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# Improvement of lime softening treatment of secondary effluent by the addition of fly ash

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## ABSTRACT

The sludge generated in the lime softening of recirculating cooling water has a large bulk volume and is difficult to dehydrate. The current study tested the effect of fly ash addition on the pollutant removal efficiency and the sludge properties improvement in the softening process. The hardness and alkalinity removal efficiency before and after the addition of fly ash were evaluated. The effects of fly ash addition on chemical sludge properties, such as the sludge water content, particle size, and microstructure, were also investigated. With a fly ash dosage of 270 mg/L, the removal rates of calcium hardness, total hardness, and total dissolved solids increased by 20.7, 11.5, and 6.1% compared with those obtained in the absence of fly ash. The addition of fly ash can improve the stability of treated water by reducing the scaling potential. Fly ash can improve the sludge properties. At a fly ash dosage of 200–250 mg/L, the median diameter of the flocs is four times bigger than the flocs produced in the absence of fly ash, thereby accelerating the sludge settling speed. After fly ash addition, the sludge water content decreased, facilitating sludge dehydration and resulting in a more compact sludge.

*Keywords:* Alkalinity; Fly ash; Hardness; Lime softening; Secondary effluent; Sludge property

## 1. Introduction

In power plants, 60–80% of cooling water was consumed from the total water consumption. Considering water resource conservation, some power plants substitute freshwater with reclaimed water from domestic wastewater treatment plants (WWTP) as the cooling and make-up water in their recirculating cooling water systems. After treatment, secondary effluents still contain a considerable amount of pollutant, as indicated by the turbidity, hardness, alkalinity, and microbe content. These pollutants can cause scale formation, corrosion, and/or bacterial growth when reclaimed water is directly reused [1]. Therefore, secondary effluents require further treatment to remove impurities and to meet the quality requirements for recirculating cooling water [2–4]. At present, lime softening is the most widely used approach to treat reclaimed water from the secondary effluents of

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WWTPs in power plants. Lime softening is effective in removing hardness and color [5] and also enables the further removal of phosphorous and suspended solid. However, calcium carbonate crystals produced during the softening process are often deposited in the internal wall of the pipeline as well as in the infrastructure, eventually blocking the infrastructure and the pipelines. This phenomenon increases the maintenance costs as well as the maintenance difficulty. Furthermore, the bulk volume and difficult dehydration of lime sludge increases the complexity of chemical sludge removal. Therefore, alternative cost-effective methods that improve the sludge properties and reduce the difficulty in sludge treatment and disposal have to be developed.

Fly ash is the residual solid material generated during coal combustion for energy production in power plants. Given the bulk production volume and potential environmental impact of fly ash, it is sometimes recognized as an environmental pollutant. Depending on the source and makeup of the coal being burned, the components of fly ash vary considerably. However, fly ash generally contains substantial amounts of silicon dioxide ($SiO_2$) (both amorphous and crystalline), calcium oxide (CaO), aluminum oxide ($Al_2O_3$), and iron oxide ($Fe_2O_3$). Fly ash also contains trace elements, such as Mg, K, Na, S, Ti, P, and Mn [6]. Fly ash can be used as a material for construction, soil amendment, and sludge conditioning [7,8]. In addition, fly ash is used as a low-cost adsorbent for the removal of metals, dyes, and other organic compounds in wastewater because of its high adsorption capacity [9–11].

This study aims to increase the impurity removal efficiency and improve chemical sludge properties by incorporating the use of fly ash in conventional lime softening. The efficiencies of hardness, alkalinity, and total dissolved solid (TDS) removal before and after fly ash addition were compared. The effects of fly ash on the sludge properties, such as water content, particle size, and microstructure, were also evaluated. The results can serve as references in the use of reclaimed water for cooling in power plants.

## 2. Materials and methods

### 2.1. Raw water

The secondary effluent of a wastewater treatment plant in Shandong Province, China, was used as the raw water in this study. Wastewater treatment process in this plant includes a conventional activated sludge process that can remove nitrogen and phosphorus. Table 1 summarizes the water quality of the secondary effluent collected from the plant in September 2011–April 2012.

### 2.2. Source and component of fly ash

Fly ash used in this experiment was derived from a certain local thermal power plant. The surface area of the fly ash is about $350\,m^2/kg$, and the component of the fly ash is listed in Table 2.

### 2.3. Batch experiments

Jar tests were conducted to evaluate the effect of fly ash addition on the lime softening system. A certain amount of fly ash was added into a beaker containing 1,000 mL of raw water. The mixture was stirred at a speed of 500 rpm for 2 min. Lime was then added into the beaker to obtain a 260.5 mg/L of lime concentration. The mixture was rapidly stirred at a speed of 500 rpm for 3 min, slowly stirred at a speed of 80 rpm for 15 min, and then precipitated for 30 min. A portion of the supernate was analyzed to determine the calcium hardness, total hardness, total alkalinity, and TDS. The chemical sludge was then collected to analyze the sludge properties, such as water content, size distribution, and microstructure.

A comparative test was performed to evaluate the effect of fly ash addition on the floc sedimentation speed. Aside from hydrated lime, $Al_2(SO_4)_3$ was added into the raw water to serve as a coagulant. Fly ash was also added into the contrast system. The dosing sequence of the reagents was as follows: hydrated lime was added into the reaction system and reacted for 4 min after fly ash was added and reacted with water for 2 min. $Al_2(SO_4)_3$ was then added into the mixture to obtain a coagulant concentration of 10 mg/L (calculated on the basis of $Al_2O_3$). After reacting for 15 min, the mixture was left to precipitate statically for 30 min. The water was sampled every five minutes during the course of sedimentation to determine water turbidity. All the experiments were conducted in triplicate.

Calcium hardness and alkalinity were measured via complexometric titration and acid/base titration, respectively. A PHS-3B acidity meter (Shanghai Precision & Scientific Instrument Co., Ltd.) was used to determine the pH of the water sample, and a 2100AN turbidimeter (HACH Co., Ltd., USA) was used to measure the turbidity. The sludge particle size was determined using a Winner 2000 laser particle size analyzer (Winner instruments, China). The microstructure of the sludge was observed under an S-3500 N scanning electron microscope (Hitachi Ltd., Japan).

Table 1
Raw water quality of this test

| Parameter | Mean ± SD | Parameter | Mean ± SD |
|---|---|---|---|
| Turbidity/NTU | 12.51 ± 2.89 | Total hardness/mg $CaCO_3$ $L^{-1}$ | 512.13 ± 74.53 |
| pH | 7.76 ± 0.23 | Calcium hardness/mg $CaCO_3$ $L^{-1}$ | 361.67 ± 62.28 |
| Total alkalinity/g $CaCO_3$ $L^{-1}$ | 258.99 ± 20.45 | TDS | 1494.56 ± 46.23 |
| Conductivity/μs·$cm^{-1}$ | 1916.00 ± 118.30 | | |

Table 2
Component of the fly ash used in this experiment

| Component | Mean value (%) |
|---|---|
| Active $SiO_2$ | >7 |
| $Al_2O_3$ | 10 |
| CaO | 1 |
| $CaSO_4$ | 7 |
| $Na_2O$ | 0.2 |
| $K_2O$ | 0.6 |

The gravimetric method was used to determine the sludge water content and TDS, and all analyses were carried out in duplicate.

## 3. Results and discussion

### 3.1. Effects of fly ash dosage on hardness, alkalinity, and TDS removal

In the lime-only softening process, the removal rates of calcium hardness, total hardness, and TDS were 44.2, 34.0, and 18.1%, respectively, when lime dosage was 260.5 mg/L. The addition of fly ash improved the hardness and TDS removal rates to some extent (Fig. 1). The removal rates of calcium hardness, total hardness, and TDS increased as the fly ash dosage increased. Calcium hardness, total hardness, and TDS removal rates were 64.9, 45.5, and 24.2%, respectively, when fly ash dosage reached 270 mg/L. These values are higher by 20.7, 11.5, and 6.1%, respectively, compared with those obtained in the absence of fly ash.

Total hardness includes calcium hardness and magnesium as well as carbonate hardness and noncarbonate hardness. Under alkali condition, the active $SiO_2$ in fly ash reacts with $Ca^{2+}$ in raw water via the typical alkali–silica reaction to generate a calcium silicate precipitate as shown in Eq. (1):

$$SiO_2 + Ca^{2+} + 2OH^- = CaSiO_3 \downarrow + H_2O \tag{1}$$

The calcium hardness of treated water was 140.16 mg/L at a fly ash dosage of 270 mg/L, indicating one-third decrease compared with that obtained in the lime-only softening process. This result suggests that the cooling water circulation ratio can increase by one-third when the reclaimed water treated by a combination of fly ash and lime is used as makeup water.

During this process, active $Ca^{2+}$ in fly ash dissolves in wastewater. However, the amount of dissolved $Ca^{2+}$ in the water is considerably lower than the amount of $Ca^{2+}$ removed by the alkali/silica reaction when the fly ash dosage is low. A higher number of active $Ca^{2+}$ in fly ash dissolves in the wastewater as the fly ash dosage further increases. The amount of active $SiO_2$ dissolved in water depends on the water pH: a higher pH causes a higher number of active $SiO_2$ to dissolve. In this experiment, lime dosage was kept constant; hence, water pH was invariant, thereby keeping the amount of $SiO_2$ that participated in the alkali/silica reaction constant. Further increases in the fly ash dosage led to an excess amount of $Ca^{2+}$ (dissolved from fly ash) in the water after the fly ash dosage was sufficient to react with all of the $Ca^{2+}$ in water, thereby resulting in increased calcium hardness of water. Therefore, the removal rates of calcium hardness and total hardness decreased when fly ash dosage exceeded 360 mg/L.

Lime softening cannot reduce noncarbonate hardness because magnesium salts react with lime to produce an equivalent molar concentration of calcium hardness that does not belong to carbonate hardness.

$$Mg^{2+} + Ca(OH)_2 \rightarrow Mg(OH)_2 \downarrow + Ca^{2+} \tag{2}$$

Therefore, the total hardness removal rate was lower than of calcium hardness.

The total alkalinity fluctuated between 46 and 82 mg/L, and the removal rate varied from 70 to 83% when lime dosage was 260.53 mg/L. The alkalinity removal rate was the lowest in the absence of fly ash. Meanwhile, at fly ash dosages below 225 mg/L, higher fly ash dosages resulted in higher alkalinity removal rates. The alkalinity removal rate decreased as the fly ash dosage increased when the fly ash dosage exceeded 225 mg/L. The highest alkalinity removal

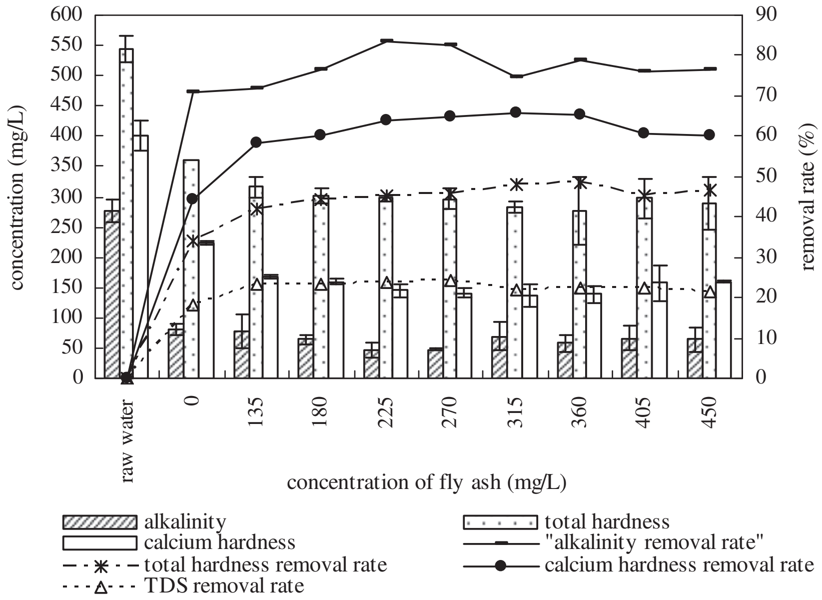

Fig. 1. Effects of fly ash dosage on total hardness, calcium hardness, alkalinity, and TDS removal rates.

rate (80%) was obtained when fly ash concentration ranged from 225 to 270 mg/L. The total alkalinity includes bicarbonate alkalinity ($HCO_3^-$), carbonate alkalinity ($CO_3^{2-}$), hydroxide alkalinity ($OH^-$) and some residual pollutants, such as, weak base ($NH_3 - NH_2$), organic weak acid salt and inorganic weak acid salt in the secondary effluent. Bicarbonate alkalinity is the main component of the total carbonate in the raw water used in this experiment. $Ca(OH)_2$ in lime milk can react with $HCO_3^-$ to form a calcium carbonate precipitate ($CaCO_3$) and $H_2O$, resulting in the reduction in alkalinity. The $CaCO_3$ generated in this process has colloidal properties and is capable of adsorbing and enmeshing weak organic acid residues, such as humic acid, aliphatic acid, and acetic acid in the secondary effluent. Furthermore, fly ash is porous and has large specific surface areas that can adsorb weak organic acids, thereby contributing to the decrease in alkalinity [12]. A higher amount of active CaO in fly ash dissolves in the water and reacts with water to generate $Ca(OH)_2$, when the fly ash dosage exceeded 270 mg/L, leading to a slight increase in alkalinity [13]. The fly ash used in this study contained approximately 1% CaO; thus, the dissolution of 270 mg/L of fly ash in water produced 0.2 mmol $OH^-$, consequently increasing water alkalinity by 5 mg/L.

TDS removal rate in the lime-only softening is limited. The addition of fly ash can improve TDS removal efficiency to some extent. The addition of a high dosage of fly ash increased the amount of $SiO_2$ that participated in the alkali/silica reaction when fly ash dosage was below 270 mg/L. Consequently, a higher amount of calcium was removed through the formation of the $CaCO_3$ sediment. Fly ash contains approximately 10% $Al_2O_3$. $Al_2O_3$ dissolves in water to form an $Al(OH)_3$ colloid, which can adsorb inorganic or organic compounds, thereby reducing TDS [14]. Further increases in the fly ash dosage did not result in an increased TDS removal rate because of the increased amount of $Ca^{2+}$ (from the fly ash) dissolved in the water when fly ash dosage exceeded 270 mg/L. Hence, TDS increased instead.

The addition of 270 mg/L of fly ash is recommended to achieve the optimal hardness, alkalinity, and TDS removal rates.

### 3.2. Stability of the treated water

Stable water does not scale or corrode materials during transport or usage. The Langlier saturation index (LSI) is generally used to represent water stability, which can be expressed by Eq. (3) [15]:

$$\text{LSI} = \text{pH} - \text{pHs} \tag{3}$$

This equation represents the equilibrium relation between $CaCO_3$ and the solution containing $CO_2$, where pH represents the measured pH, when $CaCO_3$ in the solution is saturated. Water is stable and will not deposit or corrode when LSI is zero. An LSI greater than zero indicates that $CaCO_3$ is oversaturated in the wastewater and may settle, leading to deposition corrosion. An LSI below zero indicates that $CaCO_3$ in the water is unsaturated and can be further dissolved. Thus, water can cause corrosion [16]. pHs can be expressed as follows [17]:

$$\text{pHs} = \log\left(\frac{k_{a2}\gamma_{\text{Ca}^{2+}}[\text{Ca}^{2+}]\gamma_{\text{HCO}_3^-}[\text{HCO}_3^-]}{\text{Ksp}}\right) \tag{4}$$

where $K_{a2}$ is the observed dissociation equilibrium constant of bicarbonate, which is $3.715 \times 10^{-11}$ at 15℃ and $4.477 \times 10^{-11}$ at 25℃; $\gamma_{\text{Ca}^{2+}}$ is the activity coefficient of calcium; [$\text{Ca}^{2+}$] is the calcium concentration, mg/L; $\gamma_{\text{HCO}_3^-}$ is the activity coefficient of bicarbonate; [$\text{HCO}_3^-$] is the bicarbonate concentration; and Ksp is the solubility product constant of calcium bicarbonate dissociation, which is $6.02 \times 10^{-9}$ at 15℃, and $4.57 \times 10^{-9}$ at 25℃.

$\log \gamma$ can be expressed as:

$$\log \gamma = \frac{0.5(Zi)^2\sqrt{2.5 \times 10^{-5} \times \text{TDS}}}{1 + \sqrt{2.5 \times 10^{-5} \times \text{TDS}}} \tag{5}$$

where $Zi$ is the electric charge and is equal to 1 for $\text{HCO}_3^-$ and 2 for $\text{Ca}^{2+}$.

In this study, the treated water quality before and after the addition of fly ash at a lime dosage of 260.5 mg/L is shown in Table 3.

At this water quality, the LSIs of treated water before and after fly ash addition were 0.40 and 0.26 for 15℃, and 0.56 and 0.46 for 25℃, respectively. And these two temperatures are typical wastewater temperature in warm season and cold season in North China. Therefore, both types of treated water exhibit a deposition potential and require subsequent pH adjustments. In addition, the LSI in the presence of fly ash was lower than that in the absence of fly ash, indicating that the addition of fly ash can improve the treated water stability to a certain degree.

Addition of fly ash can not only increase alkalinity and hardness removal efficiency, improve the treated water stability, but also remove more residual organic pollutants. Under the optimal condition, the removal rate of COD increased by 13%, which could be proved by Table 4. Furthermore, after the addition of fly ash, pH of the system decreased slightly, resulting in the treated water transferring from alkaline zone to neutral region.

### 3.3. *Effect of fly ash dosing on floc properties*

#### 3.3.1. *Floc sedimentation rate*

In the above research, only the effect of fly ash addition on the soluble index was tested, hence, flocculant was not involved in the softening process. Since some tiny suspended flocs still presented in the supernatant after sedimentation in both softening processes, increasing turbidity in the effluent, $\text{Al}_2(\text{SO}_4)_3$ was added in the softening process to evaluate the effect of fly ash addition on the floc sedimentation speed and improve the effluent quality. The obtained values were subsequently used to determine the floc sedimentation rate. The results are shown in Fig. 2.

The sedimentation rate of the chemical floc was significantly low for the lime-only softening process. After 30-min sedimentation, the turbidity of treated water remained above 15 NTU. However, the addition of fly ash accelerated the sedimentation rate. The turbidity reached approximately 5 NTU after sedimentation for only 5 min when fly ash dosage exceeded 150 mg/L. In addition, the floc settling speed increased as fly ash dosage increased (Fig. 2). However, further increases in fly ash dosage did not increase the sedimentation rate when fly ash dosage exceeded 150 mg/L. Moreover, the turbidity of treated water at

Table 3
Treated water quality before and after fly ash addition at a lime dosage of 260.5 mg/L

| | [$\text{Ca}^{2+}$]/mol $\text{L}^{-1}$ | [$\text{HCO}_3^-$]/mol $\text{L}^{-1}$ | TDS/mg $\text{L}^{-1}$ | pH |
|---|---|---|---|---|
| Effluent after lime-only softening | $1.58 \times 10^{-3}$ | $0.41 \times 10^{-3}$ | 1,108 | 9.1 |
| Effluent after treatment with a combination of lime and fly ash | $1.46 \times 10^{-3}$ | $0.57 \times 10^{-3}$ | 1,060 | 8.9 |

Table 4
COD removal rate and pH variation at different fly ash dosage

| Fly ash dosage (mg/L) | 0 | 135 | 180 | 225 | 270 | 315 | 360 |
|---|---|---|---|---|---|---|---|
| Average COD removal rate (%) | 39.5 | 44.1 | 46.0 | 52.2 | 52.8 | 50.3 | 53.3 |
| pH | 9.10 | 8.44 | 8.47 | 8.69 | 8.90 | 9.02 | 9.08 |

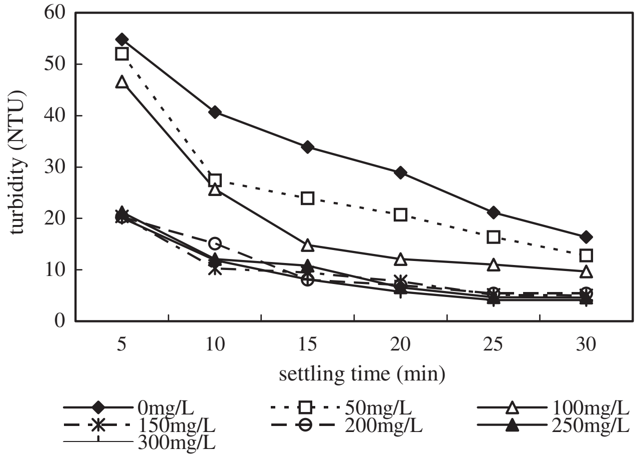

Fig. 2. Change of turbidity with time at different fly ash dosages.

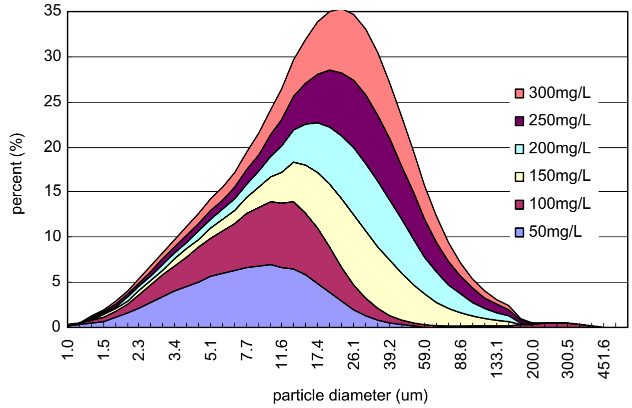

Fig. 3. Change in particle size percentage with the fly ash dosage.

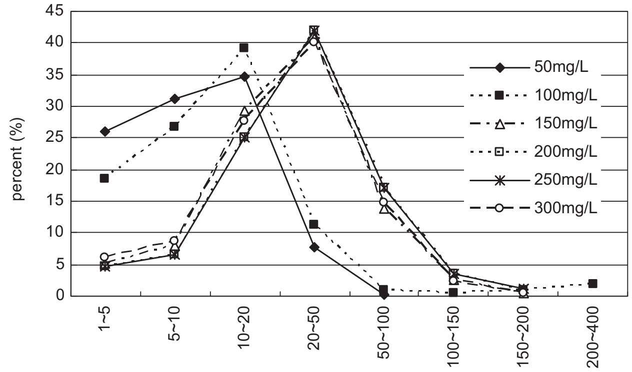

Fig. 4. Distribution of particle size.

this fly ash dosage was below 5 NTU after sedimentation for 25 min. Fly ash, as a multiporous material, has high capacities for adsorption, flocculation, and filtration, resulting in higher turbidity removal efficiencies [18,19]. Fly ash can also serve as a core material for coagulation because of its high adsorption capacity. Moreover, floc sedimentation rate significantly increased because of the high density of fly ash. Therefore, the addition of fly ash can increase turbidity removal efficiency and accelerate floc sedimentation rate, contributing to the reduction of the sedimentation tank volume as well as the construction cost.

### 3.3.2. *Particle size distribution of chemical sludge*

The particle size distribution of the sludge flocs significantly affects their settling properties. The larger the flocs are, the faster the floc settlement. Therefore, addition of flocculant is usually performed to increase floc size and accelerate floc settlement velocity in WWTP [20]. The size distribution of the flocs significantly changes after flocculation. In this study, the particle size distribution of the flocs produced in the coagulation reaction stage was determined at the beginning of the sedimentation stage. The results are shown in Figs. 3 and 4.

The peak in the curve in Fig. 3 shifted to the right when fly ash dosage was increased from 50 to 250 mg/L, indicating that the particles gradually increased in size as the small particle content decreased and the large particle content increased. However, the percentage of particles smaller than 20 μm increased, whereas that of particles larger than 20 μm decreased, when fly ash dosage was increased from 250 to 300 mg/L. The proportion of particles larger than 20 μm was lower than 8% of the total particles when fly ash dosage was only 50 mg/L, whereas particles larger than 20 μm accounted for more than 50% of the total particles, when the fly ash dosage ranged from 150 to 300 mg/L (Fig. 4).

The median diameters (MDs) of the flocs at different fly ash dosages are listed in Table 5. The MD of flocs was 5.13 μm in the absence of fly ash. However, the MD of the sludge flocs increased as fly ash dosage increased. The MD of the flocs reached 25 μm when the fly ash dosage ranged from 200 to 250 mg/L, which is four times higher than those of flocs produced in the absence of fly ash. This phenomenon was observed during the course of the experiment. The floc MD did not increase significantly when fly ash dosage exceeded 200 mg/L, which is consistent with the previously described results.

The porous structure of fly ash allows the use of this substance as core material to form large flocs in the coagulation process. Therefore, floc particle size

Table 5
Change in the median diameter and water content of sludge with the fly ash dosage

| Fly ash dosage (mg/L) | Median diameter (μm) | Water ratio (%) |
|---|---|---|
| 0 | 5.13 | 75 |
| 50 | 7.62 | 72 |
| 100 | 9.83 | 70 |
| 150 | 22.82 | 66 |
| 200 | 25.78 | 65 |
| 250 | 25.73 | 62 |
| 300 | 25.50 | 59 |

increased after the addition of the appropriate amount of fly ash. Furthermore, the excess small particles of fly ash dispersed in the wastewater accounts for the higher percentage of small particles when the fly ash dosage exceeded 250 mg/L, because the fly ash utilized in this study mainly consisted of small and medium-sized particles, of which those smaller than 20 μm accounted for approximately 35%.

### *3.3.3. Sludge water content*

The addition of fly ash can promote the dehydration of sludge. The moisture content of the sludge in the lime-only process was 75%. The sludge moisture content decreased as fly ash dosage increased. At the fly ash dosage of 300 mg/L, the sludge moisture content dropped to 59% (Table 5), indicating a 40% reduction in the sludge volume. In the experiment, the sludge generated in the presence of fly ash was more compact. In addition, the sludge volume in the presence of fly ash was smaller than that in the absence of fly ash, although the dosage of the chemical reagent was twice as in the absence of fly ash.

### *3.3.4. Microscopic structure of the sludge*

The microstructures of the chemical sludge produced in the lime-only softening process as well as that in the softening process with lime-and-fly ash combination are shown in Fig. 5. The lime-only softening sludge is similar to a cotton fluff with a large volume and a loose structure. Some undissolved CaO crystals

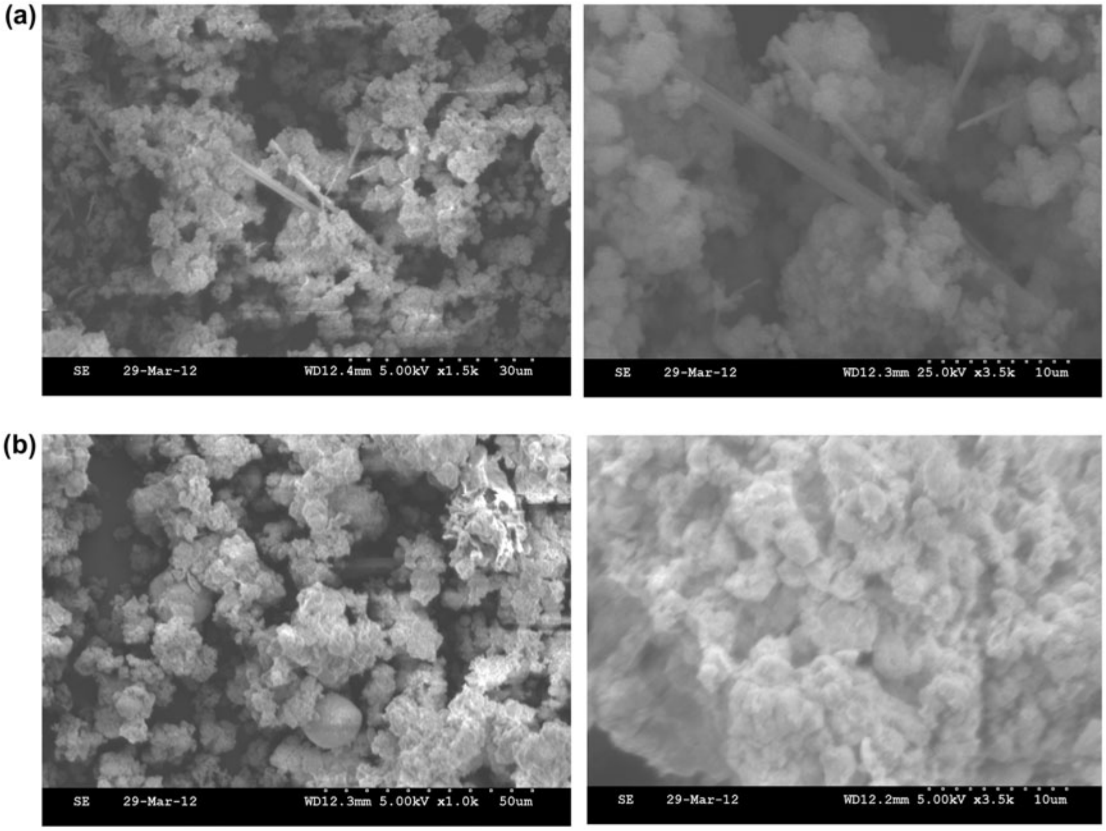

Fig. 5. Stereoscan images of chemical sludge. (a) Image of chemical sludge obtained after the lime-only process. (b) Image of the chemical sludge obtained after the softening process with a combination of lime and fly ash.

were also present. Meanwhile, the structure of the sludge produced after treatment with the lime and fly ash hybrid was more compact. No undissolved CaO crystal was found, and majority of the flocs adsorbed onto the fly ash surface. The porous structure of fly ash enables the adsorption of chemical sludge flocs and serves as the floc core, thereby resulting in an increased floc weight and accelerated sedimentation rate.

## 4. Conclusions

Compared with the lime-only softening process, the addition of fly ash increased the hardness, alkalinity, and TDS removal rates in the secondary effluent of the WWTP. The treated water was more stable after the addition of fly ash, resulting in a lower scaling potential in the subsequent transportation pipes and structures. Furthermore, the addition of fly ash can improve the chemical sludge properties. The sludge sedimentation rate significantly increased after fly ash was added, facilitating the decrease in settling tank volume and in the occupied area. Fly ash can affect the sludge structures, increase sludge floc size, and reduce the difficulty in sludge dewatering. $CaCO_3$ and fly ash are the major components of the chemical sludge obtained after treatment with combined lime and fly ash. This chemical sludge can be used as construction materials and can therefore add to the economic benefits of WWTPs.

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