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Systematic research on rheological behavior for flocculant-conditioned sludge suspension

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ABSTRACT

Rheology characteristics of raw sludge and cationic polyacrylamide (CPAM) conditioned sludge were both studied by steady test, thixotropy test, and dynamic test, respectively. Results of steady test showed Herschel–Bulkley, Bingham and Casson models could accurately describe the flow behavior of raw sludge and conditioned sludge, as the CPAM dosage was lower than 3 mg/g. An initial peak point for CPAM conditioned sludge at a low shear rate was observed, where the higher this peak was, the more energy was needed to break floc network. However, at a larger shear rate, the trend of rheogram was similar to that of raw sludge. Thixotropy was prominent for conditioned sludge, and the thixotropic loop was expanded with the increase of CPAM dosage. The strain sweep test demonstrated that the strength of floc network was intensified, leading to the larger elastic modulus. MGKV model which connected Maxwell model and series of Kelvin–Voigt modules was developed to characterize the creep recovery of sludge before and after conditioned. Furthermore, CPAM has more significant effect on delay time λ_1, which reduced by about 90% as the dosage was up to 3.0 mg/g. A smaller value of λ_1 explicated that conditioned sludge could restore to a new stable status in a shorter time. Compared to λ_2 and λ_3, λ_1 has more significant dominance during the entire creep recovery test.

Keywords: Sludge; Rheology; Organic flocculant; MGKV model

1. Introduction

Production of municipal sludge in China sharply increased with the rapid growth of urbanization in recent years, which may continue over the next decades because of the backwardness of disposal technology. Rheology is the science to study flow and deformation of materials, which was an important evaluation scale for monitoring and optimizing various unit operations in wastewater treatment, such as pump, mix, landfill, storing, thickening, dewatering, stabilization, heat exchangers and sludge transportation as well [1–4]. During the above processes, dewatering is perhaps the most complex, due to the complicate rheology after the addition of high molecular weight polyelectrolytes.

Some researchers have shown that sewage sludge belongs to a typical non-Newtonian fluid, which exhibits shear thinning and pseudoplastic behavior [5–7]. The non-linear relationship between shear stress and the shear rate was usually determined and characterized by Bingham, Ostwald, Herschel–Bulkley, Sisko, Careau, and Cross models [8–10]. The above process models generally focused on liquid-like behavior and shear thinning properties. While, viscoelasticity was paid less attention, which was important during sludge squeeze dewatering. Under certainly

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applied stress, concentrated sludge would produce distortion, and viscoelastic model could provide critical parameters to expound the deformation mechanisms. As the applied stress was low, sludge was considered as a rigid material in solid-like regime, exhibiting solid behavior (elastic characteristic). However, as the applied stress was high, it started to flow (viscous characteristic). Especially, as the applied stress reduced to zero, a partial elastic recovery was observed, which may be related to the elastic energy stored in inter-particle bounds [11]. In order to exactly understand viscoelastic properties between different materials, various mechanical models based on rheological theory were developed. Maxwell and Kelvin–Voigt as classical models, were widely used to describe the viscoelastic behavior of sludge. Farno et al. [12] indicated that fractional Kelvin–Voigt model could accurately fit the experimental data obtained from creep and frequency sweep tests compared to classical Kelvin–Voigt model. And for thermal hydrolyzed sludge, Kaye–Bernstein–Kearsley–Zapas model was developed [13]. Liang et al. [14] established a Burgers–Ludwik model to simulate sludge viscoelastic behavior under yield stress, and simulate viscoelastic plastic hardening behavior over yield stress. Ma et al. [15] proposed that Wagner-type model was able to characterize time-strain separability and to match viscoelastic responses with steady-state flow, which provided a promising step on predicting flow behaviors for sludge.

In addition, due to sludge complicate properties, many pretreatment methods were introduced [16–28], where the chemical condition was widely used with low price and utilization convenience. Some valuable information about the effect of flocculant on sludge rheology has been concluded. Chen et al. [5] discussed the viscoelastic characteristics of the sludge conditioned by organic flocculant. Hong et al. [8] indicated that once optimum polymer dosage has been reached, the sludge structures no longer undergo any changes but it does impact the liquid sludge properties. Koseoglu et al. [29] concluded the effect of six different chemical additives on sludge liquid characteristics. Ruiz-Hernando et al. [30] reported the effect of ultrasonic, thermal and alkali conditioning on sludge rheology, who indicated that the three treatment methods could significantly reduce sludge viscosity and thixotropy. Dieudé-Fauvel and Dentel [31] suggested that polymer dosage was the main parameter to influence sludge rheological and structural properties. And some researchers analyzed the rheological behavior of sludge after thermal hydrolysis [13,22,32]. Also, Ekama [33] studied the effect of sludge age.

Although many researchers studied sludge rheology, and some results were obtained. However, these results were not unified, due to the different rheometric methods and techniques. Meanwhile, the above researches little focused on the effect of flocculant dosage, and little referred to sludge creep and recovery, resulting in poor understanding of sludge viscoelastic performance. In order to get fully insight into the rheology of sludge conditioned by flocculant, all the rheology characteristics including liquid, thixotropy and viscoelastic behavior were systematically measured and assessed in this study. The MGKV model composed of Maxwell and series of Kelvin–Voigt models, was developed to describe creep recovery behavior of sludge suspensions.

2. Material and methods

2.1. Sludge materials

Municipal sludge was picked up from the Beijiao sewage treatment plant located in Taiyuan, Shanxi Province, China. Then the municipal sludge was stored at 4°C (for less than 1 week) before experiments, to ensure no temporal variability for the sludge sample. The primary characteristics of the sludge were measured by a series of tests according to CJ/T 221-2005 (China standard for municipal sludge analysis). Total suspended solids content (TSS), median particle size, pH, and specific surface area were 2.62, 71.89 μm, 6.88 and 946.7 cm^2/g, respectively.

2.2. Sludge conditioning

In order to carry out the next experiments successfully, the raw sludge was concentrated to about 7% (w/w) of TSS, meaning that sludge concentration was 70 g (dry sludge solid)/L. Organic flocculant cationic polyacrylamide (CPAM) with different dosage was employed to condition sludge to improve sludge physicochemical characteristics. Firstly, CPAM powder was first suspended in distilled water and then was stirred by mechanical stirrer with 250 rpm for 20 min to form a homogeneous suspension of 10 g/L. Secondly, the CPAM solution was added into sewage sludge to form mixed suspensions, which was stirred by mechanical stirrer in 200 rpm for 5 min to allow flocculant incorporation onto the particle surfaces, and then it was gently stirred at 50 rpm for 15 min to promote sludge flocs growth. Finally, different kinds of uniformly mixed sludge suspensions with a solid content of 7% and various flocculant dosages were obtained, which would be used for the next experiments.

2.3. Rheological experiment

Rheological experiments were performed on a rheometer (Hakke Mars60, Germany), which was equipped with a cup and a bob geometry (inner diameter of cup: 29 mm, outer diameter of bob: 25 mm, length: 34.4 mm) and connected with a water bath to allow reliable rheology measurements. In order to reduce the effect of long-term retention of sludge suspensions on rheological behavior [34], before each measurement, sludge sample was strongly pre-sheared for 5 min at a shear rate of 500 s^{-1} and then left at rest for 10 min [35,36] except for creep and recovery tests (to avoid sludge flocs or particles damaged). Each rheological test was carried out three times, and the average value was employed for subsequent analysis.

Rheological experiments mainly including steady test, strain sweep test and creep recovery test, were performed, respectively. For a steady test, shear rate logarithmically increased from 0.01 to 500 s^{-1}, and 20 data points were extracted to be analyzed. In strain sweep tests, the strain logarithmically varied from 0 to 2 to obtain a linear viscoelastic region. Creep recovery tests were carried out in the linear viscoelastic regime at applied constant shear stress. The response time linearly increased from 0 s to 600 s (creep stage) firstly, and then decreased from 600 to 1,200 s as the applied stress was removed (recovery stage).

2.4. Improved viscoelastic model

Kelvin–Voigt and Maxwell models are the two kinds of classical viscoelastic models. Kelvin–Voigt model represents solid-like behavior and consists of parallels of spring and dashpot modules. While Maxwell model is composed of a series of spring and dashpot modules. However, sludge has complex characteristics, and the individual model could not describe its viscoelastic behavior accurately. Therefore, an improved viscoelastic equation defined as MGKV was introduced. MGKV model was constitutive of multiple Kelvin–Voigt models and one Maxwell model, that is, parallels of spring (with elastic modulus G_1, ……, G_n) and dashpot (with viscosity η_1, ……, η_n) modules connected with series of spring and dashpot modules. The MGKV model was shown in Fig. 1, and the constructive equation was described as Eq. (1).

$$\gamma t = a + \sum_{1}^{i} b \times e^{-\frac{t}{\lambda_i}} \quad (1)$$

where $a = \tau_0 \times t_1/\eta_0$, $b = \tau_0/G_1$, …….; γ refers to shear strain; τ refers to shear stress, Pa; λ refers to delay time, s; η refers to viscosity, Pa·s; G refers to elastic modulus, Pa; t refers to creep experiment duration.

3. Results and discussion

3.1. Effect of CPAM on liquid characteristics of sludge

Liquid characteristics of sludge were observed by the steady shear test, which reflected sludge flow behavior. Fig. 2 shows the liquid characteristics of sludge conditioned with different dosages of CPAM. Shear-thinning behaviors were obtained for raw and conditioned sludge, respectively. Obviously, there was an initial peak point for conditioned sludge at a low shear rate, which was maybe attributed to the existence of elastic floc network. CPAM as a high molecular flocculant could provide enough inter or intra-attractive forces, such as van der Waals force, the hydrogen bonding, and the electrostatic forces between oppositely charged polymers and sludge particles. As a result, the higher strength of the floc network and higher viscosity were both achieved [5]. The initial peak was the point at which the network bonds were broken, and the higher this peak was, the more energy was needed [10]. As the shear rate was beyond the peak, the network was broken. Interestingly, the trend of rheogram in various dosages was similar to that of raw sludge at a larger shear rate, meaning the flocculant could only result in shifting of the rheogram, which was consistent with Dieudé-Fauvel's [31]. Also, the conditioned sludge exhibited a non-zero initial value, which was close to the fitted value τ_0 (yield stress) in Table 1, indicating that sludge suspensions belong to pseudoplastic fluid with yield stress [29,37]. While, as the CPAM dosage was up to 3 mg/g, the initial peak was lower. With the increase of CPAM amount, more positive charges were added to sludge particles. Ultimately, sludge maybe has the same electrical charge with CPAM. As a result of that, the adsorption between CPAM and sludge particles stopped. When the CPAM was up to 3 mg/g, destabilized colloids could be suspended again, indicating the dosage was excessive and the initial peak was lower.

Herschel–Bulkley model, Casson model, Bingham model, Power-law model, and Newtonian fluid model were all adopted to analyze the flow behavior differences for raw and treated sludge, respectively. Table 1 gives the main parameters for various models. It can be seen that the determined coefficient (R^2) of Newtonian fluid model was minus compared to others, suggesting that sludge suspension belonged

Table 1
Model parameters for CPAM-conditioned sludge

Concentration (mg/g) Model parameters		0	1.0	1.5	2.0	2.5	3.0
Herschel–Bulkley	R^2	0.9990	0.9976	0.9891	0.9976	0.9576	0.6441
	k	0.0252	0.0165	0.3606	0.1966	0.0668	5.906
	n	0.9192	0.9555	0.4712	0.6183	0.7291	9.244×10^{-7}
	τ_0	3.394	4.179	4.4626	4.352	5.418	5.906
Casson	R^2	0.9990	0.9976	0.9892	0.9984	0.9595	0.1976
	k	0.0133	0.0116	0.0882	0.0053	0.048	0.021
	τ_0	3.361	4.163	5.267	4.235	5.206	8.667
Bingham	R^2	0.9985	0.9974	0.9193	0.9835	0.9543	0.5929
	k	0.0151	0.0125	0.0111	0.0165	0.0117	0.0055
	τ_0	3.595	4.267	6.1870	5.825	6.052	5.414
Power-law	R^2	0.9349	0.8673	0.8290	0.9354	0.8466	0.4777
	k	0.7361	1.38	4.1312	2.154	4.153	5.216
	n	0.427	0.3137	0.1503	0.2885	0.1494	0.0534
Newtonian fluid	R^2	0.5762	–0.6097	–2.415	–0.6634	–1.39	–1.889
	k	0.0256	0.0249	0.0292	0.0335	0.0294	0.0214

Note: R^2-correlation coefficient; k-consistency index, Pa·s; n-flow index; τ_0-yield stress, Pa.

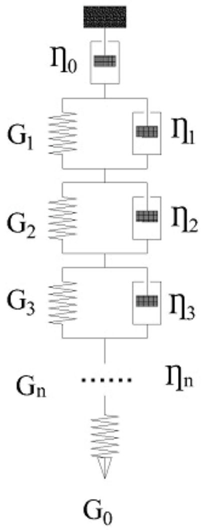

Fig. 1. MGKV model.

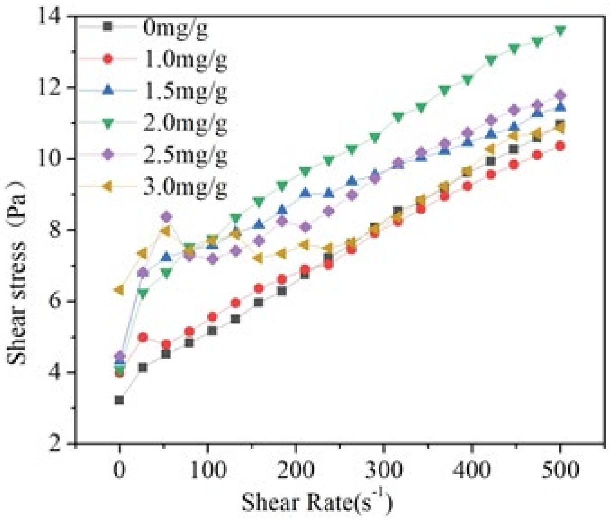

Fig. 2. Impact of flocculant CPAM dosage on a steady shear test.

to non-Newtonian fluid, and could only be described by non-Newtonian model. Herschel–Bulkley model, Casson model and Bingham model have greater superiority (except for 3 mg/g) than the Power-law model. As CPAM was at 3 mg/g, sludge has a different nature, and determined coefficient was about 0.5, manifesting that it could not be described by the above-mentioned rheological models. As shown in Table 1, the yield stress (τ_0) increased as a whole with the increase of CPAM dosage, but the flow index (n) decreased for Herschel–Bulkley, Casson and Bingham models, indicating that non-Newtonian fluid characteristic was improved.

In addition, the relationship between CPAM dosage and dynamic viscosity is shown in Fig. 3. Obviously, as the shear rate achieved 52 s^{-1}, dynamic viscosity decreased sharply from 372 to 0.08 Pa·s (reduced by 99.98%), indicating the significant shear thinning behavior once again. Furthermore, CPAM dosage also has an important effect on the dynamic viscosity. Larger dynamic viscosity was observed at a higher

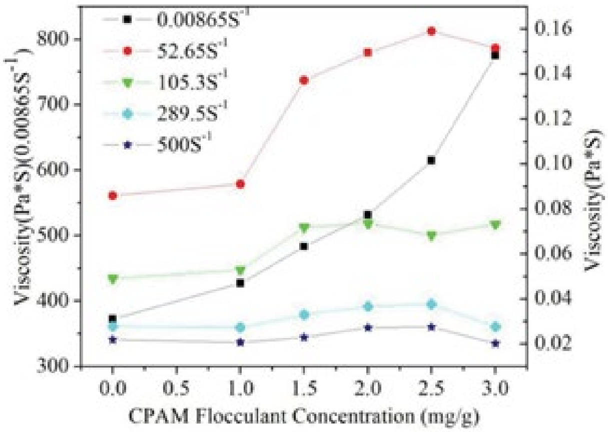

Fig. 3. Impact of flocculant CPAM dosage on viscosity.

dosage. However, as the dosage was up to 3 mg/g, the trend was in contrast, which could deduce that CPAM was excessive, and it was consistent with the above statement.

3.2. Effect of CPAM on thixotropy

Thixotropy was an important rheological phenomenon, which described the variation of material microstructure with time at shearing action [10]. Thixotropy meant that material's viscosity decreased continuously with an increased shear rate or extended shear time, and it was a quite general agreement in the scientific at present community [22]. The main mechanism was the competition between solids interactions and viscous forces as long as no settling happened [38,39].

In the present paper, thixotropic behavior was obtained by the following procedures. At first, shear rate linearly increased from 0 to 500 s^{-1} (duration from 0 to 60 s), and then maintained for 30 s at a shear rate of 500 s^{-1}, and at last decreased from 500 to 0 s^{-1} (duration from 90 to 150 s). At the same time, hysteresis loop was obtained, which was introduced to evaluate the thixotropy for various kinds of sludge. Obviously, as shown in Fig. 4, the hysteresis loop area was small for raw sludge because of its extremely weak floc network. And shear time or shear history has a strong effect on sludge thixotropy. The orientation of particles in the flow state was randomization, which was the same as the result of molecule Brownian motion on the whole. Moreover, research has reported that fractured flocs could reversibly form and even develop into larger sizes in stagnancy, which named as re-flocculate [40]. The dynamic floc structure in sludge could lead to these differences in the hysteresis loops. In the shear-increase step, large flocs would undergo breakage and deformation. And total recovery could not be achieved during durable shearing, as a result of that hysteresis occurred. Interestingly, the hysteresis loop area increased by three times (from 308 to 1231 Pa/s) as CPAM dosage increased from 0 to 3 mg/g, indicating that CPAM promoted sludge thixotropic properties significantly, maybe due to the formation of stable structure for CPAM conditioned sludge.

In addition, yield stress also could be obtained from thixotropy, which mainly showed at the initial and end of shear. While the former took into account inherent inertia of sludge suspensions, resulting in a larger value (especially at the start of shear phase) compared with the real one. Fig. 4 indicates that the yield stress was up to 10 Pa at the start of shear phase, which was larger than that obtained from rheogram. However, at the end of shear phase, the inertia effect was ignored, and the derived yield stress was only around 2 Pa, which was consistent with those of some previous literature [37,41]. Furthermore, Fig. 4 shows that CPAM dosage has a slight effect on yield stress, indicating that yield stress obtained by the hysteresis loop could be an unstable parameter, maybe due to the random destruction of flocs. However, a positive correlation between CPAM dosage and yield stress was observed.

3.3. Effect of CPAM on viscoelasticity

Viscoelastic behavior of materials is crucial to accurately design operational processes [42], and it can be used in computational fluid dynamic simulation. Viscoelastic behavior could be studied by a strain sweep test and creep recovery test.

3.3.1. Strain sweep tests

To accurately describe sludge viscoelasticity, composite modulus G^*, its real part G' and imaginary part G'' were all employed. Composite modulus is referred to as deformation resistance of particle arrangement. The deformation resistance was strong at large value of G^*. The real part G' was

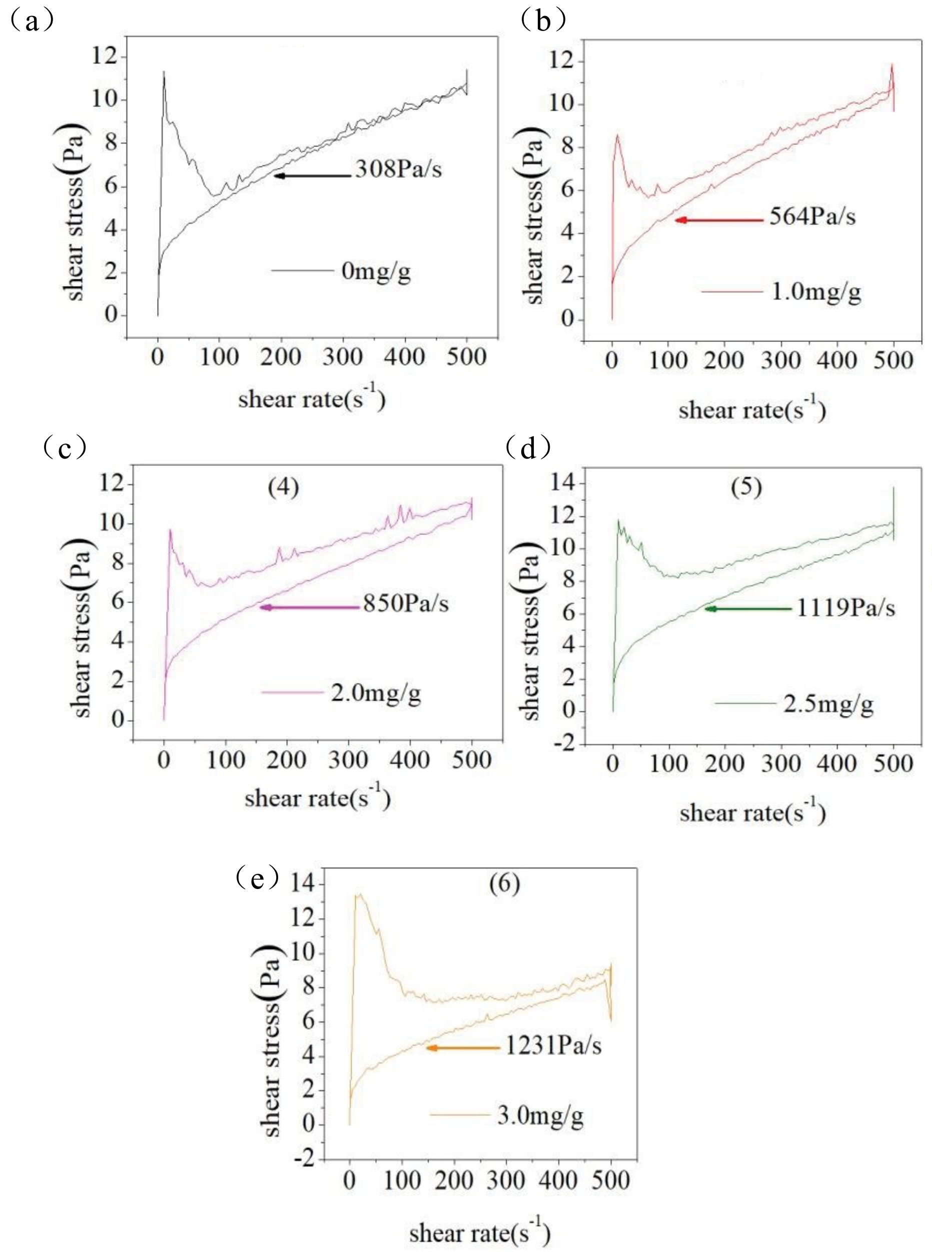

Fig. 4. Impact of CPAM dosage on thixotropy.

determined as storage modulus, expressed by ratio of elastic stress to strain, and the imaginary part G'' was defined as loss modulus, calculated by ratio of viscous stress to strain. Storage modulus expressed the elastic storage capacity during deformation. While loss modulus, also called the viscous modulus, was a measure of dissipation during deformation.

In the linear viscoelastic region, sludge composite modulus (G^*) is shown in Fig. 5. It can be seen that G^* of sludge conditioned with CPAM increased significantly compared with that of raw sludge. At the CPAM dosage of 2.5 mg/g, G^* increased from 67.4 to 186.1 Pa. In contrast, as dosage exceed the above value, G^* decreased to 156 Pa again, which was consisted of Chen's [5]. The polymerization properties of CPAM could provide strong bridge actions and adsorption capacity. As a result, the network strength of floc increased, leading to larger resistance to deformation. However, as the dosage was excessive, the charge reversal of local patches in conditioned sludge occurred. Therefore, the floc network appeared instability, resulting in a smaller value of G^*. It was suggesting that CPAM has an important influence on the physical characteristics of sewage sludge.

Fig. 6 shows the elastic modulus and viscous modulus at a different dosage of CPAM. Obviously, with the increase of flocculant dosage, G' increased significantly in contrast to G'', and the value of G' was much larger, demonstrating that conditioned sludge has distinct solid-like characteristics. Interestingly, as the strain was smaller, the G' was almost independent of the shear strain, which pointed out the range of linear viscoelastic region, and could provide a theoretical basis to accurately design digester in wastewater treatment.

3.3.2. Creep recovery tests

The viscoelasticity of sludge suspensions was also often explained by creep recovery tests, which described the deformation vs. time as the applied stress was at a constant. Fig. 7 shows the relationship between strains and time for raw and CPAM conditioned sludge, which obtained from

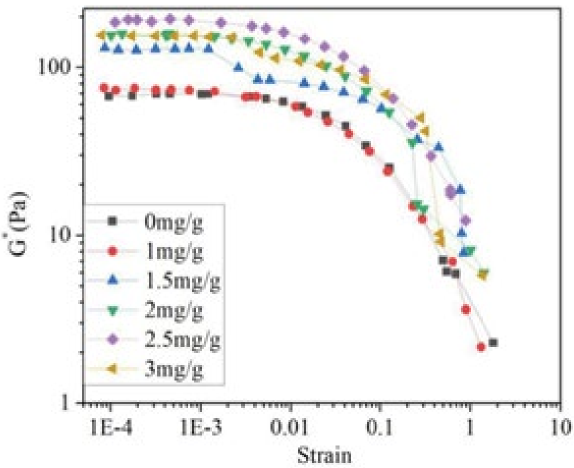

Fig. 5. Impact of CPAM on composite modulus.

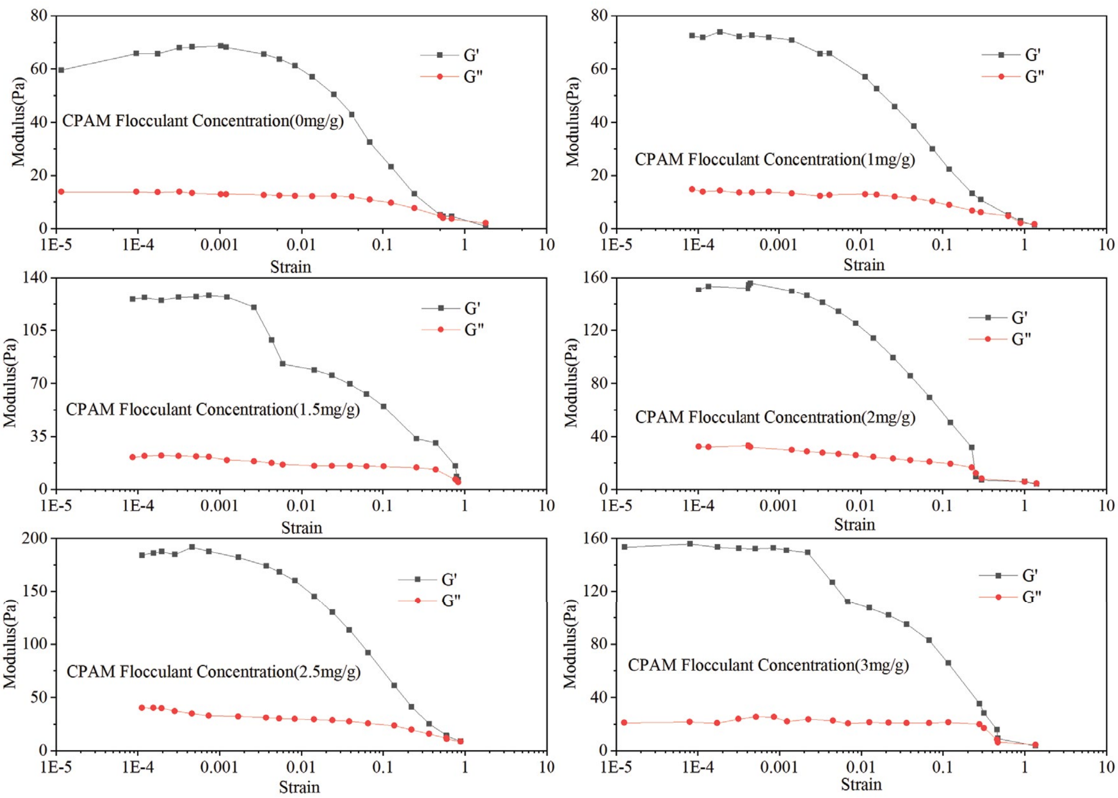

Fig. 6. Impact of CPAM on elastic and viscous modulus.

the creep recovery test at an applied stress of 0.2 Pa. As the applied stress was suddenly removed, the strain decreased abruptly, which reflected the elastic nature for all sludge suspensions, and it could be directly simulated with a spring module in the Maxwell model. The deferred recoverable behavior could be modeled by a series of Kelvin–Voigt modules. Obviously, the Kelvin–Voigt solid nature of sludge suspensions has a dominant advantage during creep recovery tests. Also, the part that could not be recovered was determined as the viscosity ratio. It can be seen that the reduction for all kinds of sludge suspensions was irregular variation. However, for raw sludge, the minimum viscosity ratio was observed, testifying its weak elasticity again. In addition, CPAM has an inhibitory effect on sludge suspensions recovery from Fig. 7. It was interesting that the strain at an initial stage of creep was always less than that of recovery for all sludge suspensions, maybe due to the viscous effect.

The MGKV model (with three function units) was developed to describe the creep recovery of sludge suspensions, and the fitted parameters are shown in Table 2. The higher determined coefficient ($R^2 > 0.9$) indicated that the viscoelastic behavior of sludge suspensions could be well predicted by MGKV model. Amazedly, part function exponent was negative, maybe due to the inhomogeneity of flocs or particles, indicating that micro-mechanism were needed to be analyzed in depth because of limited rheology theory. In addition, CPAM reduced the delay time (λ_1, λ_2 and λ_3) greatly, especially λ_1, which was smaller than λ_2 and λ_3, meaning that λ_1 had a dominance during the entire creep recovery of sludge suspensions. With the increment of CPAM dosage, λ_1 decreased significantly. As the dosage was 1 mg/g, λ_1 reduced by 90% (from 338 to 31 s). Smaller λ_1 explicated that conditioned sludge could restore to another stable status within a shorter time. Furthermore, a reasonable creep

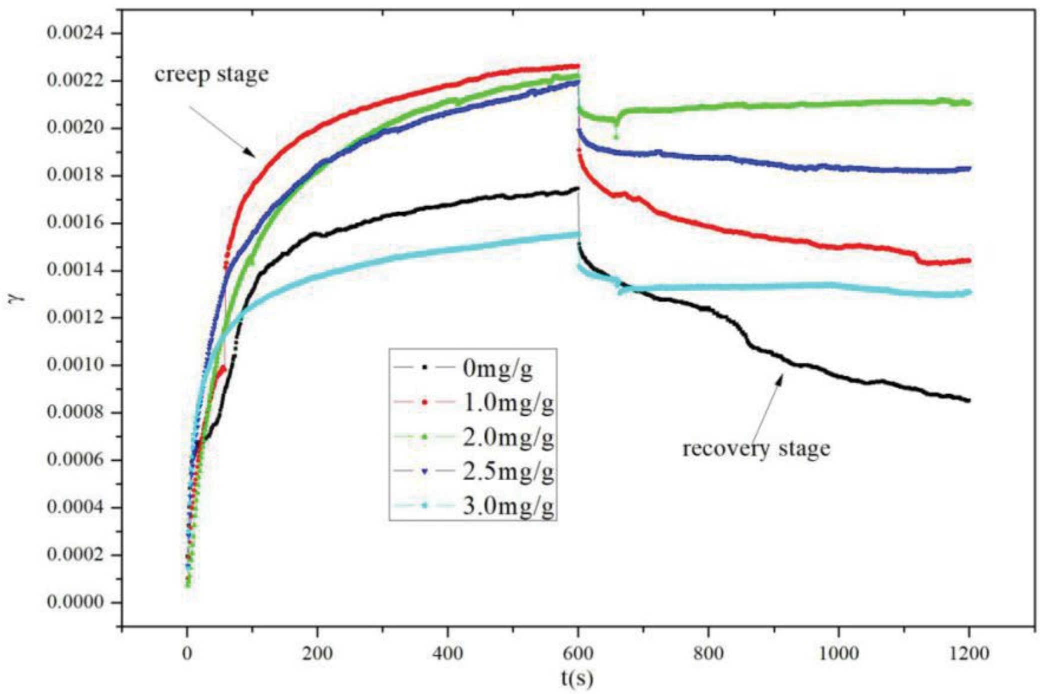

Fig. 7. Impact of CPAM flocculant dosage on creep recovery tests.

Table 2
Fitting parameters for MGKV model (Eq. (1)) describing creep recovery response of raw and CPAM conditioned sludge suspensions

Dosage	0 mg/g	1 mg/g	2 mg/g	2.5 mg/g	3 mg/g
a	–0.0054	0.0012	0.0021	0.0017	0.0012
b	–0.0361	-6.7577×10^{-4}	-7.9081×10^{-4}	-7.5206×10^{-4}	-7.5894×10^{-4}
λ_1	338.3109	31.2596	24.4808	15.6978	9.2071
c	0.0361	–0.0162	–0.0329	–0.0421	–0.0256
λ_2	373.2075	261.1774	208.2812	287.8494	276.0668
d	0.0057	0.0158	0.0316	0.0414	0.0252
λ_3	285.23	318.9143	213.3264	299.5107	286.7770
R^2	0.9523	0.9102	0.9895	0.9567	0.9173

recovery time could be calculated by the MGKV model, so as to accurately analyze rheological data for better-conducting sludge treatment.

4. Conclusion

The current work got an insight into rheology of sludge conditioned by flocculants, all the rheology characteristics including liquid, thixotropy and viscoelastic behavior were systematically measured and assessed. Specific conclusions were as follows.

- Steady tests showed prominent shear thinning was observed for all sludge suspensions before and after conditioning with CPAM, and all the samples except for sludge conditioned with CPAM of 3 mg/g, could be accurately described by Herschel–Bulkley, Bingham and Casson equations.
- Thixotropy of raw sludge suspension was extremely weak, which could be significantly improved by CPAM. As CPAM dosage increased from 0 to 3 mg/g, the area of thixotropy loop increased by 3 times, maybe due to the stable structure formation in CPAM conditioned sludge. In addition, yield stress has an effect on thixotropy. A suitable test method for yield stress should be further studied due to the disadvantages of current measurement methods.
- The strong bridge actions and adsorption capacity of CPAM could form floc network with larger strength, resulting in larger resistance to deformation. While, as the CPAM achieved at 3 mg/g, the strength of the floc network was impaired due to the destabilized colloids. As a consequence, complex and elastic modulus all decreased in strain sweep tests.
- MGKV model was developed, which was composed of Maxwell and a series of Kelvin–Voigt modules. As a result of that MGKV could accurately characterize the creep recovery properties for sludge suspensions, and delay time λ_1 had a dominant impact.

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