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Rheological properties of sewage sludge during enhanced anaerobic digestion with microwave-H_2O_2 pretreatment

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A B S T R A C T

The rheological behavior of sludge is of serious concern in anaerobic digestion. This study investigated the rheological properties of sewage sludge during enhanced anaerobic digestion with microwave-H_2O_2 pretreatment (MW-H_2O_2). The results showed that MW-H_2O_2 pretreatment resulted in the improvement of sludge flowability and weakening of its viscoelastic properties. Further positive effects on the rheological properties of digested sludge during anaerobic digestion were observed. The flowability was improved with a low level of apparent viscosity. The decrease of the consistency index and increase of the flow behavior index indicated that the strength of the inner structures and non-Newtonian flow characteristics of digested sludge weakened. Both the storage modulus (G') and loss modulus (G'') decreased, indicating that the viscoelastic behavior became weak. These effects were possibly attributed to the changes of the digested sludge micro-structures, such as extracellular polymeric substances (EPS). This study concluded that anaerobic digestion for treating sewage sludge combined with pretreatment is a more favorable option than single anaerobic digestion from the perspective of rheology.

1. Introduction

Large amounts of sewage sludge are annually produced from the biological wastewater treatment process. In China, approximately 6.25 million dry tons of sewage sludge were produced in 2013 (Yang et al., 2015). In the U.S., the amount of biosolids annually generated from stabilized sewage sludge is greater than 8 million dry tons (Peccia and Westerhoff, 2015). Due to the characteristics of sewage sludge, including the high degree of water bounding (Coelho et al., 2011), low biodegradability (Kuglarz et al., 2013) and existence of chemical pollutants, such as heavy metals, pathogens and persistent organic pollutants (Hua et al., 2008), sludge treatment and disposal present rising challenges for wastewater treatment plants (WWTPs). It is well known that sewage sludge is a non-Newtonian fluid, that exhibits rheological characteristics, such as shear-thinning, thixotropy, yielding properties and viscoelasticity (Campbell and Crescuolo, 1982; Chaari et al., 2003; Dick and Ewing, 1967; Eshtiaghi et al., 2013; Seyssiecq et al., 2003). The rheological properties of sludge strongly affect sludge treatment and disposal processes, especially hydrodynamic processes, such as mixing, pumping, dewatering, drying and landfilling (Baudez and Coussot, 2001; Chaari et al., 2003; Seyssiecq et al., 2003; Spinosa and Lotito, 2003; Xia et al., 2009). The rheological properties of sludge are not only important design parameters for these processes (Brehmer et al., 2012; Örmeci, 2008; Troesch et al., 2009), but also serve as control parameters for the optimization of some treatments, such as sludge conditioning and dewatering (Abu-Orf and Dentel, 1999; Dursun et al., 2004; Marinetti et al., 2010; Ormeci, 2007, 2008; Wang and Dentel, 2010). Therefore, the rheological parameters are important in sludge management and treatment processes (Ratkovich et al., 2013).

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Among the sludge treatment processes, anaerobic digestion (AD) is a promising technologies to treat sewage sludge due to the capacity of energy recovery, mass reduction and pathogen removal (Cao and Pawłowski, 2012). In addition, the biological degradation of organic matter in sludge is an important stabilizing process for land application (Chen et al., 2012). Due to the low biodegradability of activated sludge, anaerobic digestion is always limited with a long sludge retention time (SRT) and low methane production efficiency. To improve the biodegradability of activated sludge, pretreatment technologies such as thermal hydrolysis (Pilli et al., 2014), microwave (Liu et al., 2015b), ultrasonic (Houtmeyers et al., 2014), chemical (Hai et al., 2014) and hybrid pretreatment (Tyagi et al., 2014) were used. As an alternative thermal treatment, microwave irradiation has proved to be effective in enhancing sludge anaerobic digestion (Appels et al., 2013; Chi et al., 2011; Jang and Ahn, 2013; Toreci et al., 2011; Zheng et al., 2009). Moreover, hybrid pretreatment, with chemicals such as H_2O_2 can effectively enhance sludge AD under more acceptable operation conditions (Liu et al., 2015a), including ambient pressure and a low heating temperature ($\leq$100 °C). For the consideration of energy consumption, the scale-up of microwave pretreatment for enhancing sludge AD is limited. However, Pilli et al. (2014) pointed out that energetically self-sustainable or even surplus energy produced in the enhanced AD process with thermal pretreatment could be achieved for treating concentrated sludge with solid concentrations higher than 3%.

For AD, the rheological properties of sludge play key roles in processes such as mixing, pumping and recirculating. Eftekharzadeh et al. (2007) used Computational Fluid Dynamics (CFD) to evaluate anaerobic digester mixing based on site-specific sludge rheology data. The results showed that doubling the solid concentration from 2% to 4% within the digester reduced the active volume more than half, and thus, larger mixing pumps are essential to maintain effective mixing. Considering the mixing, pumping efficiency and power consumption, the digester design is directly related to the rheology of feeding sludge and digested sludge. However, as described by Baudez et al. (2011), most of the investigations focused on the rheology of activated sludge, and the digested sludge rheology was only recently investigated. Due to the viscosity, yield stress, fluid consistency increases with the increase of the solid concentrations (Markis et al., 2014), sludge rheology is particularly important in the AD of concentrated sludge. According to Hidaka et al. (2013), although the AD of sewage sludge with Total Solid (TS) concentrations higher than 10% could successfully operate under mesophilic conditions, the high viscosity would be of serious concern in full-scale operation. According to previous studies, the rheology of sewage sludge depends on the sludge origin, temperature (Feng et al., 2014a) and solid concentrations (Markis et al., 2014). In addition, the application of pretreatment technologies can modify the sludge rheology. Ruiz-Hernando et al. (2010) used ultrasonic waves to pretreat secondary sludge. Both steady state viscosity and thixotropy significantly decreased with specific energy. Feng et al. (2014a) reported that the viscosity of municipal sludge was reduced and the thixotropy became weak due to thermal hydrolysis (T = 170 °C, 60 min) treatment and that the viscoelasticity of the treated sludge remarkably decreased, i.e., the storage modulus decreased by 92.5%. In addition, the sludge rheology, in the form of viscosity and shear stress, could be dramatically reduced through AD, which was mainly due to the decrease of the solid concentrations (Dai et al., 2014).

Although the rheological evolution of sewage sludge through AD or pretreatment has been investigated separately in previous studies, few studies have reported on the rheological evolution of sewage sludge throughout AD combined with pretreatment. To develop pretreatment technologies for enhancing sludge AD, it is necessary to determine whether pretreatment has further positive effects on the rheological properties of sludge in digesters or not. Therefore, the objectives of the present study were to investigate the rheological characteristics of sludge during enhanced AD with MW-H_2O_2 pretreatment, and evaluate the effects of MW-H_2O_2 pretreatment on the rheology and micro-structures of sludge in digesters.

2. Materials and methods

2.1. Sludge pretreatment

The dewatered sewage sludge was collected from the Qinghe WWTP in Beijing, China, with a treatment capacity of 550,000 m^3/d for the A^2/O process. The dewatered sludge was diluted with deionized water to reduce the total solid concentrations to approximately 8%. For AD, the inoculum was collected from mesophilic anaerobic digesters treating sewage sludge in the Xiaohongmen WWTP in Beijing, China.

For microwave pretreatment, a customized industrial microwave oven (Baoding Julong Microwave Energy Equipment Co., Ltd., China) was used that operated at 2450 MHz, 600 W and ambient pressure. The optimized MW-H_2O_2 pretreatment process proposed by Wang et al. (2009) and Xiao et al. (2012) was used in this study. The pH of raw sludge was first adjusted to 10.0 with a 5 mol/L NaOH solution, and then, the sludge was heated to 80 °C to inhibit catalase activity, which can occur in living aerobic cells, to avoid the decomposition of H_2O_2 by the catalase that exists in the sludge (Wang et al., 2009). Afterwards, H_2O_2 was added (A.R., 30%, w/w) at a dosage of 0.2 g/g TS, and the sludge was continuously heated to 100 °C, to realize an advanced oxidation process (Xiao et al., 2012). The pretreatment process was conducted in 1-L beakers with plastic caps, but without sealing. Three-hundred grams of sludge in the beaker was heated at a rate of 20 °C/min to reach the target temperature, without holding at 80 or 100 °C. During heating, the sludge was mixed with a lift mixer at a speed of 50 rpm. The characteristics of the raw and pretreated sludge are shown in Table 1.

2.2. Semi-continuous anaerobic digestion

Three reactors were established in 2-L glass bottles with 1.8 L of effective working volume and equipped with a motor and stirring paddle each. The stirring process was set at a rate of 112 rpm in an interval pattern (1 min stirring and 1 min breaking). One of the reactors was fed with raw sludge as the control. Another parallel one was fed with a mixture of MW-H_2O_2 pretreated sludge and raw sludge ($\text{Mass}_{\text{pretreated sludge}}/\text{Mass}_{\text{raw sludge}} = 1/1$), referred to MW-one stage. A two stage configuration reactor, referred to MW-two stage, was fed with a mixture of MW-H_2O_2 pretreated sludge and raw sludge and was used to evaluate the potential toxic effect of residual H_2O_2 in pretreated sludge on methanogens compared with the MW-one stage reactor. All of the reactors operated at 37 °C, a SRT of 20 d, and an organic loading rate (OLR) of approximately 2.92 g Volatile Solids (VS)/(L·d). For the MW-two stage reactor, the SRT of the first stage reactor was set at 2 d and the SRT of the second reactor was set at 18 d. All of the reactors operated semi-continuously by feeding and discharging sludge once per day. At the beginning, the reactors only contained the inoculum sludge with a TS of 2.58%. The activated sludge or pretreated sludge with TS of approximately 8% was fed into the reactors once per day. The produced biogas was firstly absorbed through a 3 mol/L NaOH solution for removing CO_2 and H_2S, and the remained biogas was considered to be methane and was automatically recorded by an AMPTS II instrument (Bioprocess Control Company, Sweden).

Table 1
Characteristics of raw sludge and pretreated sludge.

Parameters	TS (w/w %)	VS (w/w %)	VS/TS (w/w %)	pH	SCOD (mg/L)	Proteins (mg/L)	Polysaccharides (mg/L)
Inoculums	2.58 ± 0.03	1.44 ± 0.03	54.98 ± 0.53	7.34 ± 0.03	362 ± 11	60.93 ± 0.02	66.86 ± 0.13
Raw sludge	7.63 ± 0.01	5.78 ± 0.00	75.78 ± 0.07	6.84 ± 0.02	4224 ± 17	1171.29 ± 116.65	689.94 ± 73.53
Pretreated sludge	7.88 ± 0.01	5.68 ± 0.00	72.11 ± 0.00	7.34 ± 0.02	38520 ± 5221	11490.72 ± 1738.14	3308.08 ± 85.70

The digested sludge used for rheology analysis was collected from the discharge of anaerobic digestion reactors at the end of 2 SRTs, and then, the reactors were further operated for another SRT.

2.3. Rheological measurements

The sludge rheology was determined by a rotational rheometer (Haake RheoStress 6000, Thermo Fisher Scientific Inc., Karlsruhe, Germany), using a P 35 parallel plate geometry (35-mm diameter, 1 mm gap). The temperature was kept at 37 ± 0.1 °C with a water bath. The rheological properties of sludge under flow conditions was analyzed as follows: Firstly, sludge was pre-sheared at a shear rate of 200 s^{-1} for 5 min and then left at rest for 5 min. Secondly, the measurements were performed by linearly increasing the shear rate from 10 s^{-1} to 1000 s^{-1} over 3 min, holding at 1000 s^{-1} for 30 s (up curve). Finally, the shear rate was linearly decreased from 1000 s^{-1} to 10 s^{-1} over 3 min (down-curve). In addition, the Herschel-Bulkley model (Eq. (1)) was used to fit the rheological data, which are efficient in describing the sludge rheology, including pseudo-plastic or shear-thinning as well as the yield stress characteristics (Dai et al., 2014).

$$\tau = k\dot{\gamma}^n + \tau_y \tag{1}$$

where τ is the shear stress (Pa); $\dot{\gamma}$ is the shear rate (s^{-1}); τ_y is the yield stress (Pa); k is the consistency index ($Pa\cdot s^n$), which can reveal the average firmness of sample (Ruiz-Hernando et al., 2014); and n is the flow behavior index (dimensionless), which is equal to 1 for Newtonian fluids, higher than 1 for dilatant fluids and lower than 1 for pseudo-plastic fluids (Ruiz-Hernando et al., 2015). A decrease or increase of n from 1 indicates that the non-Newtonian flow characteristics are strengthened (Feng et al., 2014a).

The rheological properties of sludge at rest were analyzed by performing oscillatory measurements and creep-recovery tests. Oscillatory stress sweeps were conducted at a constant frequency (1 Hz) to determine the linear viscoelastic region (Augusto et al., 2013; Ruiz-Hernando et al., 2014; Yilmaz et al., 2012) as shown in Fig. S1 and Fig. S2, which are included in the supporting information. Frequency sweep tests were conducted by controlling the shear stress at 1 Pa. The creep-recovery tests were conducted at the constant shear stress of 1 Pa ($<\tau_y$). Firstly, the sludge was kept undisturbed for 5 min. Then, a constant shear stress of 1 Pa was exerted on sludge for 10 min. After the creep process, the applied stress was released. The recovery process was recorded for 10 min.

2.4. EPS extraction

The formaldehyde-NaOH extraction method was used to extract bound EPS (Guo et al., 2014). A sludge suspension (40 mL) was first dewatered by centrifugation in a 50-mL tube at 6500 rpm for 15 min. The supernatant was collected and replenished to a volume to 40 mL with deionized water, and considered as soluble EPS to analyze for protein and polysaccharide contents. The rest of sludge pellets were then re-suspended in 20 mL of deionized water. Afterwards, 0.12 mL of formaldehyde (36.5%, w/w) was added to the samples, which were further kept at 4 °C for 1 h. Then, 8 mL of a NaOH solution (1 mol/L) was added and kept at 4 °C for another 3 h. The liquor was centrifuged at 8000 rpm for 20 min. The supernatant was collected and replenished to a volume of 40 mL with deionized water, as bound EPS were analyzed in terms of the protein and polysaccharide contents.

2.5. Analysis methods

TS, VS, NH_3—N and COD were measured according to the standard methods procedure (APHA, 2005). Proteins were measured according to the modified Lowry method using bovine serum albumin as the protein standard (Frolund et al., 1995). Polysaccharides were measured by the Dubois method with D-glucose as the standard (Dubois et al., 1956). The molecule weight distribution was determined by using high performance size-exclusion chromatography (HPSEC), as described by Niu et al. (2013). Volatile fatty acids (VFAs) were determined by a gas chromatograph (Shimazu GC, 2014) equipped with a flame ionization detector and a DB-FFAP capillary column (30 m × 0.32 mm × 0.25 μm). The injector temperature was 220 °C, and the detector temperature was 250 °C. The program for the oven temperature was as follows: 70 °C (1 min), 20 °C/min to 180 °C (3 min), and 20 °C/min to 210 °C (3 min). Before the analyses of soluble chemical oxygen demand (SCOD), soluble proteins, polysaccharides, molecule weight distribution and VFAs, the sludge samples were centrifuged at 6000 rpm for 15 min. The supernatant was filtered through a 0.45 μm membrane filter. The methane content in biogas was analyzed by a gas chromatograph (Aglilent 4980, USA) equipped with a flame ionization detector as described by Zhong et al. (2013). SEM analysis was conducted as previously described with some modifications (Fang and Chui, 1993). Sludge bio-drying samples were firstly washed with 0.1 M phosphate buffer solution (PBS, pH 7.4), fixed in 0.1 M PBS with 2.5% glutaraldehyde for 4 h at 4 °C, then dehydrated in a series of water/ethanol solutions (20, 50, 70, 80, 90%, V/V) for 10–15 min, and then treated with a mixture of ethanol and isoamyl acetate (1:1, V/V) overnight at 4 °C. The microstructure was examined using a Hitachi SU-8020 field-emission scanning electron microscopy (Hitachi, Japan) after the samples were critical point dried with carbon dioxide and sputter-coated with gold under vacuum on a stub.

VS reduction was calculated using the Van Kleeck equation (Eq. (2)) as described by Jensen et al. (2014):

$$\text{VS reduction } (\%) = \frac{VS_{f,i} - VS_{f,e}}{VS_{f,i} - \left(VS_{f,i} \cdot VS_{f,e}\right)} \tag{2}$$

where $VS_{f,i}$ and $VS_{f,e}$ are fraction of volatile solids (VS/TS) in the influent and effluent total solids, respectively.

3. Results and discussion

3.1. Performance of the anaerobic digesters

After a start-up of 40 days, the AD reached a relatively steady state and operated for more than 3 SRTs. The average performances are shown in Fig. 1. Because the solid concentration of the feeding sludge was considerably higher than that of the inoculum sludge,

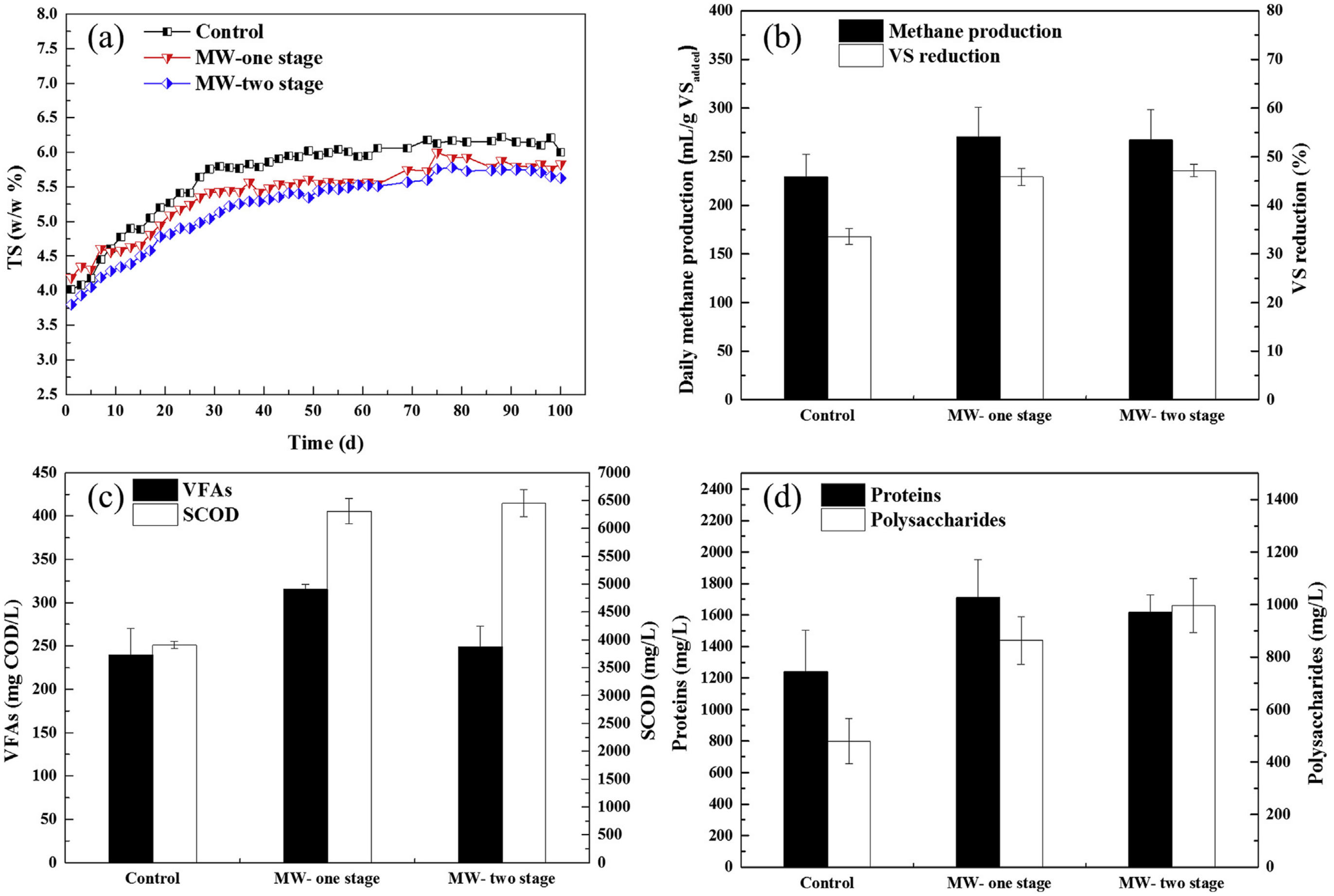

Fig. 1. Operation performance of anaerobic digestion reactors (Control: the digested sludge from reactor without pretreatment. MW-one stage: the digested sludge from the one stage reactor with pretreatment. MW-two stage: the digested sludge from the two stage reactor with pretreatment.).

the TS of digested sludge increased with time over the first 40 days. After the start-up phase, the reactors operated stably in the following period. The average TS concentrations in the reactors of the control, MW-one stage reactor and MW-two stage reactor (the second stage) were approximately 6.02 ± 0.14%, 5.66 ± 0.17% and 5.52 ± 0.18%, respectively. Compared with the feeding sludge, the solid concentrations of sludge decreased through AD. MW-H_2O_2 pretreatment led to an improvement of TS reduction compared with the control in both the one-stage and two-stage AD reactors. During stable operation, the daily methane production was increased by 18.13% due to MW-H_2O_2 pretreatment compared with the control (Fig. 1b). Although a faster stable state was achieved in the MW-two stage reactor (data not shown), the daily methane production was not further improved compared with the MW-one stage reactor. This was mostly due to biogas production in the first stage reactor even though the SRT was controlled at 2 days as proposed by Coelho et al. (2011), to avoid excessive methanization in the first stage reactor. It was found that increasingly more methane was produced daily in the first stage reactor (data not shown). In addition, the average methane concentration was approximately 46.14 ± 3.32% in the first stage reactor. The biogas produced in the first stage reactor and low methane concentration possibly resulted in the low methane conversion from biodegradable organics in sludge. In this study, the two-stage reactor did not show a better performance for enhancing sludge AD compared with the one-stage reactor.

During the period of stable operation, an obvious VS reduction during AD was achieved with MW-H_2O_2 pretreatment (Fig. 1b). The average VS reduction rates were approximately 33.60 ± 1.62%, 45.83 ± 1.72% and 47.16 ± 1.22% for the control, MW-one stage and MW-two stage AD reactors, respectively. With the MW-H_2O_2 pretreatment, the VS reduction rates increased by 36.51 ± 4.40% and 40.63 ± 7.03% for the two different AD configuration reactors. Both the one stage AD reactor and two stage AD reactor showed an enhanced performance of sludge mass reduction. Additionally, MW-H_2O_2 pretreatment influenced the properties of the activated sludge and digested sludge. The pretreatment mainly caused a breakup of sludge flocs and microbial cells. Large amounts of soluble organic matter, such as proteins and polysaccharides, were released (Table 1). The SCOD increased from 4224 ± 17 mg/L of raw sludge to 38520 ± 5221 mg/L after treatment. Although the released soluble organic matter could be converted to methane during AD, a relatively high level of residual SCOD, such as proteins and polysaccharides, still remained in the digested sludge, as shown in Fig. 1c, d. Moreover, MW-H_2O_2 pretreatment further increased the amount of SCOD in the digested sludge compared with the control. However, MW-H_2O_2 pretreatment did not result in an obvious variation of VFAs in the digested sludge. These effects of MW-H_2O_2 pretreatment on the properties of the activated sludge and digested sludge may contribute to the evolution of sludge rheology.

3.2. Effects of MW-H_2O_2 pretreatment on activated sludge rheology

The rheograms of raw sludge and pretreated sludge are presented in Fig. 2a. The shear stress increased non-linearly with the

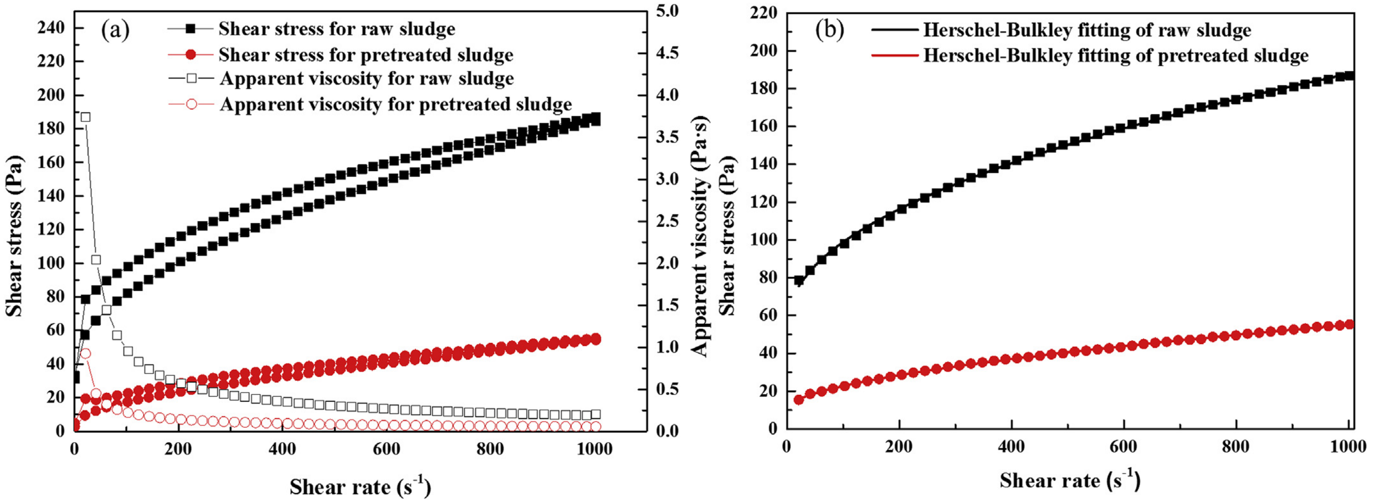

Fig. 2. Flow curves of raw sludge and pretreated sludge.

increase of the shear rate, which suggested that both the raw sludge and pretreated sludge presented non-Newtonian flow. At a low shear rate, the apparent viscosity decreased with the increasing shear rate, demonstrating a shear-thinning property. The rheological properties of the sludge within the non-linear viscoelastic region were influenced by the MW-H_2O_2 pretreatment. In particular, the apparent viscosity decreased obviously, indicating the improvement of sludge flowability. Moreover, both raw sludge and pretreated sludge presented thixotropic behavior for the formation of the hysteresis loop. Shear stresses of up-curves were higher than those of down-curves at the same shear rate. MW-H_2O_2 pretreatment resulted in the decrease of the hysteresis area from 11121.57 $Pa \cdot s^{-1}$ to 3567.94 $Pa \cdot s^{-1}$. The hysteresis area is useful to evaluate sludge thixotropy (Yen et al., 2002), the decrease of which indicated that the thixotropy of sludge weakened. The thixotropic behavior of the sludge suggested that the kinetic processes of both breakdown and build-up exist when flow is applied. The hysteresis area became small due to pretreatment, indicating that the kinetic processes could rapidly reach a steady state. In general, activated sludge shows high levels of thixotropy due to its internal structures (Jibao et al., 2015). The colloidal forces among particles tend to rebuild the structures (Eshtiaghi et al., 2013). Therefore, the decrease of sludge thixotropy with MW-H_2O_2 pretreatment is possibly the result of the weakness of colloidal forces among particles.

Furthermore, the Herschel-Bulkley model was used to fit the flow curve, and strong agreement ($R^2 > 0.999$) was obtained (Fig. 2b). According to the Herschel-Bulkley model, the yield stress (τ_y) was taken into account in the equation, which is regarded to be an indication of the specific value of stress exerted on the sludge when it begins to flow. With MW-H_2O_2 pretreatment, the yield stress of sludge obviously decreased (Table 2). The consistency index (k) and flow behavior index (n) in the Herschel-Bulkley model are two other important parameters. The value of k decreased from 4.90 to 0.81, and the value of n increased from 0.48 to 0.58 (Table 2). The consistency index (k) is a measure of average firmness. The decrease of k is consistent with the decrease of apparent viscosity. The flow behavior index (n) is related to the type of fluids. Pseudo-plastic fluids present values of n lower than 1. The increase of n revealed that the rheological properties of the pseudo-plastic fluid became weaker, suggesting that the internal structures of the sludge were broken. This is consistent with the release of soluble organic matter, such as proteins and polysaccharides, with treatment, which can be an indication of the degree of sludge flocs rupture.

The viscoelastic properties of the activated sludge and pretreated sludge in the linear viscoelastic region were analyzed according to frequency sweep tests (Fig. 3). The storage modulus (G') was larger than the loss modulus (G''). G' was 6.25 times and 2.61 times higher than G'' for activated sludge and pretreated sludge, respectively, indicating a solid-like regime. In the linear viscoelastic regime, both G' and G'' showed a weak power-law dependence across the low frequency (ω) range: $G', G'' \propto \omega^n$ (power-law index smaller than 0.2). The power-law index of G' and G'' were increased due to pretreatment. Both G' and G'' decreased obviously after pretreatment. Moreover, the ratio of G''/G', namely tan (δ), increased from 0.16 of activated sludge to 0.39 of pretreated sludge. Thus, the proportion of energy dissipation to energy storage increased. With pretreatment, the destruction and hydrolysis of sludge flocs, especially EPS, possibly resulted in the weakness of colloidal forces and network strength (Feng et al., 2014a), which led to the increase of tan (δ). Activated sludge presented $G' > G''$ in the linear viscoelastic region, exhibiting a gel-like structure (Wang et al., 2011). The higher the solid content, the stronger the

Table 2
Herschel-Bulkley parameters of different sludge.

Parameters	Raw sludge	Pretreated sludge	Control[a]	MW-one stage[b]	MW-two stage[c]
TS (w/w %)	7.63 ± 0.01	7.88 ± 0.01	5.09 ± 0.01	5.10 ± 0.01	5.15 ± 0.02
τ_y (Pa)	54.51	11.08	10.48	5.85	5.56
k ($Pa \cdot s^n$)	4.90	0.81	0.61	0.25	0.20
n	0.48	0.58	0.65	0.72	0.75

[a] Digested sludge with dilution from the control reactor.
[b] Digested sludge with dilution from the MW-one stage reactor.
[c] Digested sludge with dilution from the MW-two stage reactor.

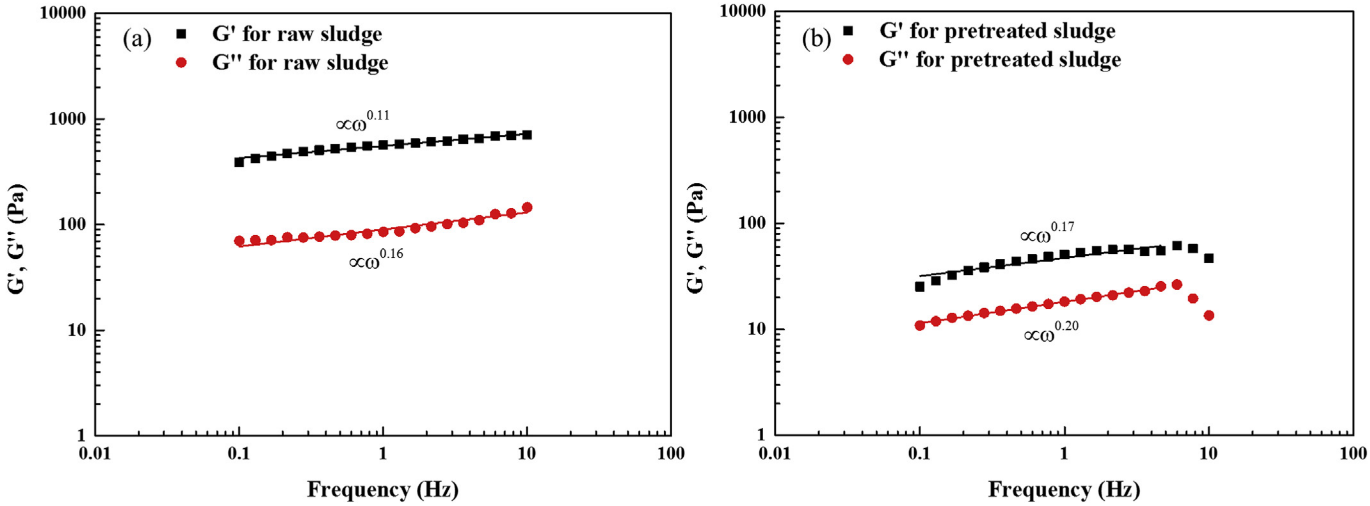

Fig. 3. Frequency sweep of raw sludge (a) and pretreated sludge (b).

colloidal forces and network strength (Feng et al., 2014a). Therefore, the gel-like structure of activated sludge, especially with a high solid content, may have a weak effect on the sludge mixing process with an intermittent operation pattern or a hydraulic process with a low shear rate. However, the pretreatment tends to change the sludge structure from gel-like to pasty-like or allow for an easy transition to a liquid-like material. Several studies (Feng et al., 2014a, 2014b) found that $G' < G''$ for treated sludge with thermal hydrolysis treatment, suggesting that the solid-like properties nearly vanished and the sludge exhibited liquid-like properties. Therefore, pretreatment of activated sludge tends to have a good influence on pumping or mixing processes for the improvement of sludge flowability in the non-linear viscoelastic region, and the weakness of sludge solid-like properties in the linear viscoelastic region.

3.3. Effects of pretreatment on digested sludge rheological properties

As mentioned above, MW-H_2O_2 pretreatment resulted in the increase of sludge flowability and weakness of the sludge solid-like properties, which were beneficial for sludge pumping or mixing processes. Furthermore, whether pretreatment has positive effects on the subsequent digested sludge rheological properties is extremely important. The rheograms of digested sludge in different reactors are presented in Fig. 4. The digested sludge still showed non-Newtonian fluids and shear-thinning properties. Notably, no thixotropic behavior was observed for the digested sludge (Fig. 4a), suggesting that colloidal forces among particles that tend to rebuild the structures disappeared. Anaerobic digestion tends to convert large sludge flocs into smaller ones (Mahmoud et al., 2006) and cause the disintegration of macromolecular organic compounds, such as proteins and polysaccharides into micro-molecular organics, such as VFAs, which can be further used by methanogens for biogas production. Moreover, the degree of proteins and polysaccharides dispersion could be improved after anaerobic digestion (Mikkelsen and Keiding, 2002), implying the strong impact of anaerobic digestion on the sludge structure. The apparent viscosity of digested sludge in reactors combined with pretreatment was lower than the control reactor, suggesting better flowability of the digested sludge with pretreatment. It is well known that the rheology of sludge, that is, apparent viscosity, was related to the sludge solid concentration (Markis et al., 2014). The low apparent viscosity was perhaps due to the low solid concentration of

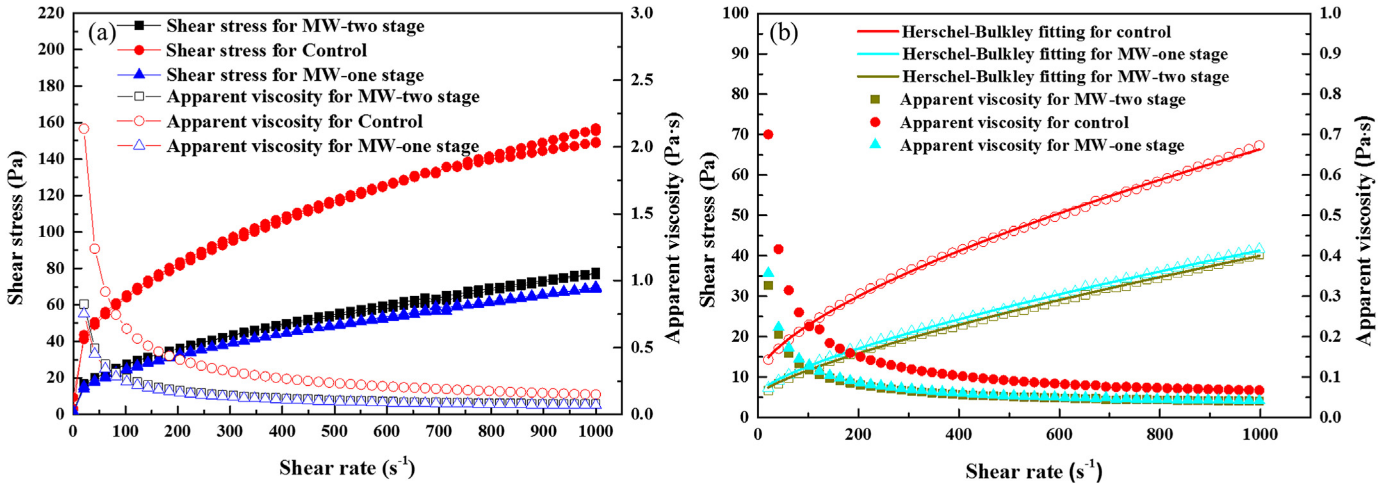

Fig. 4. Flow curves of digested sludge from different AD reactors (a. the digested sludge without dilution. b. the digested sludge with dilution).

digested sludge with pretreatment (Fig. 1a) or may be the result of changes of digested sludge micro-structures. For clarity, the digested sludge was diluted with deionized water to a solid concentration of approximately 5%, as listed in Table 2. With dilution, it was observed that the apparent viscosity of the digested sludge from reactors that received the pretreatment was still obviously lower than that of the control (Fig. 4b), suggesting that the low apparent viscosity of digested sludge with pretreatment was mostly due to the changes of the sludge micro-structures. By fitting the curves with the Herschel-Bulkley model (Fig. 4b), the yield stress (τ_y) and consistency index (k) of the digested sludge in the reactors combined with pretreatment were obviously lower than those of the control, and in contrast, the flow behavior index (n) was increased (Table 2). The pretreated sludge through AD still displayed high flowability and weak pseudo-plastic properties compared with the sludge without pretreatment. The rheological properties of the digested sludge within the non-linear viscoelastic region were not obviously influenced by the different configurations of reactors (one stage or two stage).

Similarly, the viscoelastic properties of the digested sludge within the linear viscoelastic region were analyzed by frequency sweep tests (Fig. 5). The digested sludge presented a solid-like regime ($G' > G''$). The viscoelastic properties of the digested sludge in the reactors fed with pretreated sludge became weak, as shown in Fig. 5a, b, c. The values of tan (δ) increased from 0.24 in the control reactor to 0.38 and 0.47 in the MW-one stage and MW-two stage reactors (the second stage reactor), respectively. The digested sludge from reactors that received the pretreatment presented weaker solid-like properties (elastic characteristics). In addition, it was found that the different reactor configurations impacted the viscoelastic properties within the linear viscoelastic region. The digested sludge from the MW-two stage reactor presented the weakest elastic properties. Even at nearly the same solid concentration (Fig. 5d, e, f), the digested sludge from the AD reactors that received pretreatment still presented weak solid-like properties.

Furthermore, the creep-recovery test was used to analyze the viscoelastic properties of the digested sludge which was diluted (Fig. 6). The deformation of viscoelastic material is exhibited by the variation of strain or compliance with time (Dolz et al., 2008). The parameters that are readily available from the creep-recovery

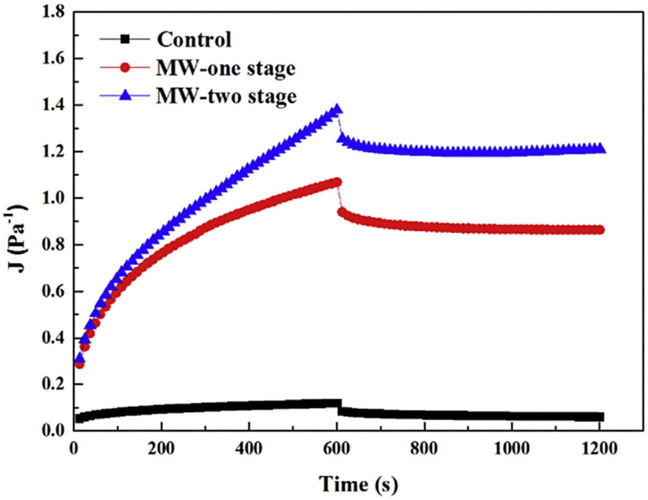

Fig. 6. Creep-recovery test for digested sludge which were diluted.

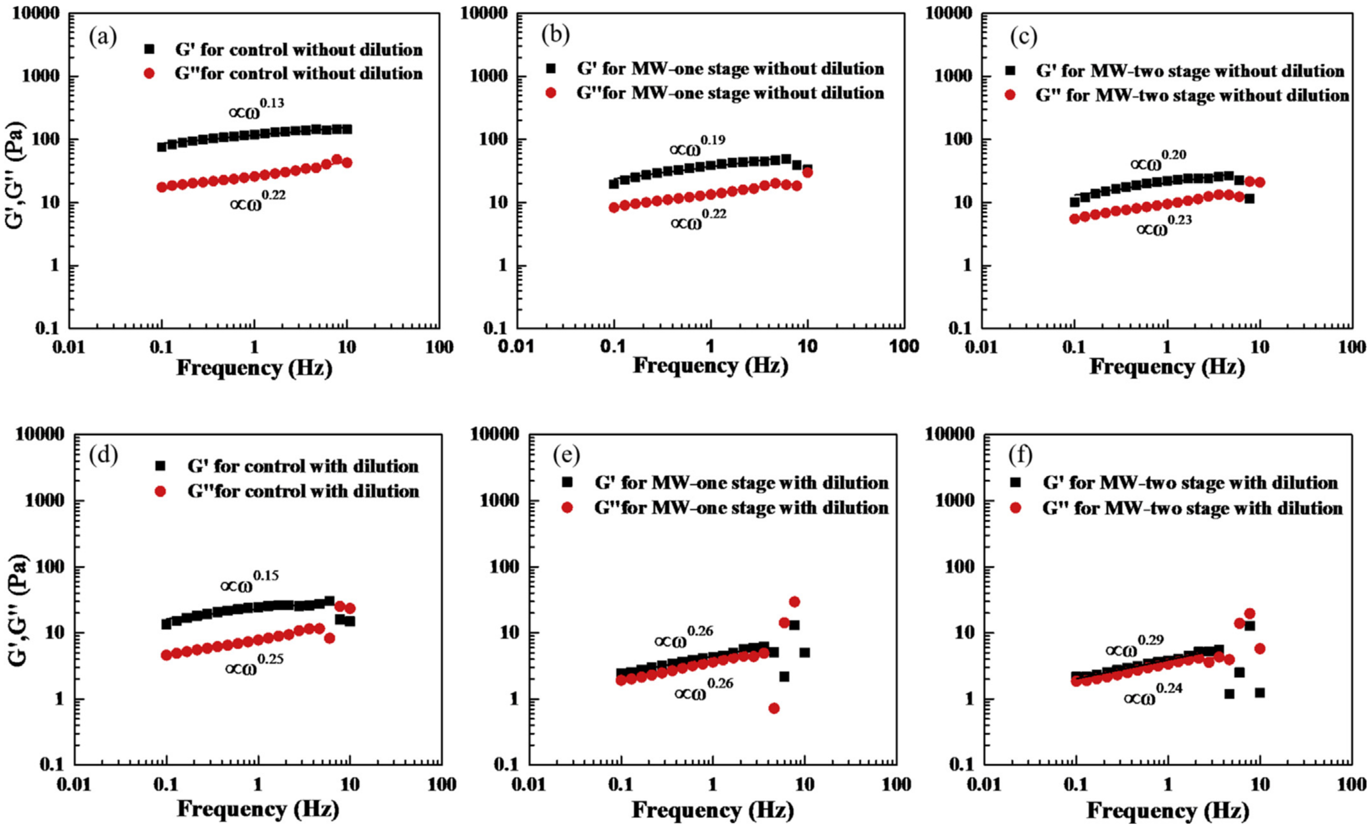

Fig. 5. Frequency sweep of digested sludge (Control (a without dilution, d with dilution), MW-one stage (b without dilution, e with dilution), MW-two stage (c without dilution, f with dilution)).

curves are the maximum creep compliance, $J_{c,max}$, and maximum recovery compliance, $J_{r,max}$ (Van Bockstaele et al., 2011): $J_{r,max} = J_{c,max} - \lim_{t\to\infty} J_r(t)$ (3). As expected, a significantly lower digested sludge compliance ($J_{c,max}$) of the control was observed, suggesting stronger internal structures. In contrast, the $J_{c,max}$ of the digested sludge from the reactors that received the pretreatment was much higher. Moreover, the digested sludge from the MW-two stage reactor presented the highest value of $J_{c,max}$. The increase of creep compliance suggested that the digested sludge from reactor with pretreatment, especially the MW-two stage reactor, was more easily deformed by a given stress (Ruiz-Hernando et al., 2014). This result is consistent with the frequency sweep tests, namely, the digested sludge from reactors with pretreatment showed less rigid or elastic structures.

3.4. *Effects of pretreatment on digested sludge characteristics*

According to the rheology analysis, the digested sludge from reactors that received the pretreatment presented a greater flow in the steady state and less elasticity in the linear viscoelastic region. These results suggested that there are weak colloidal forces in the internal structure or a less rigid structure. Activated and digested sludge are mainly composed of water, organic matters, microbial cells and EPS, which tends to aggregate, forming flocs. The characteristics of the adsorption ability, hydrophilicity/hydrophobicity and content of the main components, such as proteins and polysaccharides, of EPS critically affect the properties of microbial aggregates, e.g., mass transfer, surface characteristics and stability (Sheng et al., 2010). Moreover, EPS also has a strong relationship with sludge rheology according to previous studies (Baudez et al., 2013; Ma et al., 2014; Seviour et al., 2009).

According to the analysis of EPS (Fig. 7a), large amounts of soluble EPS existed in the digested sludge of reactors that received the pretreatment compared with that of the control, whereas the concentrations of bound EPS were relatively the same for the different reactors. In the soluble EPS, the amounts of proteins and polysaccharides were obviously higher than those of the digested sludge of the control reactor (Fig. 7b). The concentrations of proteins and polysaccharides in the bound EPS did not present obvious differences (Fig. 7c). Moreover, it was found that pretreatment partly changed the proportion of proteins and polysaccharides in EPS (Fig. 7d). The ratio of proteins to polysaccharides in bound EPS decreased when the feeding sludge underwent MW-H_2O_2 pretreatment. In addition, the ratio of proteins to polysaccharides in both soluble EPS and bound EPS presented the lowest value in the digested sludge from the MW-two stage reactor. These results imply that MW-H_2O_2 pretreatment strongly affected the concentration and components of EPS in digested sludge. Furthermore, it was observed that the molecular weight of the soluble organic matter in digested sludge was influenced by pretreatment;

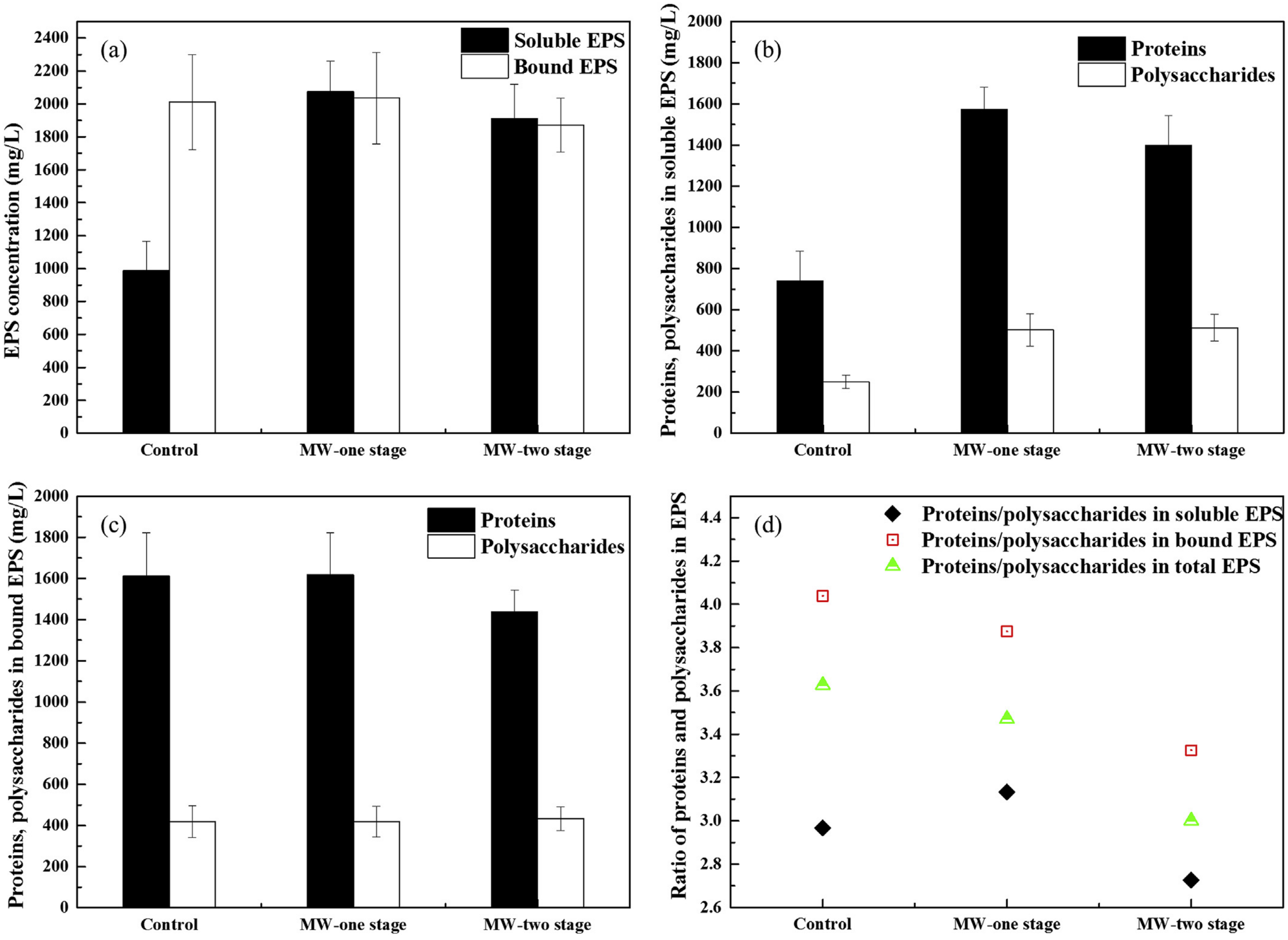

Fig. 7. The concentration and components of EPS in digested sludge.

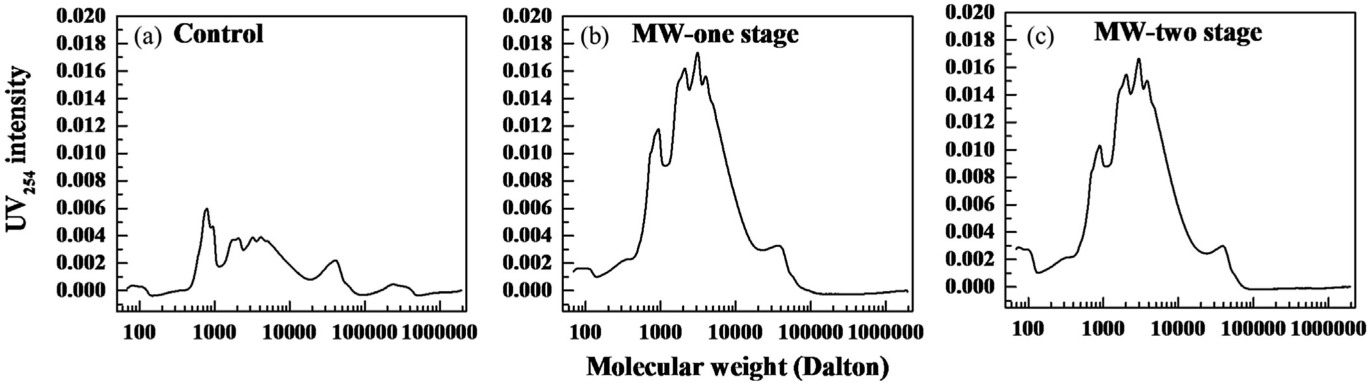

Fig. 8. Molecular weight distribution of soluble organic matters in different reactors.

however, little difference between the MW-one stage reactor and the MW-two stage reactor was observed (Fig. 8). The pretreatment process resulted in a greater amount of soluble organic matter released at molecular weights lower than 20 kDa. In addition, the morphology of digested sludge was partly changed due to pretreatment as shown in Fig. S3, which was included in the supporting information. It seems that the microbial cells were less tightly embedded in sludge flocs when the pretreated sludge fed into the digesters.

Therefore, the change of digested sludge micro-structures, e.g., the concentration and components of EPS, may have contributed to the variation of rheology between different digested sludge. It seems that the large amount of soluble organic matter in the digested sludge, in the form of proteins and polysaccharides was related to the rheological properties in the steady state flow, e.g., the lower yield stress and lower apparent viscosity. In general, a large amount of soluble organics released is an indication of sludge solubilization as a result of bound EPS solubilized or cell lysis. This solubilization process may result in the reduction of yield stress, apparent viscosity (Farno et al., 2015). Moreover, the variation of EPS components, such as the ratio of proteins and polysaccharides in bound EPS, was possibly related to the lowest elastic properties of the digested sludge from the MW-two stage reactor in the linear viscoelastic region. Although no studies have ever demonstrated that the ratio of proteins and polysaccharides in EPS was related to the viscoelastic properties of sludge, several studies have noted that this variation of EPS components might affect other sludge physical characteristics, such as filterability or dewaterability (Shao et al., 2010, 2009; Ye et al., 2012), which is also related to the sludge rheology (Hou and Li, 2003; Marinetti et al., 2010).

4. Conclusions

In this study, based on the stable operation of enhanced AD combined with MW-H_2O_2 pretreatment, the rheology evolution of sludge was investigated. After MW-H_2O_2 pretreatment, the sludge flowability was improved and the elastic properties weakened. MW-H_2O_2 pretreatment has positive effects on the rheological evolution of sludge in anaerobic digesters, namely, better flowability and weaker viscoelastic properties of the digested sludge. The strength of the inner structures and non-Newtonian flow characteristics of digested sludge also weakened. The changes of micro-structures, such as the concentration and components of EPS, possibly contributed to the evolution of the digested sludge rheology. Therefore, the AD of sludge combined with MW-H_2O_2 pretreatment is a more favorable option than the single AD process from the perspective of rheological evolution.

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Appendix A. Supplementary data

Supplementary data related to this article can be found at http://dx.doi.org/10.1016/j.watres.2016.03.073.

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