressure and cycle had no significant influence on sludge particle size. However, the distribution of sludge particle size obviously changed and the volume of small particles significantly increased. Besides, the HPH treatment significantly influenced other physicochemical properties of homogenized sludge. At a homogenization pressure of 60 MPa with one cycle, apparent viscosity of sludge liquid increased by 23.28%, and Zeta potential and surface tension decreased by 79.84% and 14.44%, respectively. However, the increase of homogenization pressure and cycle resulted in insignificant change of Zeta potential and apparent viscosity of sludge liquid.

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