

Effects of thermal hydrolysis temperature on physical characteristics of municipal sludge

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ABSTRACT

Effects of thermal hydrolysis temperature on the physical properties of municipal sludge was further studied by a series of experiments. There was a decrease in bound water content with an increase in hydrolysis temperature, while there was an increase in pH at temperatures below 120 °C, and a decrease at temperatures exceeding 120 °C. An analysis of settleability, centrifugation and vacuum filtration of the treated sludge indicated that the threshold temperature was 120 °C, which was the same as the temperature for the bound water content and particle size. In addition, raw sludge with a solids content of 100 g/L, exhibited significant non-Newtonian fluid characteristics. At thermal hydrolysis temperatures exceeding 120 °C, non-Newtonian fluid characteristics including liquid and solid characteristics were significantly weakened. The consistency index (k) decreased from 5.90 Pa·s to 0.068 Pa·s, while the flow index (n) increased from 0.31 to 0.74, suggesting that thermal hydrolysis sludge was much closer to Newtonian fluids compared to raw sludge. Modification of bound water content, particle size and viscosity with hydrolysis temperature, revealed the nature of improved dewaterability by thermal hydrolysis. The fractal dimension of the sludge floc increased from 2.74 to 2.90, meaning that the floc became more compact after thermal hydrolysis.

Key words | municipal sludge, physical characteristics, thermal hydrolysis

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INTRODUCTION

In 2011, the number of municipal sludge treatment plants had increased to about 2,739, and 175 million tons of municipal solid waste was produced in China (Hai *et al.* 2012). Municipal sludge commonly contains large amounts of water (about 97%, including free water, interstitial water and bound water), microorganisms and mineral components (Baudez *et al.* 2011). Due to the increased costs of sludge management and stringent environmental legislation, sludge reduction is becoming one of the most serious challenges.

Anaerobic digestion and dewatering are the important methods for minimizing sludge volume. Via anaerobic digestion, a proportion of organic matter in the sludge could be converted into biogas, resulting in sludge stabilization. The dewatering methods usually include mechanical dewatering, thermal press dewatering, thermal drying and electroosmotic dewatering. Mechanical dewatering has been extensively used owing to its simple operation and low energy consumption (Ayol & Muslu 2012). However, sludge is difficult to dewater owing to its complex nature, and mechanical

dewatering is near impossible without conditioning (Zall *et al.* 1987; Thapa *et al.* 2009; Feng *et al.* 2014a). Therefore, various methods, such as chemical conditioning and disintegration techniques, have been introduced for changing the floc structure, enhancing the solubility of sludge solids, reducing the pathogens and improving the sludge dewaterability. Chemical conditioning, usually including organic or inorganic flocculation, acidification or alkalization and consecutive oxidation by H₂O₂ (Vosteen & Weissenberg 2000), could change the properties of the sludge solid (such as its surface charge and particle size), and as a result, a significant improvement in solid-liquid separation is obtained. However, organic and inorganic flocculation, is based on electrical neutralization and bridging, which usually acts between colloidal particles in solutions rather than in the interior of the particles. Therefore, organic or inorganic flocculation has little effect on the bound water content of sludge. In order to decrease bound water content and enhance the hydrolysis rate of organic matter, disintegration techniques (such as mechanical methods, thermal hydrolysis,

freezing and thawing, and biological methods) are necessary, during which thermal hydrolysis is considered as the most promising method for accelerating the hydrolysis rate of organic matter and promoting biogas production (Li & Noike 1992; Müller 2001; Neyens & Baeyens 2003; Bougrier *et al.* 2008).

Extensive research has been carried out to study the effect of thermal hydrolysis on biogas production during anaerobic digestion, on chemical characteristics (such as carbohydrates solubilization or proteins solubilization) and on sludge dewaterability (Neyens & Baeyens 2003; Ma *et al.* 2011; Feng *et al.* 2014b). Several works have focused on the effect of thermal hydrolysis (temperature was lower than 105 °C) on sludge rheological characteristics (Lin & Shien 2001; Hammadi *et al.* 2012; Baudez *et al.* 2013). Verma *et al.* (2007) reported variations in sludge pseudoplasticity, as the solids concentration ranged from 10 g/L to 50 g/L after thermal-alkaline treatment (temperature: 121 °C, pH:10.25) and did not refer to the change of viscoelasticity.

However, papers on the correlation between the physical characteristics of sludge (such as particle size, bound water content, rheological behavior) and thermal hydrolysis temperature are few. Therefore, in the present paper, the particle size, bound water content, dewaterability, rheological behavior and fractal dimension of municipal sludge at different thermal hydrolysis temperatures were studied in detail, which revealed the nature of improved dewaterability by thermal hydrolysis. The analysis of sludge rheological behavior have provided a theoretical basis for the design and operation of heat exchangers, transport facilities and pumping in municipal sludge treatment plants.

MATERIAL AND METHODS

Sludge sample

Activated sludge was sampled from the discharge of a horizontal spiral filter centrifuge in a municipal wastewater treatment plant in Zibo, China, and called raw sludge. Afterwards, the raw sludge was stored at 4 °C to limit the impact of biological activity.

To study the effect of thermal hydrolysis temperature on the physical characteristics of sludge, the raw sludge of 1.4 L was batch treated in a high-pressure reactor (effective volume: 2 L) at different temperatures for 60 min. The pressure of 2 MPa in the reactor was supplied by nitrogen to prevent water evaporation, reaction between air and organic matter in the sludge. After thermal hydrolysis, the sludge, now known as treated sludge, was cooled by freshwater inside a tube in the reactor, and then was used for the following tests.

Physical characteristic analysis

Total suspended solids content (TSS) and volatile suspended solids content (VSS) were measured by a series of tests according to China's standard for municipal sludge analysis (CJ/T 221-2005). The particle size and specific surface area of sludge floc were measured by Mastersize 2000, while the calorific value of sewage sludge (dry basis) was obtained by GEME-IKAC2000. The bound water content (bound water mass /dry solid mass in sludge) was measured by a differential scanning calorimeter (Mettler Toledo, DSC1/700), which was based on the assumption that bound water did not freeze at -20 °C (Vaxelaire & Cézac 2004). Specific measurement procedures were as follows: (1) a sludge sample of about 10 mg was placed in a DSC measurement device; (2) it was then cooled from 20 °C to -20 °C at a cooling rate of 2 °C/min, and the released heat during water freezing was recorded. Because bound water is unfreezable at -20 °C, the heat released during measurement was proportional to the free water content. Then the bound water content was obtained by the difference between the total water content and the free water content. Each measurement was carried out twice, and the average value was introduced. The physical characteristics and relative errors are listed in Table 1.

Rheological test

A commercial rheometer (DHR-2) equipped with concentric cylinder geometry (cup diameter: 30.39 mm; bob diameter: 27.98 mm; length: 41.90 mm) was used to study rheological

Table 1 | Physical characteristics of raw sludge

Sludge types	TSS (%)	VSS/TSS (%)	COD(g/L)	BW (g/g)	D (μ m)	S (m ² /g)	pH	Calorific value (MJ/kg)
Raw sludge	21.3 ± 0.11	47.9 ± 0.3	1.9 ± 0.08	1.45 ± 0.10	52.1 ± 0.9	0.50 ± 0.01	7.50 ± 0.04	10.8 ± 0.4

COD – chemical oxygen demand; BW – bound water content; D – particle size of sludge floc; S – specific surface area of sludge floc.

properties of the raw and treated sludge. To avoid water evaporation during measurement, a plastic ring was fitted around the measuring geometry. Steady and dynamic tests were carried out to analyze the liquid and solid characteristics of the raw and treated sludge. During steady tests, the shear rate logarithmically increased from 0.01 to 400 s⁻¹ to obtain the viscosity and rheogram of sludge. The dynamic rheological test, also called the ‘dynamic mechanical test’ or the ‘oscillatory test’, has been proven to be a more effective method for the viscoelastic measurement of sludge (Ayol et al. 2006). In this study, shear strain sweep tests were carried out to investigate the effect of different thermal hydrolysis temperatures on sludge viscoelasticity.

Before each test, the sludge was screened by a sieve with a pore diameter of 0.6 mm to diminish errors caused by large particles. After loading, the sludge was left at rest for 1 min, which allowed us to eliminate the effect of loading shear. Each rheological test was carried out twice at room temperature, and the average value was introduced.

Dewaterability test

In order to ensure the smooth progression of dewatering experiments (centrifugation, sedimentation and vacuum filtration) involving the raw and treated sludge, all the sludge employed in the experiments were diluted to 5.4 wt%. In centrifugal experiment, samples with volume of 30 mL were collected in centrifuge tubes, where the centrifugal force and centrifugation time were 3,000 g and 10 min, respectively. For the settlement experiment, sludge samples of 100 mL were put in several measuring cylinders. As the supernatant increased for every 1 mL, the setting time was recorded. During vacuum filtration, samples with a mass of 150 g were employed, and the filterability was evaluated

by specific resistance to filtration (SRF) which was calculated using Ruth’s equation. After dewatering tests, the solids content of the dewatered sludge (filter cake) was measured. Each dewatering test was carried out twice at room temperature, and the average value was introduced.

RESULTS AND DISCUSSION

Effects of thermal hydrolysis on the physical characteristics of sludge

Sludge thermal hydrolysis involves two processes: the dissolution of suspended organic solids and the hydrolysis of macromolecular components (including proteins, polysaccharides and lipids). During thermal hydrolysis, the organic component of solids split into short-chain fragments, and then partially dissolves into the liquid phase. Therefore, the mean volume diameter of particle size decreases gradually with an increase in thermal hydrolysis temperature, while the reverse occurs for the specific surface area of sludge particles (or floc) (Figure 1(a)).

Due to the presence of solids, water within the sludge is generally separated into two categories: free water and bound water. Free water, consisting of the largest part of the sludge, behaves as pure water thermodynamically, and is not associated with the suspended solid particles, which can be eliminated by the application of low mechanical force. However, the term ‘bound water’ does not have a uniform definition, and its operational definition depends upon the measurement method for the bound water (Erdincler & Vesilind 2000). In this study, the DSC method was introduced, therefore, bound water was defined as ‘unfreezable water’, usually containing vicinal water and hydration water. Vicinal

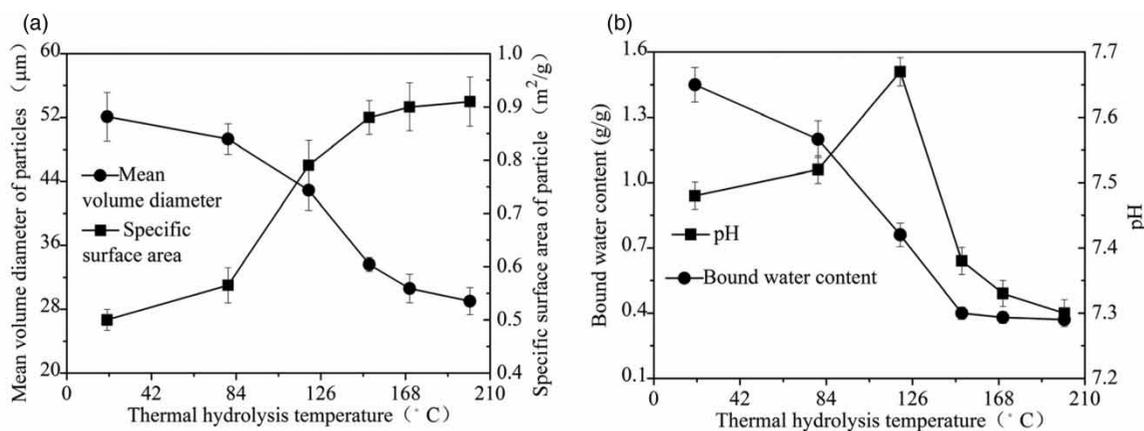


Figure 1 | Physical characteristics of sludge at different thermal hydrolysis temperatures: (a) particle characteristics; and (b) bound water content and pH.

water is held tightly to the particle surface and within cells, as long as it is associated with a solid surface (Vesilind 1994). The proportion of bound water is minimal compared to the total water contained in the sludge, but it is generally greater compared to the solid phase.

As shown in Figure 1(b), the bound water content decreased by about 50% at 120 °C (from 1.45 to 0.76 g/g), and when the temperature was higher than 170 °C, the rate at which the decrease occurred lessened. After thermal hydrolysis, a large amount of organic matter (proteins, polysaccharides, lipids and other intracellular macromolecules) in sludge cells were disrupted and hydrolyzed. Consequently, the bound water, originally trapped inside sludge cells, was released to free water, and the overall free water content increased.

Figure 1(b) also shows the variation of pH at different thermal hydrolysis temperature. The value of the pH first slightly increased from 7.48 to 7.65 at 120 °C, which may be due to the volatilization of acidic matter. Then, the pH decreased with temperature, which could be attributed to the degradation of macromolecules. Proteins could degrade into amino acids and lipids could hydrolyze into low chain fatty acids.

Effects of thermal hydrolysis on the rheological characteristics of sludge

Usually, rheological behavior of sludge mainly contain two types: a liquid characteristic, determined by viscosity; and a solid characteristic (evaluated by yield stress or storage modulus).

Effect of thermal hydrolysis on liquid characteristic of sludge

Sludge viscosity, determined by the ratio of shear stress to shear rate, describes a liquid characteristic, and is a measure of the resistance generated by the movement between two adjacent layers of a fluid.

Figure 2 indicates the rheogram of sludge with a solids concentration of 100 g/L. The sludge, treated at different temperatures, still displayed shear-thinning behavior. However, the relationship between the shear rate and shear stress changed gradually with an increase in temperature, which suggested a variation in organic substances after thermal hydrolysis. At a temperature of 80 °C, the solid organic matter in the sludge dissolved slightly and its physical characteristics were modified slightly, hence the rheological curve was similar with the raw sludge. With an increase in

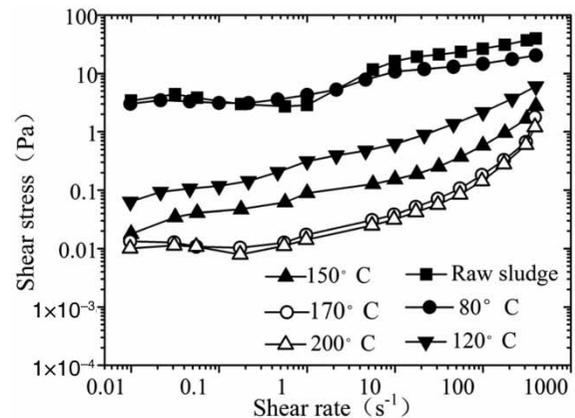


Figure 2 | Flow curve of 10% municipal sludge at different hydrolysis temperatures.

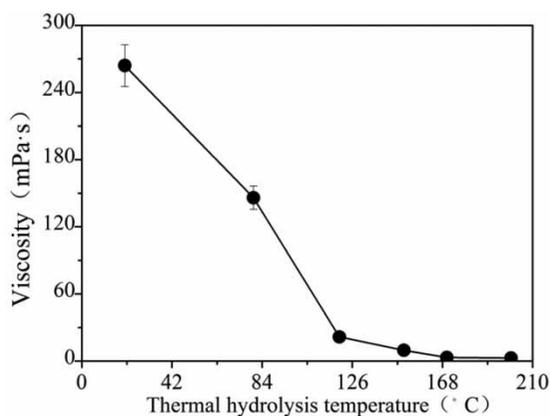
temperature, the organic component hydrolyzed, leading to weakened non-Newtonian characteristics of the sludge.

To accurately discuss the difference between the raw and treated sludge, Bingham, Power law, Herschel–Bulkley and Newtonian fluid models were employed to fit these rheograms. The results (shown in Table 2) indicated that the power law model was more suitable than other models when the temperature was lower than 120 °C for the sludge with a solids content of 100 g/L. When the treated temperature reached 120 °C, the relationship between shear stress and shear rate of the treated sludge could be accurately described by the Newtonian fluid model. Note that the rheology data of treated sludge at higher temperature presented curve apparently at very low shear rate on a log–log coordinate. However, the deviation between curve and straight line caused by the minute rheology data is negligible. Therefore, the Newtonian fluid model is also appropriate for the treated sludge at higher temperature. Furthermore, with an increase in temperature, n increased from 0.268 to 1, which clearly indicated that the treated sludge was much closer to the Newtonian fluid. Due to the existence of extra polymeric substances (EPS) in the raw sludge, electrostatic and gel-like interactions were closely related to non-Newtonian behavior. However, after hydrolysis, the colloidal properties of macromolecules were destroyed, corresponding to better fluidity.

Figure 3 shows the evolution of sludge viscosity (at a shear rate of 100 s⁻¹) with thermal hydrolysis temperature, illustrating that when the temperature reached 120 °C, sludge viscosity decreased by 91.8% (from 264 to 21.6 mPa·s), and when the temperature was higher than 170 °C, the sludge viscosity changed slightly. The rule of viscosity change suggested that a higher temperature (higher than 170 °C) had little effect on the colloidal properties of the sludge.

Table 2 | Rheological model parameters at different thermal hydrolysis temperatures

Hydrolysis temperature (°C)		Raw sludge	80	120	150	170	200
Models							
H-B	R^2	0.951	0.972	0.997	0.805	0.969	0.907
	k	5.90	5.039	0.068	0.004	0.004	0.002
	n	0.312	0.230	0.741	1	1	1
	τ_0	3.66	0.455	0.164	0.053	0.003	0
Bingham	R^2	0.741	0.666	0.981	0.972	0.971	0.913
	k	0.095	0.044	0.015	0.006	0.004	0.002
	τ_0	8.04	6.00	0.316	0.040	0.003	0
Power-law	R^2	0.956	0.974	0.992	0.969	0.971	0.914
	k	6.40	5.51	0.107	0.006	0.004	0.002
	n	0.268	0.218	0.668	1	1	1
Newtonian fluid	R^2	0.310	—	0.952	0.972	0.973	0.923
	k	0.124	—	0.016	0.006	0.004	0.002

**Figure 3** | Relationship between thermal hydrolysis temperature and viscosity.

Effect of thermal hydrolysis on the solid characteristics of sludge

Viscoelastic material means that they can flow (viscous characteristic), but when the applied stress reduces to zero, a partial elastic recovery is observed which may be related to the storage of elastic energy in inter-particle bounds (elastic characteristic) (Seyssiecq *et al.* 2003). Usually, three parameters are used to describe the material viscoelasticity: complex modulus (G^*), its real part G' (storage modulus, ratio of elastic stress over strain), and its imaginary part G'' (loss modulus, ratio of viscous stress over strain). Storage modulus and loss modulus (also called viscous modulus) represent elastic storage capacity and dissipation during deformation, respectively. Municipal

sludge exhibits viscoelastic properties, which could be investigated by shear strain sweep test.

The evolution of viscoelastic parameters with shear strain at a constant frequency of 1 Hz is shown in Figure 4. Storage and viscous modulus both decreased with an increase in temperature: from about 90 and 19 Pa (raw sludge) to 0.03 and 0.02 Pa (sludge treated at 170 °C), respectively, indicating that more energy could be stored in the floc structure for the raw sludge when the shear stress was removed. Furthermore, the critical shear strain for the raw sludge (about 70%) was much higher than that of sludge treated at 170 °C (about 9%). In raw sludge, organic matter tends to aggregate to form a three-dimensional gel-like biofilm matrix, and EPS interact with water in a manner similar to gels (Keiding *et al.* 2001). Therefore, a larger amount of energy was needed to cleave the network to achieve a stable state. However, EPS and other organic substances were destroyed after thermal hydrolysis, leading to the disruption of floc structure and the reduction of attractive forces among particles.

Effects of thermal hydrolysis on the fractal dimension of sludge

Sanin (2002) pointed out that the fluid behavior of non-Newtonian fluids is due to the colloidal properties of suspension. Fractal dimension is usually used to describe the complex irregular structure of floc. Shih *et al.* (1990) proposed a method to calculate quality fractal dimension of colloid structures. The relationship between the elastic constant of the gel structure (including store modulus,

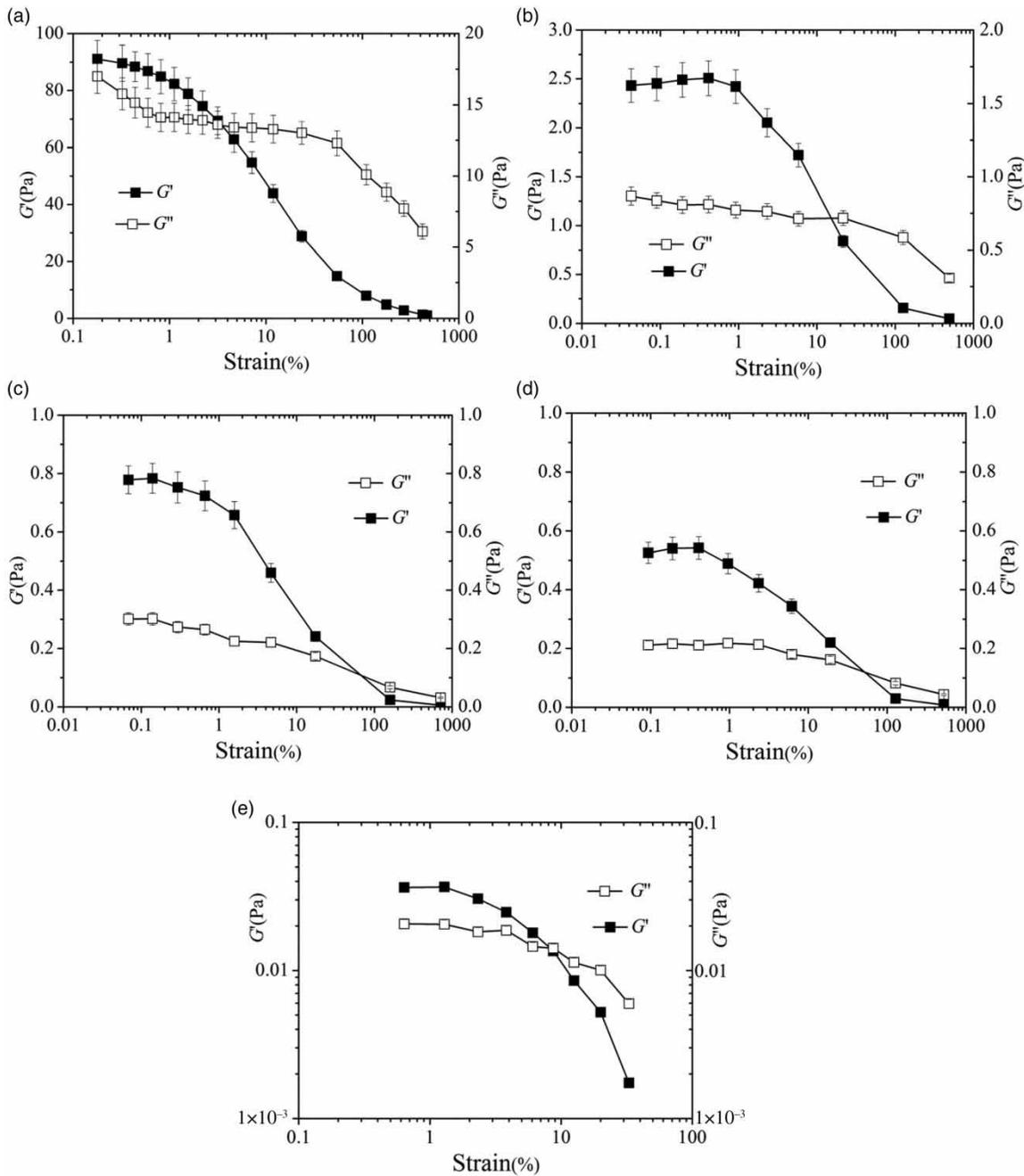


Figure 4 | Relationship between viscoelasticity and shear strain at different thermal hydrolysis temperatures: (a) raw sludge; (b) thermal hydrolysis at 80 °C; (c) thermal hydrolysis at 120 °C; (d) thermal hydrolysis at 150 °C; and (e) thermal hydrolysis at 170 °C.

critical shear strain and limiting viscosity) and sludge concentration, can be represented by the following equations:

$$K \propto \phi^{1/(d-d_f)} \quad (1)$$

$$\gamma_0 \propto \phi^{1/(d-d_f)} \quad (2)$$

where K is the elastic constant, d is the Euclidean dimension and d_f is the fractal dimension of the floc. According to the double logarithmic curve analysis (correlation between elastic constants and sludge concentration), the quality fractal dimension of colloid structures can be determined. In this study, the relationships between the storage modulus,

limiting viscosity and solids concentration were introduced to estimate the floc structure of the raw and treated sludge.

Figures 5 and 6 illustrate the correlations between storage modulus, limiting viscosity and solids concentration of the raw and the treated sludge, respectively. Results showed that $\lg G'$ and $\lg \mu$ were in direct proportion to $\lg \varphi$ (φ : solids concentration of sludge), and the values of the determination coefficient (R^2) were more than 0.95. The fractal dimension was 2.82 and 2.88 for the raw and treated sludge by the relationship between the storage modulus and solids concentration, respectively, while the fractal dimension was 2.74 and 2.90 by the relationship between limiting viscosity and solids concentration. The reduction of the fractal dimension of the floc structure after thermal hydrolysis, indicated the floc structure of treated sludge was more compact than that of the raw sludge. In raw sludge, the flocs, formed by the interaction of adsorption bridging among divalent metal cation and microbes, were broken up, leading to the reduction of floc

size. As a result, the fractal dimension for treated sludge was larger than that of raw sludge.

Effects of thermal hydrolysis on sludge dewaterability

Sludge dewaterability is one of the greatest problems in a sewage treatment plant. Moisture distribution within the sludge has always been considered essential for the examination of the dewatering problem.

Figure 7 shows sludge dewaterability (settlement, centrifugation, vacuum filtration) treated at different thermal hydrolysis temperatures. The theory of sedimentation indicates that the fall speed of particles is proportional to particle density and inversely proportional to suspension viscosity. After thermal hydrolysis, the sludge viscosity decreased, and the fractal dimension increased, indicating that settleability could be improved. When it was higher than 150 °C, settleability improved gradually. Furthermore,

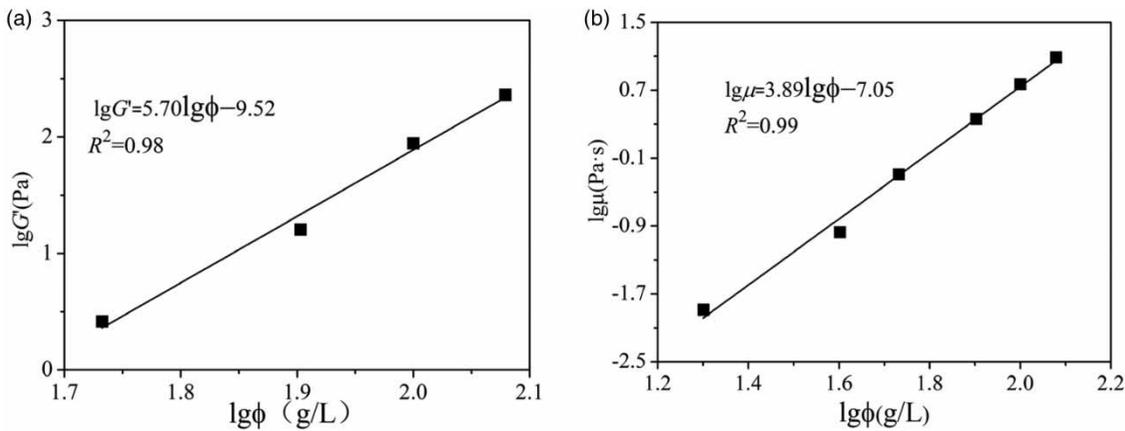


Figure 5 | Relationship between elastic constant and solids concentration of the raw sludge: (a) G' versus solids concentration; and (b) μ versus solids concentration.

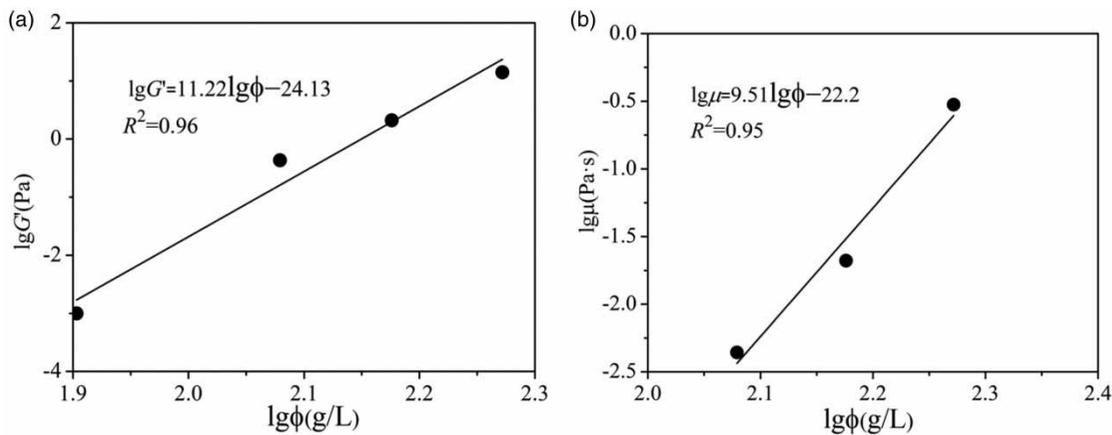


Figure 6 | Relationship between elastic constant and solids concentration of treated sludge: (a) G' versus solids concentration; and (b) μ versus solids concentration.

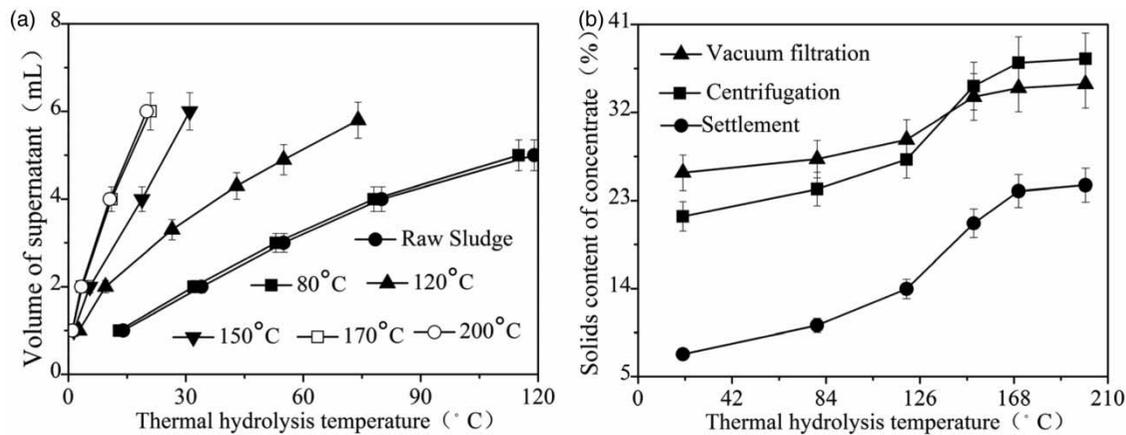


Figure 7 | Sludge dewaterability at different thermal hydrolysis temperature: (a) settlement curve; and (b) solids content of dewatered sludge for different dewatering methods.

after hydrolysis at higher temperatures, the color of the supernatant became dark brown, which may be as a result of the polycondensation reaction between amino and aldehyde groups.

The behavior of a water molecule during the dewatering process is widely dependent on its proximity to solids. Generally, free water can be totally removed by mechanical force. However, bound water is almost impossible to remove using the same method. Hence, to a certain extent, the bound water content of sludge determines the limit of the filter cake moisture content. Thermal hydrolysis compels the bound water to be released, resulting in the increase of free water content. Therefore, a drier sludge cake was obtained for the treated sludge. In order to study the variation of sludge filterability, SRF was calculated and is shown in Figure 8. Due to the reduction of viscosity, particle size and compressibility, the SRF decreased with the increase of hydrolysis temperature. As a result, the filterability of sludge was improved. It seemed that 120 °C was the temperature

threshold for improving sludge dewaterability, and as the temperature exceeded 170 °C, the tendency for change became gradual, both of which were similar to that of other physical characteristics.

CONCLUSIONS

Thermal hydrolysis is an important pretreatment method for improving sludge dewaterability, by modifying the physical characteristics of sludge, including its bound water content, rheology and fractal dimension. Usually, municipal sludge is a colloidal system and contains significant numbers of microorganisms (such as proteins, polysaccharides and lipids) and inorganic particles. Due to the different degradation temperatures of these organic substances, the physical characteristics of sludge differ at various thermal hydrolysis temperatures. With the increase of temperature, particle size, bound water content and fractal dimension reduced by about 44%, 79% and 7%, respectively, suggesting the breakup of sludge floc and the degradation of organic macromolecules. Therefore, the non-Newtonian fluid characteristics of the sludge were weakened. When the thermal hydrolysis temperature exceeded 120 °C, the treated sludge with a solids content of 100 g/L, could be described by the Newtonian fluid model. Due to the reduction of bound water, viscosity, and compressibility, the dewaterability of the sludge improved.

Furthermore, this paper provides new insight into the rheological behavior of treated sludge at different thermal hydrolysis temperatures and provides reliable flow property parameters to engineers for accurately designing the translated pipe, pump, and heat exchanger. These fluid models are especially useful in hydrodynamic optimization and computational fluid dynamic (CFD) simulation.

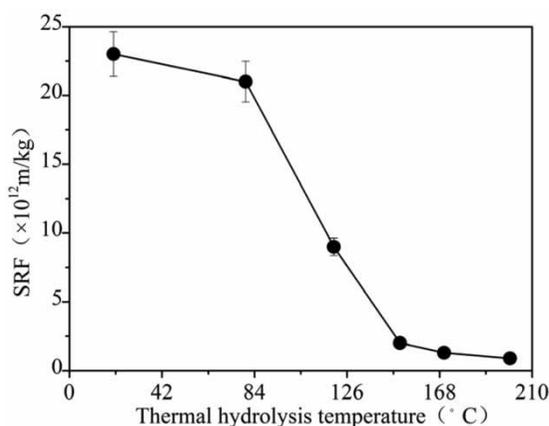


Figure 8 | SRF of treated sludge at different thermal hydrolysis temperatures.

However, the thermal hydrolysis temperature is usually higher than 80 °C, the dewatering equipment and replacement parts should be manufactured using heat resistance materials, resulting in high maintenance costs. Moreover, a large amount of biogas was produced during this pretreatment. Consequently, additional research should be carried out to evaluate economic costs and the energy balance of the thermal hydrolysis process.

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