Improving the sludge disintegration efficiency of sonication by combining with alkalization and thermal pre-treatment methods

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ABSTRACT

One of the most serious problems encountered in biological wastewater treatment processes is the production of waste activated sludge (WAS). Sonication, which is an energy-intensive process, is the most powerful sludge pre-treatment method. Due to lack of information about the combined pre-treatment methods of sonication, the combined pre-treatment methods were investigated and it was aimed to improve the disintegration efficiency of sonication by combining sonication with alkalization and thermal pre-treatment methods in this study. The process performances were evaluated based on the quantities of increases in soluble chemical oxygen demand (COD), protein and carbohydrate. The releases of soluble COD, carbohydrate and protein by the combined methods were higher than those by sonication, alkalization and thermal pre-treatment alone. Degrees of sludge disintegration in various options of sonication were in the following descending order: sono-alkalization > sono-thermal pre-treatment > sonication. Therefore, it was determined that combining sonication with alkalization significantly improved the sludge disintegration and decreased the required energy to reach the same yield by sonication. In addition, effects on sludge settleability and dewaterability and kinetic mathematical modelling of pre-treatment performances of these methods were investigated. It was proven that the proposed model accurately predicted the efficiencies of ultrasonic pre-treatment methods.

Key words | alkalization, combined pre-treatment, sludge disintegration, sonication, thermal pre-treatment, waste activated sludge

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INTRODUCTION

Huge amounts of waste activated sludge (WAS) are inevitably produced in activated sludge processes, during the treatment of industrial and municipal wastewaters. The WAS has to be treated prior to final disposal, due to its highly putrescible nature. However, its treatment is the major economic problem in the treatment plants. At present, aerobic and anaerobic sludge digesters are mostly used to digest the WAS in many treatment plants. But the extremely slow hydrolysis of WAS limits the efficiency of the digesters and therefore larger digester volumes are needed due to long hydraulic retention times. To accelerate the rate limiting hydrolysis and to improve the digester performance, some pre-treatment methods such as acidification (Chen *et al.* 2007), alkalization (Torres & Llorens 2008), Fenton oxidation (Erden & Filibeli 2011), microwave irradiation (Eskicioglu *et al.* 2007), ozone pre-treatment (Lahnsteiner & Vranitzky 2010), sonication (Luning *et al.* 2008; Şahinkaya & Sevimli 2009), and thermal pre-treatment (Phothilangka *et al.* 2008) can be applied before the sludge digestion.

Alkalization is one of the most effective chemical sludge pre-treatment methods. Its disintegration mechanism is based on the disruption of sludge flocs, destruction of cell walls and membranes by hydroxyl anions (OH^-) and transfer of extracellular and intracellular polymeric substances from the solid phase into the aqueous phase (Neyens *et al.* 2003). This pre-treatment method has important advantages such as easy operation, simple device and high efficiency. Thus, alkalization has been recently combined with other sludge disintegration methods such as microwave irradiation (Doğan & Sanin 2009), thermal pre-treatment

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(Vlyssides & Karlis 2004), and sonication (Kim et al. 2010; Li et al. 2010) to achieve a higher degree of sludge disintegration.

Thermal pre-treatment, which is a physical method, is applied in the temperature range from 40 to 180 °C (Neyens et al. 2003). It causes the degradation of sludge gel structure and release of organic matter in the sludge flocs. This method can be applied at low (<100 °C) and high (>100 °C) temperatures. The main operating parameter in thermal pre-treatment is temperature. As temperature rises, the sludge disintegration increases. However, the very high energy consumption to heat the huge amounts of WAS is the major disadvantage of this method. In addition, recalcitrant soluble organics and toxic/inhibitory intermediates are generated at very high temperatures (>180 °C) as a result of Maillard reactions. Therefore, the biodegradability of sludge decreases (Wilson & Novak 2009).

Sonication is the most powerful method, which effectively disintegrates the WAS even in short pre-treatment times. For this reason, it is successfully applied in some treatment plants (Luning et al. 2008). It is based on the application of ultrasonic irradiation to the WAS. When the sludge is exposed to ultrasonic irradiation; hydro-mechanical shear forces, which are the predominant mechanism in sludge disintegration (Pilli et al. 2011), are generated as a result of acoustic cavitation. These forces disrupt the sludge flocs, break out the cell walls and membranes, and finally release the intracellular and extracellular organic substances into the aqueous phase of sludge. However, the major disadvantage of sonication is its high energy requirement.

The combined sludge pre-treatment methods are rather new and aim to improve the sludge disintegration performance by using synergistic effects of different disintegration mechanisms. The combination of sonication with other methods such as alkalization and acidification has been scarcely investigated so far. However, although thermal pre-treatment of sludge was extensively examined, there has not been any study on the sludge disintegration by the combination of sonication and thermal pre-treatment methods in the literature up to now. Therefore, we proposed to enhance the performance of the ultrasonic sludge disintegration method by using it together with alkaline and thermal pre-treatment methods and to compare the disintegration efficiencies of these methods based on chemical parameters. In this study, the individual effects of alkalization, thermal pre-treatment and sonication methods on the sludge disintegration were firstly experienced separately, and then the combined effects of sono-alkalization and sono-thermal pre-treatment methods were investigated. The effects of combined pre-treatment methods on the sludge disintegration were evaluated based on the quantities of increase of soluble chemical oxygen demand (sCOD), protein and carbohydrate. Based on the experimental results, an empirical mathematical model was developed to predict easily the process performance by inputting the sonication times. In addition, the sludge dewaterability and settleability were monitored to determine the influences of pre-treatment methods on the important physical properties of sludge.

MATERIALS AND METHODS

Waste activated sludge (WAS)

The WAS sample used in the experimental studies was supplied from the Basarakavak domestic wastewater treatment plant (in Konya, Turkey). The sludge sample was taken from the return activated sludge line and stored in the dark at +4 °C before its usage. The raw WAS had a water content of 99.6%, pH of 7.5, total chemical oxygen demand (tCOD) of 3,735 mg/L, sCOD of 55 mg/L, total solid (TS) of 3,935 mg/L, volatile solid (VS) of 2,450 mg/L, suspended solid (SS) of 3,350 mg/L, volatile suspended solid (VSS) of 2,230 mg/L, capillary suction time (CST) of 18 s and settled sludge volume (SSV_{30}) of 98%. The solid content of the sludge was adjusted to 1% TS content by precipitating gravitationally and diluting with ultrapure water before the experiments. The sludge at 1% TS content had a tCOD of 10,375 mg/L and sCOD of 90 mg/L.

Disintegration methods

According to the study of Şahinkaya (2011) performed in the ultrasonic density range from 0.35 to 1.50 W/mL, increase in the ultrasonic density up to 1 W/mL led to a critical increase in the disintegration of sludge. However, further rises in the density had negligible effects on the sludge disintegration, when compared with the ultrasonic power applied into the WAS. As a result, it was determined in that study that the most efficient ultrasonic density was 1 W/mL based on the chemical parameters such as sCOD, carbohydrate and protein. In our study, therefore, the aim was to improve the sludge disintegration efficiency of sonication by combining alkalization and thermal pre-treatment methods at a fixed ultrasonic density of 1 W/mL.

Sonication was conducted with an ultrasonic homogenizer (Bandelin, HD3200, Germany), which was equipped with a titanium probe (Bandelin, TT13, Germany). The ultrasonic frequency was fixed at 20 kHz which is the best for sludge disintegration (Pilli *et al*. 2011). The maximum high frequency power output was 200 W. The applied power into the sludge was adjusted by an energy input control on the ultrasonic device. One hundred milliliters of WAS sample was poured into a beaker with the probe placed at the middle of the sample, which was dipped 2 cm into the WAS (Şahinkaya & Sevimli 2009). Sludge samples were sonicated for different times up to 10 min at a constant ultrasonic density of 1 W/mL. As the rise of temperature may also contribute to the disintegration of sludge (Pilli *et al*. 2011), the temperature was not controlled during the batch experiments. The sludge pH was not adjusted. However, the change in pH values was less than 0.5 unit, after sonication.

Alkalization was carried out in a jar test apparatus (Velp, FC 6S, Italy) equipped with six beakers containing 250 mL of WAS under rapid mixing condition (90 rpm) at room temperature. NaOH is the most effective alkaline reagent for the disintegration of WAS (Li *et al*. 2008). It was therefore used as an alkaline source in this study. As the applied base dosage is a more effective parameter than alkalization time, alkalization was conducted in the range of NaOH from 0.01 to 0.50 N for the constant disintegration time of 0.5 h. The desired NaOH concentrations in the sludge were adjusted by using 5 N NaOH solution. After alkalization, pH of the pre-treated sludge was adjusted to about 7.5 by using 0.1 and 5 N H_2SO_4 solutions.

Low temperature thermal pre-treatment was performed at 60, 80 and 100 °C for 1 h without mixing. The experiments were done in a pre-heated oven (Binder, Germany) with 250 mL WAS sample using covered volumetric flasks to impede the evaporation of aqueous phase of sludge.

The sono-alkalization method is the combination of sonication and alkalization at a fixed NaOH concentration (0.05 N). Total pre-treatment time was 0.5 h. After the addition of the required dosage of NaOH which was equal to 0.05 N in 100 mL WAS, the sludge was mixed for 0.5 min and the sludge was sonicated at the ultrasonic density of 1 W/mL for the ultrasonic irradiation times of 0.5, 1, 2.5, 5, 7.5 and 10 min. The sonicated sludge was kept at room temperature for the remaining part of the total pre-treatment time. The pre-treated sludge was neutralized by using 0.1 and 5 N H_3PO_4 solutions at the end of the pre-treatment time.

The sono-thermal pre-treatment method is the combination of sonication and thermal pre-treatment. In this combination, the rise of temperature occurring during sonication was aimed to be utilized as an advantage for the subsequent low temperature thermal pre-treatment in order to improve the sludge disintegration. After the ultrasonic pre-treatment at 1 W/mL for varying times from 0.5 to 10 min, the sonicated sludge samples (100 mL) were thermally disintegrated in a pre-heated oven at 80 °C for 1 h in the covered volumetric flasks.

Analysis

After the pre-treatment experiments, the sludge samples at room temperature were used for the analysis. The sCOD, tCOD, SS, TS, CST and water content measurements were performed according to Standard Methods (APHA/AWWA/WEF 2005). After the sludge was centrifuged at 3,000 rpm for 10 min, the supernatant was filtered through a membrane filter (0.45 μm), and then the filtrate was used for the measurements of COD, protein and carbohydrate concentrations in soluble phase. The soluble carbohydrate and protein were measured by the phenol sulphuric acid method (Dubois *et al*. 1956) and the modified Lowry method (Pierce, assay kits, USA), respectively. The TVS and VSS were determined according to the method of Tiehm *et al*. (2001). The pH and turbidity of the sludge were measured by using a pH meter (WTW, 340i, Germany) and a turbidimeter (Hach, 2100P, Germany), respectively.

The degree of sludge disintegration (DD_{COD}), which is the main parameter for the evaluation of sludge disintegration performance of the pre-treatment methods, was calculated as the ratio of the sCOD increase by the pre-treatment to the maximum possible sCOD increase (Equation (1)):

$$DD_{COD}(\%) = \left[\frac{sCOD - sCOD_0}{tCOD - sCOD_0}\right] \times 100 \tag{1}$$

where, sCOD is the sCOD concentration of the pre-treated sludge (mg/L), $sCOD_0$ is the sCOD concentration of the raw (untreated) sludge (mg/L) and tCOD is the tCOD concentration of the raw sludge (mg/L).

The settling ability of sludge was represented by the settling volume in a measuring cylinder in 30 min (SSV_{30}) (Spellman 2009). Despite the fact that the settling ability measurement is not a standard method; it was used to determine the changes in the sludge settling property after the pre-treatment. The CST analyses were carried out to determine the dewatering capacity of WAS samples via a CST meter (Venture, USA). The microscopic examination of the sludge was performed by using a light microscope

(Olympus, BX51T, Germany) to determine the effects of pre-treatment methods on filamentous bacteria and sludge flocs.

RESULTS AND DISCUSSION

The effects of pre-treatment methods on sludge disintegration

The pH values of the alkaline pre-treated sludge samples by using 0.01, 0.05, 0.10 and 0.50 N NaOH were measured as 8.7, 12.1, 12.6 and 12.9, respectively. The application of 0.05 N NaOH resulted in an extreme increase in pH and further dosages did not alter the sludge pH significantly. As represented in Figure 1, NaOH dosage has a significant influence on sludge disintegration until reaching a pH of 12.1 (at 0.05 N NaOH), because alkalization is a disintegration process depending on the sludge pH. The higher dosages have a limited effect on the sludge disintegration, when comparing the increase in soluble carbohydrate, protein and COD to the alkaline dosages used (Figure 1). Therefore, the optimal NaOH dosage was found as 0.05 N NaOH. Similar to our results, Li *et al*. (2008) also determined the optimum condition as NaOH concentration of 0.05 N in the sludge for the alkalization time of 0.5 h, based on DD_{COD}. Consequently, sono-alkalization was performed at a fixed NaOH concentration of 0.05 N.

The rise of temperature during low temperature thermal pre-treatment of WAS resulted in a negligible increase in the sludge disintegration, as shown in Figure 2. DD_{COD} values increased to 6.1, 7.2 and 8.0% at 60, 80 and 100 °C, respectively. Similarly, COD, carbohydrate and protein concentrations in soluble phase increased with rising temperature. However, thermal pre-treatment at low temperature was not effective as expected, compared with alkalization and sonication methods. Thus, the temperature of 80 °C was selected as the optimum temperature in low temperature thermal pre-treatment, and sono-thermal pre-treatment was performed at a constant temperature of 80 °C, after sonication of WAS.

Prolonging of sonication at fixed ultrasonic density (1 W/mL) resulted in an increase in ultrasonic energy applied to the WAS and the rise of sludge temperature up to about 80 °C. The rise in sludge temperature indicated that the acoustic cavitation was more violently generated in the WAS as the pre-treatment time was prolonged. Thus, sludge disintegration by sonication was successively improved with prolonging of sonication. The DD_{COD} increased to 3.5, 5.4, 10.3, 13.1, 18.7 and 22.0% as a result of sonication at 0.5, 1, 2.5, 5, 7.5 and 10 min, respectively (Figure 3(b)). Similar to the increasing trend of DD_{COD}, the releases of soluble COD, carbohydrate and protein were also enhanced, as shown in Figure 3. Thus, the sludge solubilization was steadily improved. While the

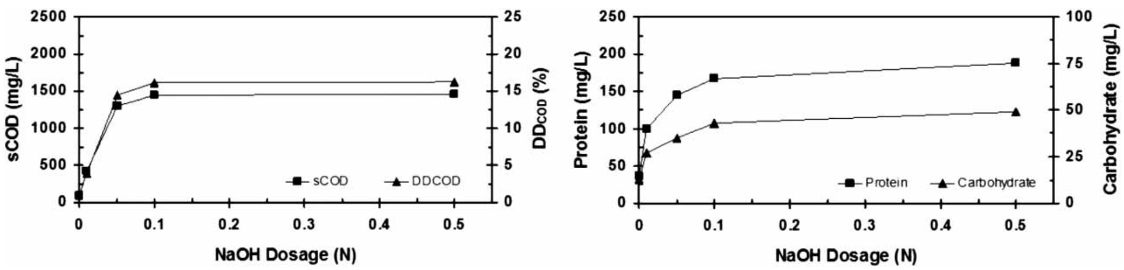

Figure 1 | The effect of alkalization on sludge disintegration.

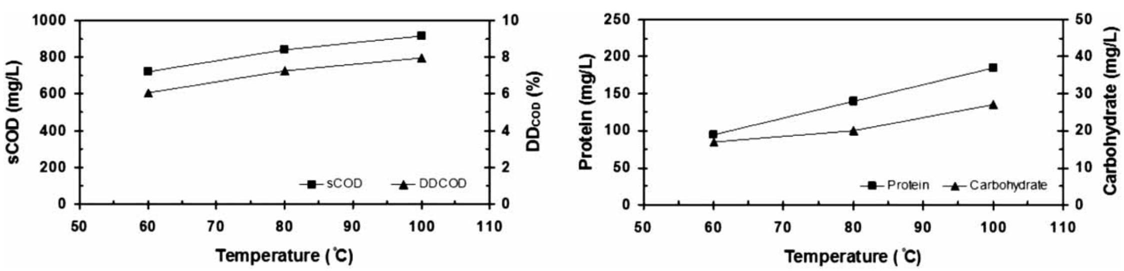

Figure 2 | The effect of thermal pre-treatment on sludge disintegration.

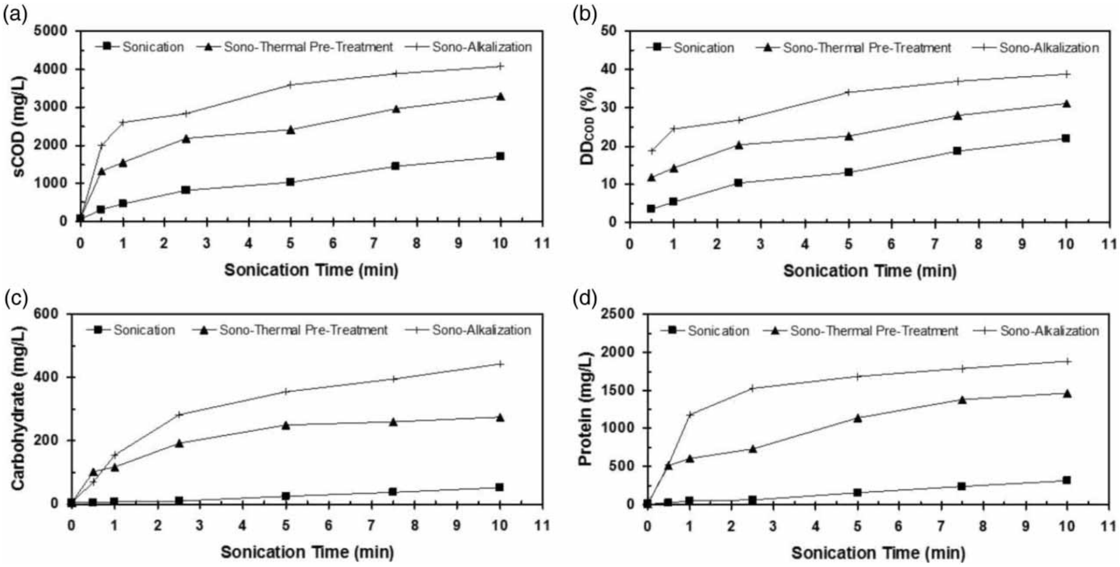

Figure 3 | The effect of ultrasonic pre-treatment methods on sludge disintegration.

sludge was rapidly disintegrated in the first 2.5 min of sonication, the disintegration rate was reduced in the remaining pre-treatment time. Therefore, it was determined that the sludge disintegration occurred in two stages: rapid and subsequent slow disintegration stages.

In the combination of sonication and alkalization methods, the DD_{COD} values at the sonication times of 0.5, 1, 2.5, 5, 7.5 and 10 min had risen sharply to 18.6, 24.4, 26.7, 34.0, 37.0 and 38.8% by the end of the pre-treatment time of 30 min (Figure 3(b)). The sCOD, carbohydrate and protein concentrations were increased, similarly to DD_{COD} (Figure 3). In this combined method, sludge flocs were firstly dissipated by hydro-mechanical shear forces generated by ultrasonic irradiation and thus microbial cells released from the disrupted flocs were more effectively exposed to OH^- ions. Due to these synergistic effects of two different disintegration mechanisms, the sludge solubilization was significantly improved. As a result, the sludge disintegration efficiency of the sono-alkalization method was higher even than the total of alkalization and sonication methods.

In the combination of sonication and thermal pre-treatment methods, the rise of temperature during sonication was purposed to be utilized as an advantage to enhance the sludge disintegration. We could not find a similar combination on sludge pre-treatment in the literature. Therefore, we aimed to provide a new point of view on sludge disintegration by sono-thermal pre-treatment. As a result of the thermal pre-treatment of the sonicated sludge, the sludge disintegration was improved. After the thermal pre-treatment of WAS sonicated for 0.5, 1, 2.5, 5, 7.5 and 10 min, the DD_{COD} increased to 11.9, 14.2, 20.3, 22.7, 28 and 31.1%, respectively (Figure 3(b)). The improvement in sludge disintegration resulted from the disintegration of microbial cells released from the sludge flocs by hydro-mechanical shear forces with the effect of rising temperature. As seen from Figure 3, carbohydrate, protein and sCOD concentrations were increased as a result of the release of extracellular and intracellular materials.

As a result of the combination of sonication with alkalization and thermal pre-treatment methods, sludge disintegration was enhanced due to the synergistic effects. As shown in Figure 3, sono-alkalization was the most effective method for the sludge disintegration at even lower ultrasonic energy consumptions. In addition, the sludge was much more rapidly disintegrated by the sono-alkalization method. Therefore, the combination of sonication with alkalization not only increased the sludge disintegration, but also reduced the ultrasonic energy consumption (by shortening the sonication time) to obtain the same disintegration yield, compared with sonication alone. Therefore, sono-alkalization can be a new and cheaper alternative pre-treatment method for the disintegration of WAS in the wastewater treatment plants.

Kinetic modelling of sludge pre-treatment

In this study, a new kinetic mathematical model described by Wu *et al.* (2011) was applied to the experimental DD_{COD} data (shown in Figure 3(b)) to determine the effect of sonication time on sludge disintegration and to predict the efficiencies of sludge disintegration methods. For this purpose a new relationship between the sonication time (t) and disintegration degree (DD_{COD}) has been developed. As the NaOH dosage in sono-alkalization and temperature in sono-thermal pre-treatment were fixed, these conditions were not included in the modelling of sono-alkalization and sono-thermal pre-treatment methods. Therefore, the independent variable of the model was the sonication time. The model can be described as follows (Equation (2)),

$$\chi = \frac{t}{(a + b \cdot t)} \tag{2}$$

where, χ is the DD_{COD} of the pre-treated sludge at the corresponding time t (min) and t is the sonication time. The a and b are two constants of the model relating to the initial disintegration rate ($1/a$) and the theoretical maximum disintegration capacity ($1/b$), respectively. To determine the characteristics of a and b, Equation (2) can be linearized as given in Equation (3),

$$\frac{t}{DD_{COD}} = a + b \cdot t \tag{3}$$

The experimental data of DD_{COD} were linearized by using Equation (3) (Figure 4). A straight line was obtained by plotting t/DD_{COD} for each ultrasonic pre-treatment method (sonication, sono-alkalization and sono-thermal pre-treatment) versus sonication time t, and the a and b coefficients of Equation (3) were calculated from the slope of the line and the intercept, respectively (Figure 4). The model characteristics (a and b) and the coefficient of determination (R^2) are also presented in Table 1. As seen from Table 1, the initial disintegration rate ($1/a$) and the theoretical maximum disintegration capacity ($1/b$) were highest in the in sono-alkalization method. These results of the proposed model were also in good agreement with the experimental results shown in Figure 3(b).

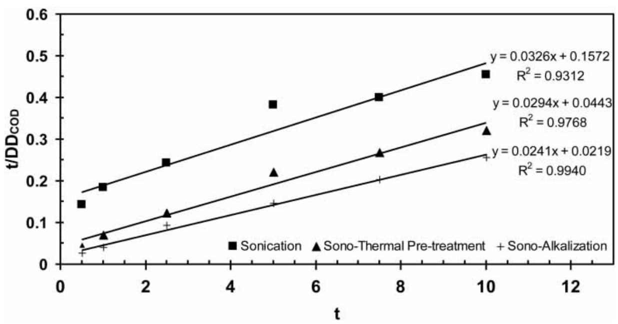

Figure 4 | Typical model verifications of ultrasonic pre-treatment methods.

Figure 5 illustrates the simulated data generated from Equation (3) for each ultrasonic sludge disintegration method and experimental results. Figure 5 also shows the comparison of the experimental and simulated data. The comparative model results in high R^2 values, indicating that the proposed kinetic model could be used for predicting the disintegration degree of sludge.

The effects of pre-treatment methods on sludge characteristics

As turbidity indicates the change in particle size which significantly affects the sludge filterability, it was evaluated together with the dewatering property of sludge. As shown in Figure 6(a), turbidity of the pre-treated sludge increased due to the reduction of particle size. Turbidity of the WAS pre-treated by sono-thermal pre-treatment increased due to

Table 1 | Determination coefficients and characteristics of the proposed model

Pre-treatment methods	a	b	R^2
Sonication	0.1572	0.0326	0.9312
Sono-alkalization	0.0219	0.0241	0.9768
Sono-thermal pre-treatment	0.0443	0.0294	0.9940

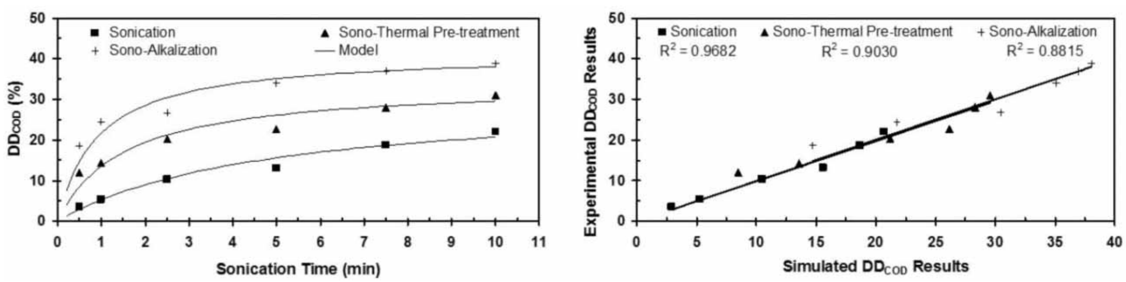

Figure 5 | The correlations between the experimental and simulated data for DD_{COD}.

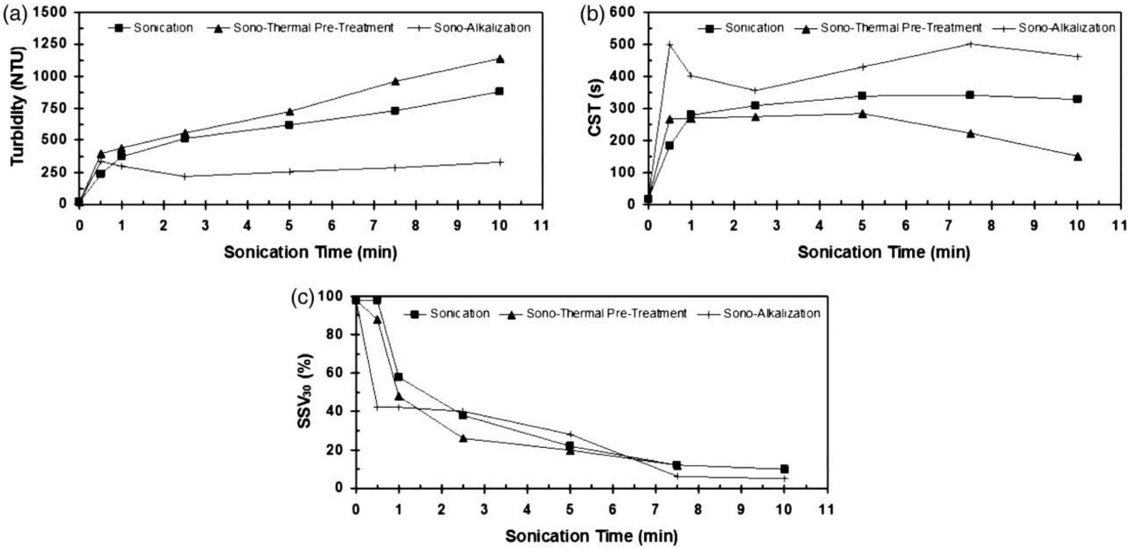

Figure 6 | The effect of ultrasonic pre-treatment methods on sludge characteristics.

the improvement of the sludge disintegration and the reduction of particle size, compared with the sonicated sludge. However, in sono-alkalization, the solubilized organic matter was composed of a gel-like material containing a large amount of water and some fine particles at extremely high pH (Li *et al.* 2008). Therefore, the turbidity decreased in the sono-alkalization method due to the re-flocculation of fine particles.

The dewatering ability of sludge deteriorated compared with the raw sludge (Figure 6(b)). The deterioration in dewaterability might occur for two main reasons: (1) the increase in the concentration of hydrophilic polymeric substances such as carbohydrate and protein, and (2) the reduction in the particle size (Wang *et al.* 2006). Consequently, as the maximum sludge disintegration was obtained by sono-alkalization, the dewaterability of the sono-alkaline pre-treated sludge was the worst. However, the application of thermal pre-treatment after sonication caused an improvement by thermal conditioning, compared with sonication.

The settling property of raw sludge was so poor due to filamentous bacteria present in the WAS. However, as demonstrated in Figure 6(c), the settling ability of the pre-treated sludge might be improved due to three reasons: (1) the re-flocculation of the fine particles due to the release of intracellular and extracellular materials (Feng *et al.* 2009) and the increase in particle cohesion and density (Gonze *et al.* 2003); (2) the disintegration of filamentous microorganisms; and (3) thermal conditioning by the effect of rising temperature.

CONCLUSION

In the present study, the effects of alkalization, sonication, thermal pre-treatment, sono-alkalization and sono-thermal pre-treatment methods on both the sludge disintegration and sludge properties and modelling of performances of ultrasonic sludge pre-treatment methods were investigated. The following conclusions can be drawn from this experimental study:

- The maximum sludge disintegration degrees by sonication for 10 min, alkalization at 0.05 N for 0.5 h and thermal pre-treatment at 80 °C for 1 h were 22.0, 14.4 and 7.2%. However, the disintegration degrees significantly increased to 38.8% by sono-alkalization and to 31.1% by sono-thermal pre-treatment methods. Therefore, the degrees of sludge disintegration obtained by sono-alkalization and sono-thermal pre-treatment methods were more than the total of the disintegration degrees achieved by two methods individually. In these combined methods, the sludge disintegration increased due to the synergistic effects of different disintegration mechanisms. Among the five pre-treatment methods, sono-alkalization caused much more release of extracellular and intracellular materials. In addition, the energy consumption in sono-alkalization was the least, compared with sonication and sono-thermal pre-treatment methods. While the sludge disintegration degree of 22.0% was achieved with sonication for 10 min, sono-alkalization with 1 min sonication increased the disintegration degree to 24%. Therefore, it is also possible to reach the higher sludge solubilization with even less ultrasonic energy consumption, compared with sonication and sono-thermal pre-treatment methods. As a result, sono-alkalization method is a new effective alternative for the sludge pre-treatment.

- The sludge disintegration performances of ultrasonic pre-treatment methods were simulated by using the kinetic mathematical model. It was proven that the proposed model was a useful tool to predict accurately the sludge disintegration performance by inputting sonication times.
- Compared with sonication, while sono-thermal pre-treatment improved the dewatering property of sludge, sono-alkalization caused a deterioration in the dewaterability of sludge. On the other hand, all ultrasonic pre-treatment methods enhanced the sludge settleability.

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