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Physical characteristics of conditioned anaerobic digested sludge – A fractal, transient and dynamic rheological viewpoint

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Abstract

The changes in the physical characteristics of unconditioned and conditioned anaerobic digested sludge (ADS) biosolids, such as capillary suction time (CST), yield stress, average size and fractal dimensions, were investigated through a CST test, transient and dynamic rheological test and image analysis. The results showed that the optimum polymer dose range was observed when CST or its reciprocal value was employed as an indicator. There were good correlations between the yield stresses determined from both a controlled shear stress test and a strain amplitude sweep test. The yield stress and storage modulus (G') increased as the polymer dose increased in most cases. A frequency sweep test revealed that polymer conditioning could extend the frequency sweep ranges for their elastic behaviors over viscous behaviors as well as the gel-like structure in the linear viscoelastic range. These results implied that more deformation energy was stored in this rigid structure, and that elastic behavior became increasingly dominant with the addition of the polymer in most cases. In addition, both the average sizes and two-dimensional fractal dimensions for conditioned ADS biosolids presented a similar up-climax-down variation trend as the polymer doses increased, whereas the critical polymer doses at the highest average sizes or two-dimensional fractal dimensions, were different. Correlation analysis revealed that the conditioned ADS dewaterability was not correlated with the yield stresses, while the average sizes or the two-dimensional fractal dimensions for conditioned ADS biosolids could be taken as the indication parameters for ADS dewaterability.

Key words: anaerobic digested sludge; conditioning; dynamic oscillatory test; yield stress; fractal dimensions; polymer dose

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Introduction

Rheological parameters are very important in sludge management and treatment processes (Campbell and Crescuolo, 1982; Dentel, 1997; Lotito et al., 1997; Moeller and Torres, 1997; Slatter, 1997; Christopher, 2002; Yen et al., 2002; Seyssiecq et al., 2003; Dursun et al., 2004; Dentel et al., 2005; Ayol et al., 2006b; Dursun, 2007), and the yield stress is a parameter used to define the immediate point at which flow begins (Mezger, 2002; Ayol et al., 2006a). This is useful for determination of design parameters of above processes, especially for evaluating the pressure drops in pipes for sludge transportation by pumps (Lotito et al., 1997; Yen et al., 2002; Dentel et al., 2005), and predicting conditions in which sludge flow will enable the liquid phase to be separated from the solid phase (Ayol et al., 2006a).

It is well-known that wastewater sludges are non-Newtonian fluids (Campbell and Crescuolo, 1982; Dentel, 1997; Lotito et al., 1997; Moeller and Torre, 1997; Slatter,1997; Christopher, 2002; Yen et al., 2002; Seyssiecq et al., 2003; Dursun et al., 2004; Dentel et al., 2005; Ayol et al., 2006b; Dursun, 2007), and their flow behavior can often be described by fairly basic rheological models and analyses (Ayol et al., 2006a). In general, these biosolids are mainly composed of a large amount of bio-flocs with an average size of less than 100 μm (Christopher, 2002; Yen et al., 2002; Dursun, 2007). However, once a flocculant polymer is added to condition the sludge, the newly formed non-Newtonian fluid will become a kind of complex fluid that has decreased amount of aggregates with larger size due to flocculation of bio-flocs (Langer et al.,1994; Wu et al., 1997; Wen et al., 1997; Rattanakawin and Hogg, 2001; Zhao, 2003; Chu et al., 2004). Therefore, the conventional models employed to account for original sludge having a finite yield stress, such as the Bingham plastic, Herschel-Bulkley, or Ostwald models, failed to provide corresponding information regarding the yield stress of these conditioned biosolids (Dentel et al., 2005; Ayol et al., 2006a). Accordingly, more elaborate models or new

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methods should be developed to solve the aforementioned problems.

In recent years, a newer approach based on dynamic rheology has been employed to determine the yield stress of conditioned sludge (Ayol, 2005; Dentel et al., 2005; Ayol et al., 2006a; Dursun, 2007). In this method, the controlled amplitude can be varied in a sinusoidal manner to identify the linear viscoelastic (LVE) range, where the critical values of storage modulus (G') and strain can be calculated for this purpose. Moreover, a frequency sweep test in a sinusoidal manner was introduced to characterize the viscoelastic behavior of complex fluids. Previous studies (Dentel et al., 2005; Ayol et al., 2006a; Dursun, 2007) conducted corresponding experiments to determine the yield stress of conditioned wastewater active sludge, anaerobic digested sludge (ADS) and synthetic sludge according to the above procedures. They reported that more accurate yield stresses can be estimated, and better predictions of sludge behavior in treatment and transport processes can be attained through the dynamic rheological test.

In addition to rheological characterization for such complex fluids-conditioned wastewater sludges, the aggregate properties of these biosolids, such as their morphology, size and structure, have been focused by many researchers (Langer et al., 1994; Rattanakawin and Hogg, 2001; Chu et al., 2004). In general, natural aggregates such as those formed during water and wastewater treatment (Li and Ganczarczyk, 1989; Chu et al., 2004) have been characterized as highly porous fractal-like forms of many primary particles. A number of studies have indicated that the conditioned sludge aggregates also appear as a fractal (Wu et al., 2002; Chu et al., 2004). In fact, a powerful application of fractal geometry in describing the unconditioned and conditioned sludge aggregate structure lies in its potential to relate formation processes to its structure (Li and Ganczarczyk, 1989; Chu et al., 2004). Thus, how to measure the mass fractal dimension of sludge is of considerable interest. However, the relationship between the fractal properties of aggregates and polymer doses during the conditioning process is still unclear. Therefore, this study was conducted to investigate the effect of polymer doses on size, fractal dimensions and yield stresses of conditioned ADS aggregates. The correlation between their fractal dimensions and yield stress values was also analyzed.

1 Methods and materials

1.1 Original anaerobic digested sludge

Raw ADS was collected from the Wilmington Wastewater Treatment Facility in Wilmington, DE, USA on September 13, 2009. After ADS was collected, it was immediately transferred to the laboratory at the University of Delaware and stored at 4°C. Prior to the experiments, the sludge sample was warmed to 25°C. All measurements were performed within three days of sampling. The dry-solid content of the sludge sample was determined based on the weight loss of sludge samples that were dried at 105°C over 24 hr.

1.2 Zetag7557 polymer and conditioning procedure

A high molecular weight cationic organic polymer, zetag7557, was obtained from Ciba Specialty Chemicals, Switzerland. This compound is an acrylamide-based copolymer (Quaternized 40/60 mole ratio AETAC/AM DMAEAQ:MeCl) that has an average molecular weight of more than ten million Daltons. For ADS conditioning with zetag7557 polymer at the laboratory scale, a jar test was performed using a classical multipaddle stirring apparatus (Phipps and Bird, USA). Briefly, 1 L beakers were filled to 500 mL with sludge samples under a multipaddle stirrer. A stock solution of 0.5% zetag7557 was then poured into the sludge sample contained in each beaker while mixing at 300 r/min (G = 916.49 sec^{-1} as water at 25°C) within 5 sec, after which the samples were mixed for an additional 60 sec. The samples were then subjected to extended mixing at 30 r/min (G = 28.98 sec^{-1} as water at 25°C). Immediately after mixing, the conditioned ADS aggregate samples at different polymer doses were withdrawn and drained through a filter paper until no free water can be observed. Then the obtained paste was used for dynamic rheological test.

1.3 Rheological test for conditioned ADS

Rheological test was conducted using an advanced rheometer (Physica MCR 500, Anton Paar, Austria) with the temperature maintained at 25°C by a Peltier control. A PP 50 plate and plate sensor with 49.94 mm diameter and 2.0 mm gap was used. Two methods of rheological test were employed for yield stress determination. The first was a controlled shear stress (CSS) test, in which the shear stress was increased in a ramp manner to determine the strain (deformation) response as a function of the imposed values. Using this method, the critical point shear stress can be determined from the curve of the strain-shear stress and considered as yield stress for a sample. A second rheological test mode is commonly termed as the strain amplitude sweep test, which indicates variations in strain amplitude over time while the frequency is held constant. This method was conducted determine the LVE range. The rheogram of the moduli (G', G'', G^*) as a function of strain on the logarithmic coordinates can present that the rheological properties (including the above moduli) are independent of strain until a critical strain level (γ_c) is reached. After this point, the material's behavior is nonlinear, and the transition can be used to determine γ_c. This determination is based primarily on data collected before reaching the yield stress, in contrast to the traditional steady shear test. In addition to leaving the sample more intact, the dynamic mechanical test is also advantageous because it is simpler and model-independent (Larson, 1999; Mezger, 2000; Dentel et al., 2005).

After determining the yield stress and LVE range by strain amplitude sweep test, frequency sweep tests of the ADS samples were conducted at 0.1% of the strain value. The corresponding storage and loss modulus were measured during the test, and these two moduli comparisons can provide some information regarding the structure of the ADS samples.

1.4 Determination of physicochemical and geometric characteristics of ADS with or without conditioning

The same rheological tests were also conducted for the raw ADS without drainage. The zeta potential, conductivity and viscosity of the supernatants after 15 min of centrifugation of the raw ADS at 3000 r/min were examined according to the method described by Dursun (2007). The capillary suction time (CST) was used to indicate the dewaterability of the conditioned ADS according to the Standard Methods (APHA et al., 2005). The reciprocal of CST was employed to indicate the filterability rate (Ayol et al., 2006b; Dursun, 2007). Triplicates were conducted for each experiment to ensure the consistency of results.

The geometric characteristics of conditioned sludge aggregates including size distribution and fractal dimension were determined. The conditioned sludge aggregate samples were carefully withdrawn from the stirred beaker immediately after the flocculation procedure, and then introduced into deionized water contained in a glass square dish evenly and separately using a wide mouthed pipette for image recording. Aggregates that broke up in response to the flow shearing at the mouth of the pipette were removed and discarded. After transfer was complete, a digital camera was used to take pictures of the aggregates in the dish and the geometric characteristics were derived from the images using software Image-pro Plus 5.0. Due to the absence of spherical aggregates, they can be characterized in many ways, and the software then determines the area, perimeter, and diameter of the image for each aggregate.

The size distribution of these aggregates was determined through statistical analysis of the above geometric parameters. Their one-dimensional fractal dimension (D_1) was calculated by regression analysis of the logarithm of their perimeters versus the logarithm of their corresponding characteristic lengths (Jin and Wang, 2001; Wang et al., 2009). The two-dimensional fractal dimensions of D_{2L} or D_{2P} for the aggregates was calculated by regression analysis of the logarithm of their projected areas versus the logarithm of their corresponding characteristic lengths or perimeters, respectively (Jin and Wang, 2001; Wang et al., 2009). In this study, the longest diameter of an aggregate image was taken as the characteristic length.

1.5 Correlation analysis among the corresponding characteristics of conditioned ADS

The Pearson's correlation analysis method in SPSS 13.0 was used to investigate the linear correlations among the rheological and geometric characteristics of conditioned ADS.

2 Results and discussions

2.1 Effect of polymer zetag7557 doses on dewaterability of ADS

2.1.1 Physicochemical properties of original ADS

The original ADS was characterized prior to conditioning and rheological testing. The results are as following: pH 7.32; total solids of ADS suspension (23.23 ± 0.43) g/L; zeta potential (–13.57 ± 0.78) mV; centrate viscosity (2.25 ± 0.01) mPa·sec; and CST (271.61 ± 33.40) sec. The results indicated that ADS took negative surface charges and that the viscosity of its supernatant after centrifugation at 3000 r/min was more than twice that of water.

2.1.2 Dewaterability of ADS after conditioning with zetag7557 polymer

Figure 1 represents the characterization of ADS suspensions with regard to conditioning and dewatering. The trends in the CST results resemble variations that have previously been observed in sludges (Dursun et al., 2004; Ayol et al., 2006b; Dursun, 2007). The CST of the ADS suspension decreased sharply with the lower polymer doses due to particles flocculation in response to the added polymer, reaching a minimum of 8.18 ± 0.75 sec at approximately 12.91 kg/ton ADS, and then increased again at higher doses. The reciprocals of the CST values showed trends opposite to those of the CSTs as the amount of the polymer increased, and a more evident climax was observed in the 1/CST-polymer dose curves as well. It is clear that the optimum amount of polymer dose was 12.91 kg/ton ADS, while at a dose of 17.22 kg/ton ADS, the CST value of the conditioned ADS suspension is 8.43 ± 0.36 sec and close to that observed at the optimum polymer dosage. Therefore, these two polymer doses can be taken as the optimum dose range for ADS conditioning.

2.2 Comparison of yield stresses of ADS determined from different rheological test models

2.2.1 Yield stress determined by a controlled shear stress (CSS) test and strain amplitude sweep (SAS) test

Figure 2 gives the strain-shear stress data of ADS samples with and without polymer conditioning. As shown in Fig. 2, each strain-shear stress curve has a critical shear stress, which can be regarded as the corresponding yield

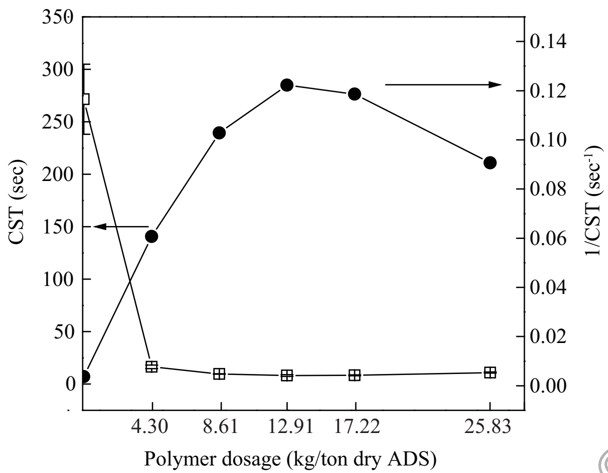

Fig. 1 Variations in CST values of ADS as a function of zetag7557 polymer doses.

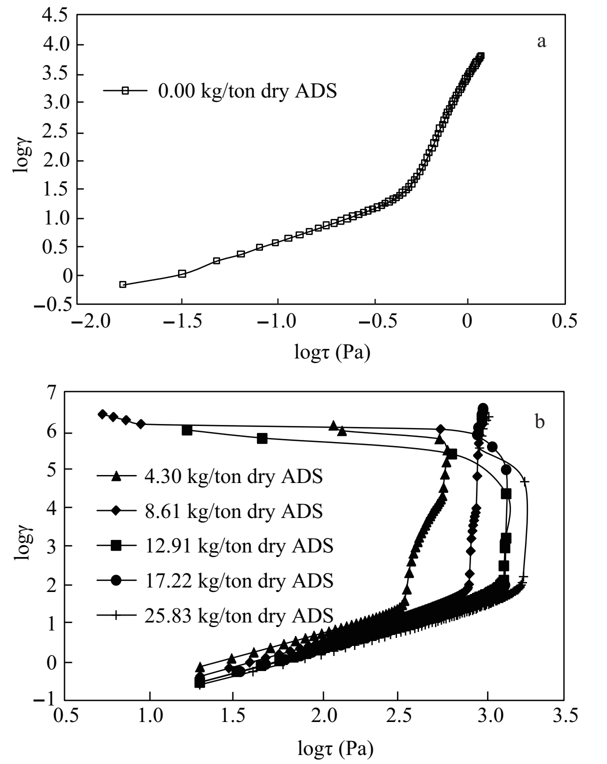

Fig. 2 Controlled shear stress (CSS) test results for unconditioned ADS and ADS conditioned with polymer zetag7557. (a) original ADS; (b) conditioned ADS. γ represents the relative shear strain, and its value is the percentage of the shear strain amplitude.

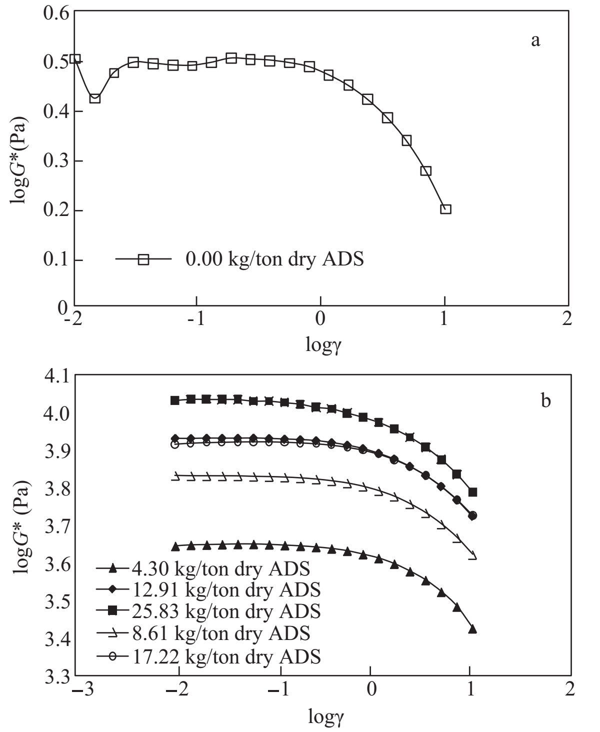

Fig. 3 Strain amplitude sweep (SAS) test results for unconditioned ADS and ADS conditioned with polymer zetag7557. (a) original ADS; (b) conditioned ADS. γ: the relative shear strain, and its value is the percentage of the shear strain amplitude.

stress of ADS. Here, the yield stress (critical stress) can be determined using the tangent crossover method (Mezger, 2002; Dentel et al., 2005; Ayol et al., 2006a; Dursun, 2007). In this method, both tangents are set in the first curve interval with low shear stress or strain and the second curve interval with high shear stress or strain and their crossover at the shear stress is taken as the yield point. It is obvious that the yield stress of the original ADS was lower than that of the conditioned ADS after drainage when compared to the critical stress at the yield point in the strain-shear stress curves shown in Fig. 2a and b. These findings indicated that the conditioning polymer imparted a significant increase in shear resistance (Dentel et al., 2005). Moreover, under most conditions, the corresponding yield stress increased as the polymer doses increased, while the close yield stresses could be observed at polymer doses of 12.91 and 17.22 kg/ton dry ADS.

The change in complex modulus (*G**) as a function of strain at constant frequency ($f = 1$ Hz) for ADS is presented in Fig. 3. For both unconditioned and conditioned ADS during the SAS test, a nearly strain-independent *G** value was observed with a constant plateau up to a critical strain value (γ_c). This *G**-strain curve interval can be taken as the LVE range, and the storage modulus was significantly greater than the loss modulus in this range. After this point, due to the disruption of the polymer-mediated network structure and the release of easy-flowing solution phase material, the decrease of the storage modulus and increase of the loss modulus showed a nonlinear behavior of ADS, and this transition was used to determine γ_c. The yield stress can then be determined as the constant *G** value multiplied by the critical strain value.

Comparison of the *G**-strain curves in Fig. 3a and b shows that *G** values for the conditioned ADS after drainage were three orders of magnitude higher than that of the original ADS, which support the corresponding results observed in the CSS test. In addition, increased polymer doses can impart a more significant increase in shear resistance except for the increase in the polymer doses from 12.91 to 17.22 kg/ton dry ADS, at which similar *G** values were observed.

2.2.2 Comparison of yield stresses based on CSS and SAS tests

Figure 4 provides the change in yield stresses based on CSS and SAS tests with increasing polymer doses. As

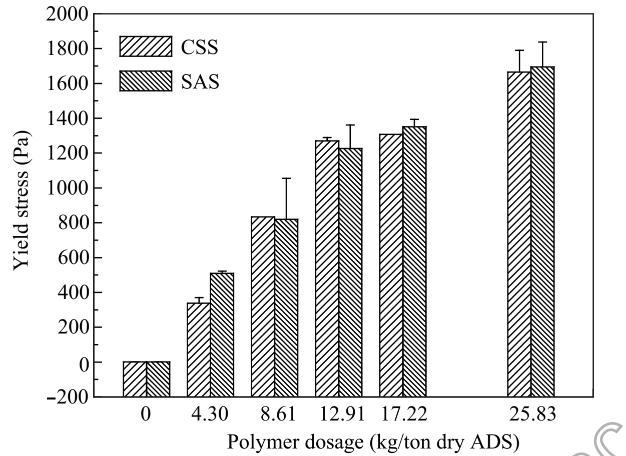

Fig. 4 Yield stress obtained by CSS and SAS tests as a function of polymer doses.

shown in Fig. 4, the yield stress obtained by SAS mode was in aline with that by CSS mode, except at zetag7557 dosage of 4.3 kg/ton dry ADS, where a difference of more than 150 Pa was observed between two tests. In addition, yield stress value increased with increasing polymer doses. However, Dursun (2007) observed a decrease in yield stress value in response to the addition of polymer at doses greater than 300 mg/L in wastewater activated sludge. The difference could attributed to the types of sludge used in studies.

2.3 Frequency sweep test for unconditioned and conditioned ADS

Figure 5 presents results of a frequency sweep test for ADS biosolids with and without conditioning. For original ADS (Fig. 5a), the storage modulus (G') was slightly higher than the loss modulus (G'') within the range of 0.1–1 Hz, demonstrating a slightly stronger elastic behavior ($G' > G''$) than viscous behavior, while in the range of 1–100 Hz an opposite trend was observed for these two moduli. As shown in Fig. 5b–f, the storage moduli (G') for the conditioned ADS biosolids at different polymer doses dominate the corresponding loss moduli (G'') in the range of 0.1–100 Hz, which demonstrated that once ADS was conditioned with zetag7557 and held less water, the frequency sweep ranges for their elastic behaviors were extended over their viscous behaviors. While for original ADS without conditioning, the water hold capacity was higher, and exhibited a less elastic phase.

In general, the phenomena of $G' > G''$ is expected for solids and pastes; however, for sludge that has low viscosity at high shear rates, presenting $G' > G''$ in the

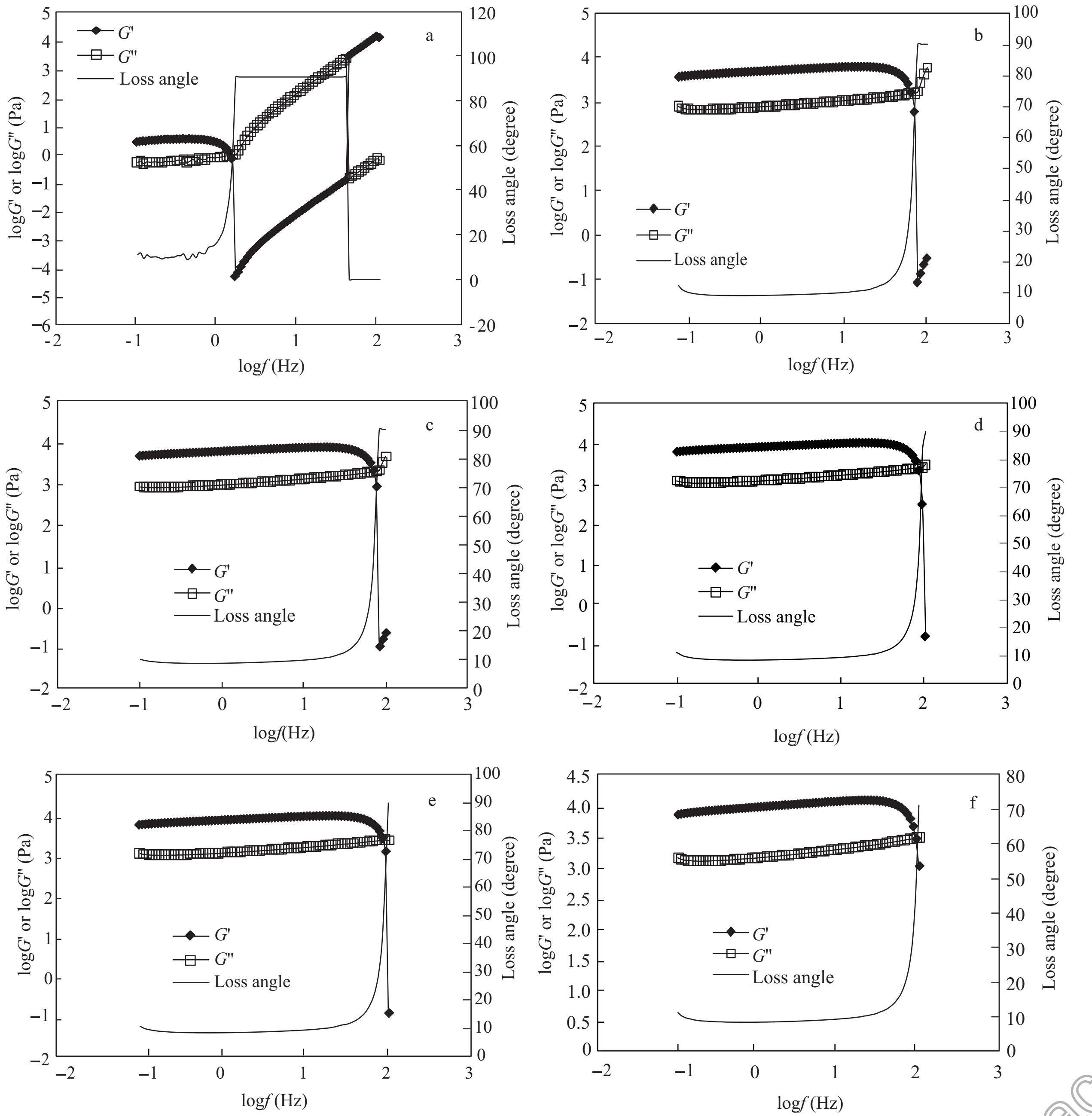

Fig. 5 Frequency sweep test results for unconditioned and conditioned ADS. (a) original ADS; (b) 4.3 kg/ton dry ADS; (c) 8.61 kg/ton dry ADS; (d) 12.91 kg/ton dry ADS; (e) 17.22 kg/ton dry ADS; (f) 25.83 kg/ton dry ADS.

LVE range, implied a gel-like structure. Therefore, the frequency sweep test curves in Fig. 5b–f implied that these conditioned biosolids showed gel-like behavior in the LVE range (Mezger, 2002; Dursun, 2007) as well.

The effect of conditioning on the elastic modulus is shown in Fig. 6. With increasing polymer doses, the storage modulus (G') values increased in most cases, while close values were observed at 12.91 and 17.22 kg/ton dry ADS. These results demonstrate that the internal structure of the conditioned ADS network becomes less flexible and more rigid as polymer dose increases in most cases, and the same internal structure was observed for conditioned ADS at polymer doses of 12.91 and 17.22 kg/ton dry ADS. An increase in the G' value also implied that more deformation energy is stored in this rigid structure, and the elastic behavior becomes increasingly dominant.

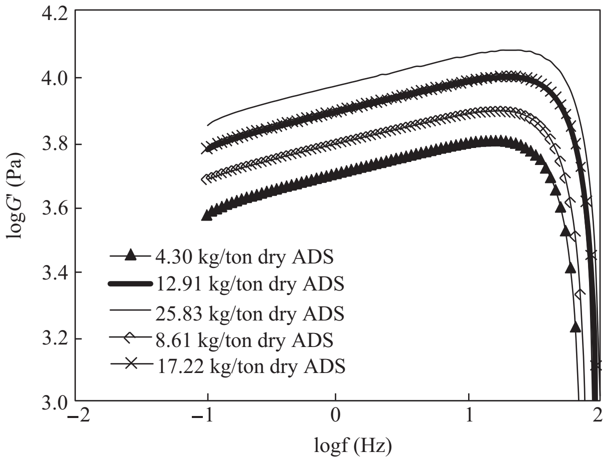

Fig. 6 Effect of conditioning on elastic modulus.

2.4 Effect of polymer doses on the geometric characteristics of ADS biosolids

The conditioned ADS biosolids were withdrawn and their pictures were recorded at different polymer doses. Figure 7 provides several images of these aggregates after 300 sec of mixing at 30 r/min during the ADS conditioning process. A few copper cords with diameters of 1 in. were used as the staff gauges. At polymer dose of 8.61 kg/ton dry ADS, about 850 conditioned ADS aggregates were taken for image recording and further statistical analysis, while at other polymer dosages, more than 1000 ADS flocs/aggregates were taken. As shown in Fig. 7, larger aggregates were observed at 12.91 and 17.22 kg/ton dry sludge, which is in accordance with the results shown in Fig. 8a. Moreover, Fig. 8a shows that a similar trend was observed in three types of average diameters, and the volumetric average diameters were higher than other two diameters.

The change in the fractal dimensions of aggregates as the polymer doses increasing is displayed in Fig. 8b. One-dimensional fractal dimensions of conditioned ADS aggregates increased as the polymer doses increased up to 8.61 kg/ton dry ADS during the conditioning process, after which they decreased, and then increased again during further polymer addition. All of the D_1 values observed at different polymer doses were between 1.0–1.10. These findings demonstrated that the boundary and surface of the conditioned ADS aggregates became slightly more irregular as the polymer doses increased to 8.61 kg/ton dry sludge, and then became less irregular at a polymer dose of 12.91 kg/ton dry sludge, finally becoming irregular at the other polymer doses.

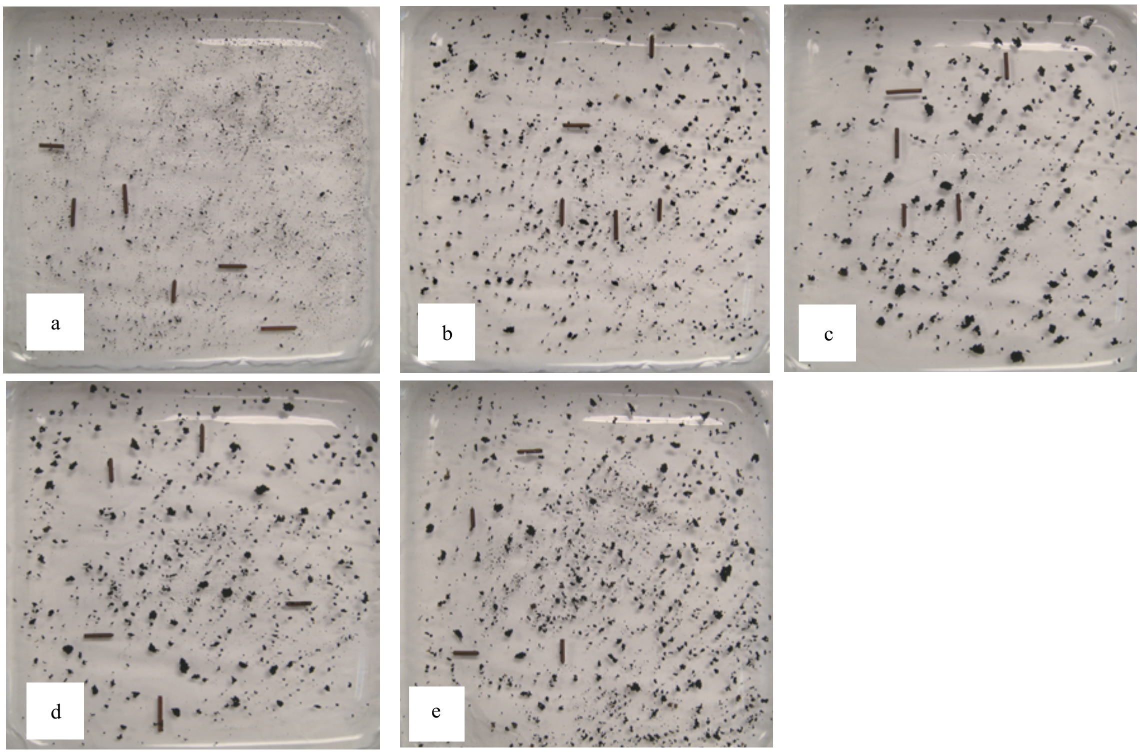

Fig. 7 Images of ADS aggregates conditioned at different polymer doses. (a) 4.30 kg/ton dry ADS; (b) 8.61 kg/ton dry ADS; (c) 12.91 kg/ton dry ADS; (d) 17.22 kg/ton dry ADS; (e) 25.83 kg/ton dry ADS.

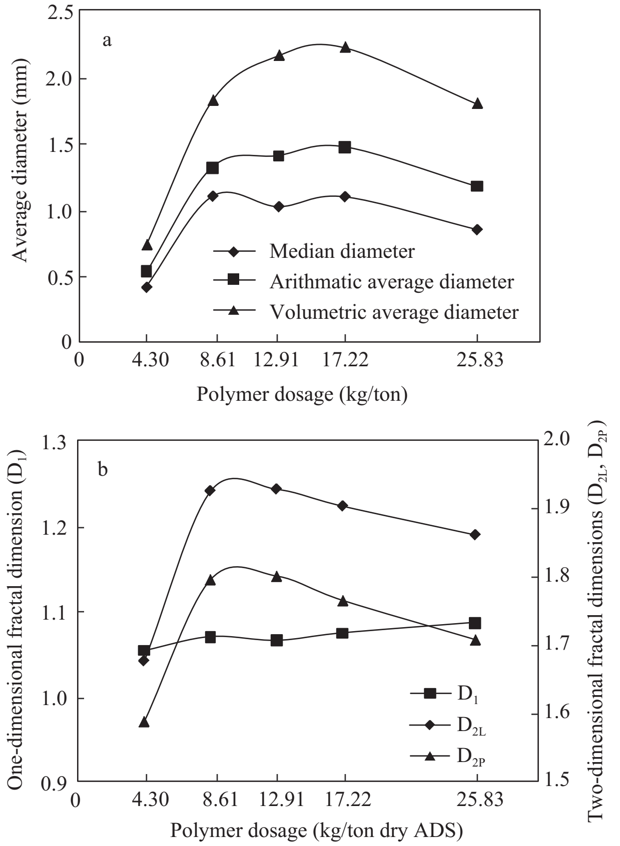

Fig. 8 Geometric parameters of ADS aggregates conditioned at different polymer doses. (a) average size; (b) fractal dimensions.

As shown in Fig. 8b, the high D_2 values were obtained at 8.61 kg/ton dry ADS to 12.91 kg/ton dry ADS, indicating that increasingly compact aggregates were formed as the polymer doses increased to about 10.00 kg/ton dry ADS, while the aggregates became less compact in the range of polymer doses of 12.91 to 25.83 kg/ton dry ADS.

2.5 Correlation between the rheological and geometric characteristics of conditioned ADS

The correlations between the CST, size, fractal dimensions and yield stress obtained during the above tests were analyzed by Pearson's correlation analysis, and the results are shown in Table 1. For conditioned ADS biosolids, CST showed good linear correlations (coefficients > 0.90) with average size and two-dimensional fractal dimensions, and there were good linear correlations among the different types of average size or two-dimensional fractal dimensions as well. However, no linear correlations were observed between one-dimensional fractal dimension or yield stresses and other parameters, only correlations between one-dimensional fractal dimensions and yield stresses appeared. Furthermore, yield stress values based on different test modes were well correlated with each other.

Based on the correlation analysis results, the conditioned ADS dewaterability was not correlated with the yield stresses because their correlation coefficients were only about 0.75. Conversely, the average size or two-dimensional fractal dimensions for conditioned ADS biosolids can be taken as indicators of ADS dewaterability.

In general, yield stress is a parameter that indicates the critical value at which a fluid begins to flow. Higher than this value, the structure of non-Newtonian fluid, such as sludge suspension, will be destroyed by shearing. During wastewater sludge dewatering, several kinds of forces including shearing or pressure will be imposed on the conditioned sludge, and it is possible that the indicators of wastewater sludge dewaterability do not coincide with yield stress because of the complex functions of these forces. The geometric characteristics of the conditioned sludge aggregates, size and fractal dimensions, have a direct effect on the interaction between water and other parts of the sludge aggregates due to different types or magnitudes of the corresponding physicochemical forces being correlated with the aforementioned geometric parameters. Therefore, the dewaterability also shows certain correlations with these geometric parameters.

3 Conclusions

The optimum zetag7557 polymer dosage for ADS conditioning was found to be 12.91 kg/ton dry ADS.

The similar yield stresses determined from the controlled shear stress (CSS) test and strain amplitude sweep (SAS) test were well correlated, and they both increased as the polymer doses increased in most cases, while similar yield stress values could be observed at 12.91 and 17.22 kg/ton dry ADS in each test mode.

The frequency sweep test demonstrated that the conditioned ADS biosolids exhibited an extended elastic behavior range when compared to the original ADS, which implied that these conditioned biosolids showed gel-like structures in the LVE range.

Table 1 Pearson's correlation coefficients between the rheological and geometric characteristics of conditioned ADS-1

	CST (sec)	Median diameter (mm)	Arithmatic average diameter (mm)	Volumetric average diameter (mm)	D_{2L}	D_1	D_{2P}	Yield stress-CSS test (Pa)
Median diameter (mm)	–0.965**							
Arithmatic average diameter (mm)	–0.995**	0.978**						
Volumetric average diameter (mm)	–0.990**	0.940*	0.990**					
D_{2L}	–0.975**	0.979**	0.975**	0.950*				
D_1	–0.535	0.531	0.589	0.611	0.570			
D_{2P}	–0.961**	0.971**	0.951**	0.916*	0.985**	0.418		
Yield stress-CSS test (Pa)	–0.710	0.593	0.715	0.778	0.666	0.893*	0.540	
Yield stress-SAS test (Pa)	–0.752	0.497	0.630	0.704	0.564	0.898*	0.426	0.993**

** Correlation at 0.01 (2-tailed); * correlation at 0.05 (2-tailed).

The internal structure of the conditioned ADS network became less flexible and more rigid as polymer doses increased in most cases, while as polymer addition increased from 12.91 to 17.22 kg/ton dry ADS, close structural flexibility and rigidity were observed.

As the polymer doses increased, the average sizes of the conditioned ADS biosolids continued to increase to their maximum values, and then decreased when the amount of polymer added exceeded the critical values. Two-dimensional fractal dimensions also presented a trend similar to the aforementioned variations, whereas the critical polymer dosage was different based on variations in the average sizes.

For conditioned ADS biosolids, good correlations were observed between the CST and average sizes, D_2, but these parameters did not correlate well with the yield stresses.

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