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Sewage sludge conditioning with coal fly ash modified by sulfuric acid

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ABSTRACT

Activated sludge process to treat municipal and industrial wastewater produces huge amounts of excess sludge. Chemical condition has been employed widely to improve sludge mechanical dewatering, but the cost is high, thus it is very important to find cheap and effective conditioners. This paper studied the improvement of sludge dewaterability with coal fly ash modified by sulfuric acid (MCFA). Through orthogonal experiments with specific resistance to filtration (SRF) as the target index, acid concentration and soaking time were verified to be the important influencing parameters in coal fly ash modification. The optimal modification conditions were: acid concentration, 4 mol l^{-1}; ratio of acid to coal fly ash, 5:1 ml g^{-1}; soaking time, 3 h. After modification the specific surface area of coal fly ash increased from 2.810 to 3.376 $m^2\,g^{-1}$. The dewaterability and the settleability of the conditioned sludge were investigated with vacuum filtrating dewatering tests, centrifugal dewatering tests and settling experiments. The results showed that SRF of the sludge significantly decreased with coal fly ash addition, and the MCFA showed much stronger conditioning capacity than the raw coal fly ash (RCFA). Under a MCFA dosage of 273%, the SRF of the sludge decreased from 1.86×10^{13} to 4.23×10^{11} m kg^{-1}, and the filter cake moisture decreased from 86.90% to 56.52%. The sludge conditioning mechanisms with MCFA mainly included improving floc formation through charge neutralization and adsorption bridging and providing the water transmitting passages by skeleton builder.

1. Introduction

Activated sludge technology has been widely applied to treat municipal and industrial wastewater, but it has a serious drawback of producing huge amounts of excess sludge. Together with more stringent regulations, more efficient sludge treatment techniques are demanded. As water content in the excess sludge is commonly more than 95% [1], reducing sludge volume by dewatering is economically valuable. Performance of sludge dewatering significantly depends on sludge properties, for example, the amount and composing of extracellular polymeric substances (EPS), the moisture content and distribution, the sludge particle size, etc. [2]. Sludge dewatering has been considered as one of the most expensive and the least understood process [3,4].

Traditionally, the excess sludge is conditioned to improve its dewatering properties, and then dewatered by mechanical force. In practice, chemical conditioning is most widely applied. It is well known that the mechanism of chemical conditioning is to destroy the colloidal frame of sludge, and flocculate the sludge flocs by added conditioners, such as calcium oxide, ferric chloride, polyacrylamide, etc. [2,5,6]. However, the conditioning chemicals are expensive and contribute significantly to the overall sludge management cost, so it is very significant to find cheap and effective conditioners.

Coal fly ash is the waste product of coal-burning power plants. Worldwide generation of coal fly ash is about 500 million tons per year [7], which may cause serious environmental problems. In recent years, coal fly ash has been used as cement additive, structural filler, and for road base stabilization [8–10]. Some researchers tested the utilization of coal fly ash as adsorbent to remove heavy metals from wastewater [7,11–13]. Because of insufficient adsorption capacity, coal fly ash was often modified by soaking in acid or alkaline solution. The chemically modified coal fly ash showed larger specific surface area and higher pore volume with low energy consumption, especially when soaked in acid solution [14–16]. However, coal fly ash recycling and reuse is still not much enough, and little attention has been paid to sludge conditioning by coal fly ash. Wang and Viraraghavan [17] conditioned sewage sludge with fly ash in order to fix heavy metals. Yang et al. [18] and Wang et al. [19] conditioned sludge from water works and sewage sludge with untreated coal fly ash, and the sludge dewaterability was greatly improved.

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Table 1
Chemical composition of the raw coal fly ash.

	Constituent									
	SiO_2	Al_2O_3	Fe_2O_3	K_2O	TiO_2	SO_3	Na_2O	CaO	MgO	MnO_2
Content %	54.83	28.79	3.77	4.94	1.71	1.23	1.30	1.22	0.92	0.02

This paper was to explore the possibility of improvement of the sewage sludge dewaterability by coal fly ash modified by sulfuric acid. The optimal conditions of coal fly ash modification were determined by orthogonal tests. The optimal conditions of conditioning and dewatering and related mechanisms were investigated. The results could provide a new approach for the practice of sewage sludge conditioning and coal fly ash utilization.

2. Materials and methods

2.1. Sludge characteristics

The excess sewage sludge used in this study was obtained from the inlet of the sludge thickening tank from Changsha Guozhen Municipal Wastewater Treatment Plant in Hunan, China. After 2 h settling the supernatant was removed, the sludge was stored at 4 °C as the experimental sample. Before conditioning and dewatering, the sludge sample was kept in a water bath at 20 °C for 30 min. Some characteristics of the experimental sludge were analyzed: dry solid content, $11.0 \pm 0.3\ g\,l^{-1}$; water content $(98.74 \pm 0.30)\%$; average sludge SRF $(1.86 \pm 0.71) \times 10^{13}\ m\,kg^{-1}$; filter cake moisture $(86.90 \pm 0.21)\%$; pH, 6.93 ± 0.22.

2.2. Coal fly ash modification

Coal fly ash used in the study was taken from Zhuzhou Huayin Thermal Power Station in Hunan, China, which was residue of anthracite combustion using a pulverized coal fired furnace with an exit gas temperature of 1140 °C. The coal fly ash was oven-dried at 105 °C for 4 h before use. Chemical composition of the raw coal fly ash (RCFA) was shown in Table 1.

Sulfuric acid was used as modification agent in this study. The modification conditions were optimized by the orthogonal experiments $L9(3^4)$ with the value of SRF as the target index. Acid concentration, ratio of acid to fly ash and soaking time with three levels were chosen in the orthogonal experiments (see Table 2). The dried coal fly ash was soaked in the sulfuric acid solution at room temperature as the orthogonal design. During coal fly ash soaking, the mixture was stirred with a speed of 30 rpm. After filtration of the mixture, the treated coal fly ash was dried at 105 °C to a constant weight, milled and sieved with 120 meshes. The modified coal fly ash (MCFA) was obtained.

In order to observe and compare the configuration of different coal fly ash samples, the RCFA and MCFA samples were tested on a field emission scanning electron microscope (FESEM) (JEOL JSM-6700F, UK), and their specific surface areas were measured with Automatic Chemisorption & Physisorption Analyzer (Autosorb 1C/TCD, USA).

2.3. Sludge conditioning and dewatering

The sludge conditioning with MCFA or RCFA was carried out as follows: a certain dosage of MCFA or RCFA was added to 100 ml sludge in a beaker of 200 ml. The mixture was rapidly stirred with a speed of 250 rpm for 30 s, and followed by a slow agitation of 30 rpm for 2 min. The MCFA or RCFA dosage was expressed as the weight ratio of MCFA or RCFA to sludge dry solids (%).

The raw sludge and the sludge samples conditioned with RCFA or MCFA were observed by microscope (OLYMPUS BX61, Japan).

The sludge conditioned was poured into a buchner funnel to filter or into centrifuge tubes to centrifuge. Filtration dewatering was done in a buchner funnel ($\Phi = 9$ cm) under 0.03 MPa vacuum pressure. The filterability of the sludge is described by the specific resistance to filtration (SRF):

$$\mathrm{SRF} = \frac{2PA^2b}{\mu\omega} \tag{1}$$

where SRF is the specific resistance to filtration ($m\,kg^{-1}$); P is the filtration pressure ($N\,m^{-2}$); A is the filter area (m^2); b is the slope of filtrate discharge curve (t/V versus V) ($s\,m^{-6}$); μ is the viscosity of the filtrate ($N\,s\,m^{-2}$); ω is the weight of cake solids per unit volume of filtrate ($kg\,m^{-3}$).

The sludge cake produced by the buchner funnel filtration process were dried at 105 °C to determine the filter cake moisture.

Centrifugation was conducted at 2000 rpm for 5 min in a batch-type laboratory centrifuge (LDZ4, China). After centrifugation, turbidity of the supernatant was measured with a spectrophotometer (721, China).

Zeta potential of the sludge supernatant was measured with a Zetasizer Nano Instrument (ZEN3600, England).

2.4. Sludge settling

The conditioned sludge was poured into a measuring cylinder of 100 ml and settled for 90 min. The variation of the supernatant volume was recorded every 5 min.

Table 2
Orthogonal experiment ($L9(3^4)$) arrangement and results for coal fly ash modification (MCFA dosage, 455%).

	Variable levels			SFR ($\times 10^{11}\ m\,kg^{-1}$)
	Acid concentration ($mol\,l^{-1}$)	Ratio of acid to coal fly ash ($ml\,g^{-1}$)	Soaking time (h)	
1	2	1:1	1	4.59
2	2	3:1	2	3.66
3	2	5:1	3	2.02
4	4	1:1	2	2.13
5	4	3:1	3	1.72
6	4	5:1	1	2.93
7	6	1:1	3	3.62
8	6	3:1	1	4.01
9	6	5:1	2	3.69
K1	3.42×10^{11}	3.44×10^{11}	3.84×10^{11}	
K2	2.26×10^{11}	3.13×10^{11}	3.16×10^{11}	
K3	3.77×10^{11}	2.88×10^{11}	2.45×10^{11}	
R	1.51×10^{11}	5.67×10^{10}	1.39×10^{11}	

Table 3
Analysis of variance for the orthogonal experiments.

Source of variance	Degree of freedom	Sum of the deviation square	*F*-Rate	*F*-Critical value	Significance
Acid concentration	2	3.77×10^{22}	4.441	19.000	–
Ration of acid to coal fly ash	2	4.84×10^{21}	0.571	19.000	–
Soaking time	2	2.90×10^{22}	3.418	19.000	–
Pooled error	2	8.48×10^{21}			

All chemicals were analytic grade. The water used was distilled water. All measurements were repeated for three times and the average values were reported.

3. Results and discussion

3.1. Coal fly ash modification

Acid concentration, ratio of acid to coal fly ash and soaking time were considered to be the most important factors for coal fly ash modification [14,15]. Results of orthogonal experiments with these three influencing parameters were presented in Table 2. The dosage of MCFA in the sludge was 455%. Among all experiments the minimum SRF was obtained with acid concentration of 4 mol l^{-1}, ratio of acid to coal fly ash of 3:1 ml g^{-1} and soaking time of 3 h, and was 1.72×10^{11} m kg^{-1}. According to *R*-value, the order of the influence on SRF was: acid concentration > soaking time > ratio of acid to coal fly ash, and *R*-value for acid concentration and soaking time were much higher than that for ratio of acid to coal fly ash. Furthermore, from the analysis of variance for orthogonal experiments in Table 3, we can see that the order of sum of the deviation square was the same as that of *R*-value: acid concentration > soaking time > ratio of acid to coal fly ash. Although the effect of all three factors was not significant, but the *F*-rate for acid concentration of 4.441 and for soaking time of 3.419 were much higher than that for ratio of acid to coal fly ash, which was only 0.571. So the acid concentration and the soaking time played important roles in coal fly ash modification. According to *K*-value, the optimum modification conditions were as follows: acid concentration, 4 mol l^{-1}; ration of acid to coal fly ash, 5:1 ml g^{-1}; soaking time, 3 h.

3.2. Effect of coal fly ash dosage on filtration dewatering

The SRF of the experimental sludge was around 1.86×10^{13} m kg^{-1}, showing a poor dewaterability. The MCFA modified with the optimum conditions (acid concentration, 4 mol l^{-1}; ration of acid to coal fly ash, 5:1 ml g^{-1}; soaking time, 3 h) was used to condition the sludge, and the influence of the coal fly ash dosage on sludge filtration dewatering was shown in Fig. 1. It was found that the sludge SRF dramatically decreased with the increase of the coal fly ash dosage, and the SRF of sludge conditioned with MCFA reduced much more rapidly than that with RCFA. With a MCFA dosage of 91% the sludge SRF decreased by 57.6% and 89.9% conditioned with RCFA and MCFA, respectively. With a MCFA dosage of 273% the sludge SRF reached 4.23×10^{11} m kg^{-1}, indicating that the dewaterability of the conditioned sludge was significantly improved. When the coal fly ash dosage exceeded 273%, the sludge SRF reduced insignificantly with the increase of coal fly ash dosage. Therefore, the coal fly ash was a good conditioner, and the modification with sulfuric acid promoted greatly its conditioning effect.

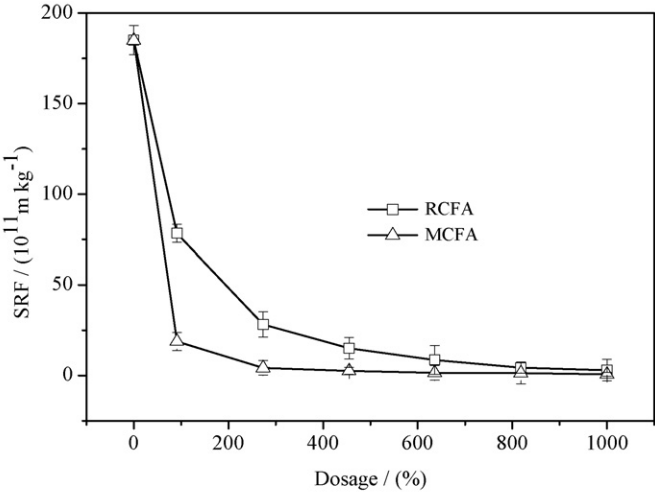

Fig. 1. Effect of coal fly ash dosage on sludge SRF.

The filter cake moisture with different MCFA dosages after sludge filtration dewatering was shown in Fig. 2. The filter cake moisture decreased as the MCFA dosage increased. With a MCFA dosage of 273% the filter cake moisture decreased from 86.90% (raw sludge) to 62.61% (conditioned sludge). Although the filter cake moisture per unit of sludge from calculation and the filtered water volume from experiments had no significant change, the filtration speed was more rapid when the sludge was conditioned with MCFA. To get 60 ml filtered liquid from 100 ml raw sludge, the filter duration of 300–315 s was need, but with a MCFA dosage of 273% the filter duration reduced to about 55 s. The raw sludge contained a lot of fine particles [20], the particles may be deformed and the porosity of filter cake gradually decreased, so the liquid permeability of the filter cake formed by these fine particles was poor during the filtration process. The MCFA particles were firm and their surface was irregular. The MCFA addition improved the cake porosity through forming channels or pores between the MCFA particles and sludge particles because of different particle size distributions, thus, the filtration resistance decreased. Karr et al. (1978) reported that the SRF of biological sludge was superficially dependent on the solids concentration [21]. We tested the effect of the sludge concentration

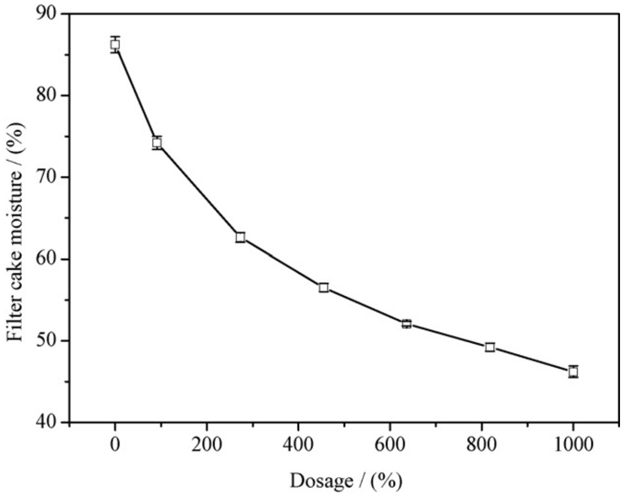

Fig. 2. Effect of MCFA dosage on filter cake moisture.

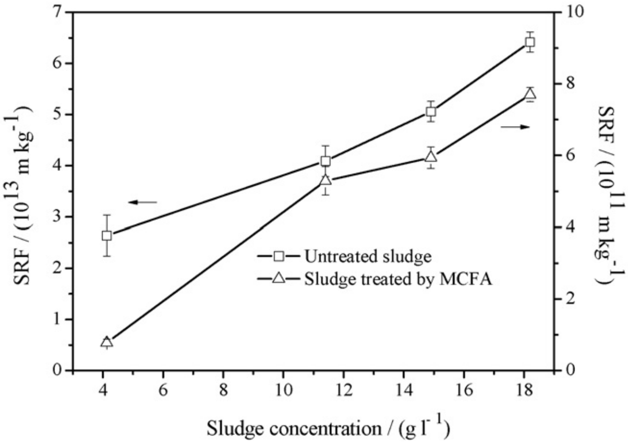

Fig. 3. Effect of sludge concentration on sludge SRF.

on sludge SRF, and added 3 g MCFA to 100 ml sludge with different sludge concentrations to verify the conditioning function of the MCFA. The results in Fig. 3 showed that the SRF of the raw sludge increased by 1.3 times with the increase of sludge concentration from 4 to 18 g l^{-1}. After conditioned with MCFA, the sludge SRF reduced by 76.3 times with the sludge concentration of 11.0 g l^{-1}. Therefore, the influence of the sludge concentration on sludge SRF can be ignored.

3.3. *Effect of coal fly ash dosage on centrifugal dewatering*

After the sludge conditioned with coal fly ash was centrifuged, the water volume generated was in the range of 85–90 ml, and there was no obvious regulation. Turbidity of the supernatant can be used as an auxiliary index to evaluate the centrifugal dewatering effect. The lower the turbidity is, the better the flocculation and adsorption capacity of fine particles is. After centrifugal dewatering, the turbidity of the supernatant was shown in Fig. 4. When the raw sludge was centrifuged, the supernatant turbidity was 234.2 NTU. When the sludge was conditioned with RCFA, the supernatant turbidity showed insignificant variation. With the addition of MCFA, the supernatant turbidity decreased obviously. The turbidity reached the lowest with 42.8 NTU with a MCFA dosage of 455%, because fine particles were effectively adsorbed by flocs formed between MCFA and sludge particles. However, when the dosage was higher than 455%, the turbidity increased slowly with the increase of MCFA dosage again.

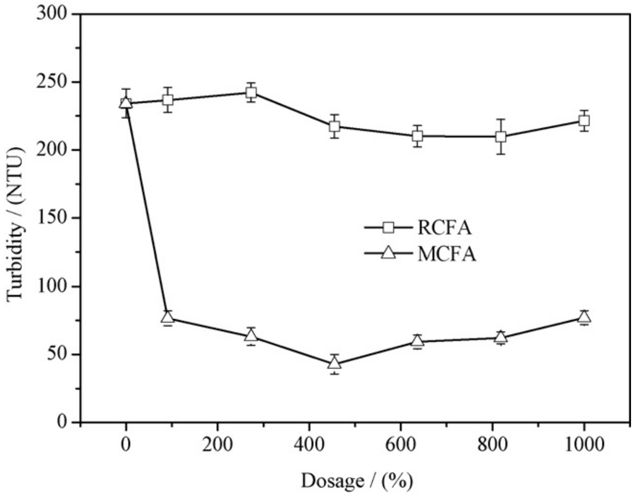

Fig. 4. Effect of coal fly ash dosage on supernatant turbidity.

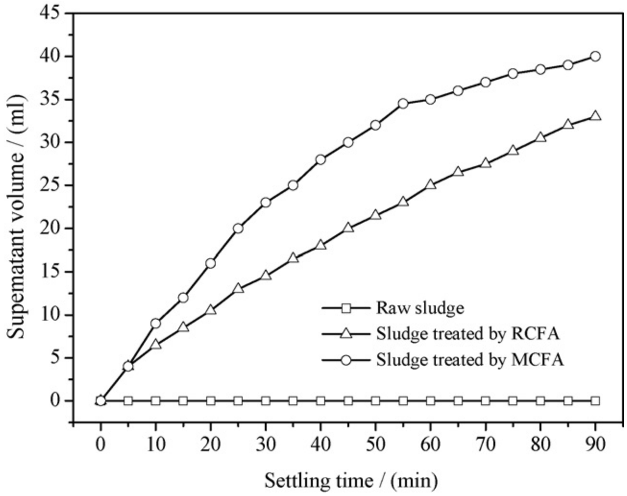

Fig. 5. Settling curve of sludge after conditioning by coal fly ash (coal fly ash dosage, 273%).

3.4. *Settleability of conditioned sludge*

The settleability of the conditioned sludge with MCFA was studied. Variation of the supernatant volume during 90 min settling time was illustrated in Fig. 5. As comparison, the settling situation of the raw sludge and the sludge conditioned with a RCFA dosage of 273% was also observed. The settling performance of raw sludge was extremely poor, and we could hardly observe the settling phenomena and the clear interface between liquid phase and solid phase. While the sludge was conditioned with MCFA and RCFA, the settling interface was very clear. The results showed that the settling velocity with MCFA was much higher than that with RCFA. After 30 min settling, the supernatant volume was 20.0 and 14.5 ml with MCFA and RCFA, respectively. After 90 min settling the supernatant volume reached the maximum without significant difference with MCFA and RCFA. The images of 1–3 in Fig. 6 show the settling of the raw sludge, the sludge conditioned with a RCFA and MCFA dosage of 273% after 60 min settling, respectively. Conditioning the sludge with RCFA or MCFA resulted in an improvement of the sludge settleability. MCFA could more effectively improve the settleability velocity of the sludge than RCFA. Fig. 7 shows that the suitable MCFA addition limit for improvement of the settleability

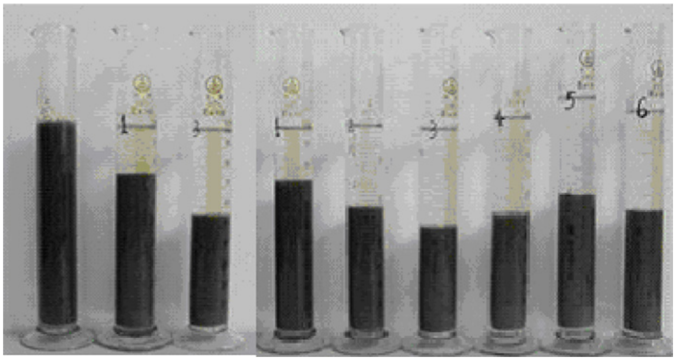

Fig. 6. Sludge settling images of raw sludge, sludge conditioned with RCFA and MCFA (settling time, 60 min). From left to right: 1, raw sludge; 2, sludge conditioned with a RCFA dosage of 273%; 3, sludge conditioned with a MCFA dosage of 273%; 4–9, sludge conditioned with a MCFA dosage of 91%, 273%, 455%, 636%, 818% and 1000%, respectively.

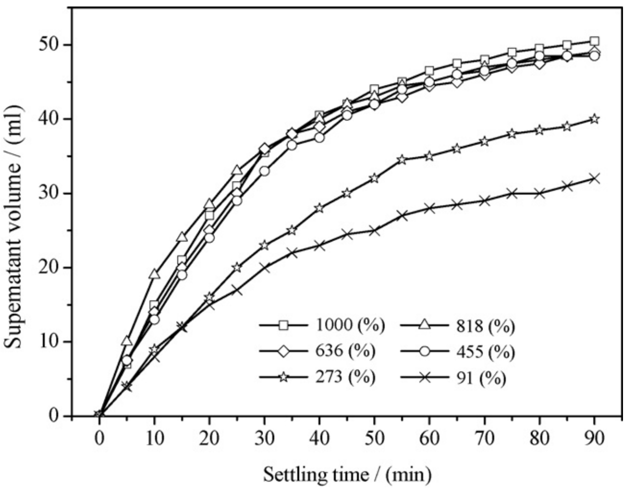

Fig. 7. Settling curve of sludge with different MCFA dosages.

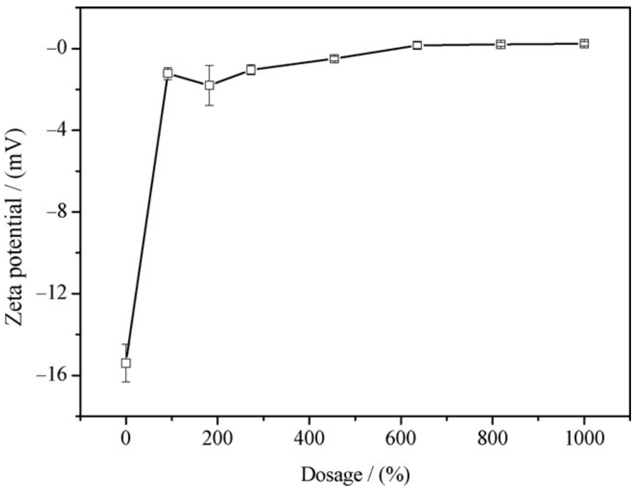

Fig. 9. Effect of MCFA dosage on Zeta potential of sludge supernatant.

was 636%, and higher MCFA addition could not improve the sludge settleability more. The sludge settling with different MCFA dosages after 60 min settling was described with the images 4–9 in Fig. 6.

The MCFA addition into sewage sludge significantly improved the sludge dewaterability, but the MCFA dosage was high, leading to the larger sludge volume and higher cost. In the future research and application the combination of MCFA with chemical conditioners, such as polymers, should be studied.

3.5. Possible mechanisms of sludge dewaterability improvement by MCFA conditioning

Normally, coal fly ash particles are spherical, and exhibit smooth surface texture. The size of coal fly ash particles is in the range from 0.01 to 0.1 mm [19,22]. From Table 1, we knew that SiO_2 content in the coal fly ash was the highest with 54.83%, and was followed by Al_2O_3 content with 28.79%. Therefore, there were lots of silicon and aluminum active sites in coal fly ash. FESEM images of RCFA and MCFA samples were shown in Fig. 8. RCFA had a shape of smooth microspheres (Fig. 8a). After modified with sulfuric acid (acid concentration of 4 mol l^{-1}, ratio of acid to coal fly ash of 5:1 ml g^{-1} and soaking time of 3 h), the surface of ball-shaped MCFA particles became very rough (Fig. 8b). The strong corrosive capacity of sulfuric acid changed greatly the surface of coal fly ash microspheres, and a portion of micropore may be attributed to the dissolution of CaO in coal fly ash particles. The specific surface area of coal fly ash significantly increased after modified with sulfuric acid, and the specific surface area of RCFA and MCFA was 2.810 and 3.376 $m^2 g^{-1}$, respectively. The experimental data were consistent with literature reported [15,23,24]. The increase of the specific surface area of the MCFA led to the improvement of fine sludge particle adsorption onto the MCFA, and the performance of sludge dewatering consequently improved.

The excess sludge particles were considered negatively charged, and excluded each other because of electrostatic interaction, which formed a relatively stable system, thus causing poor settling and dewatering performance of the sludge [6,25–27]. Fig. 9 illustrated the effect of MCFA addition on the Zeta potential of sludge supernatant. The Zeta potential for raw sludge was −15.4 mV; after conditioned with MCFA, the Zeta potential increased to 0 mV with the increase of the MCFA dosage. A lot of inner silicon and aluminum active sites with positive charge might emerge on the surface after coal fly ash modification, for example, aluminum silicate, ferric metasilicate and calcium silicate [28]. The negative charge of sludge was counteracted by the positive charge from aluminum silicate, ferric metasilicate and calcium silicate on the MCFA surface, namely charge neutralization for sludge particles. As a result, the stability of colloidal sludge particles was destroyed, and the colloidal particles congregated with each other, improving the sludge dewaterability and settleability.

Owing to the special structural characteristics, MCFA promoted the flocculation of destabilized particles acted as cores to form

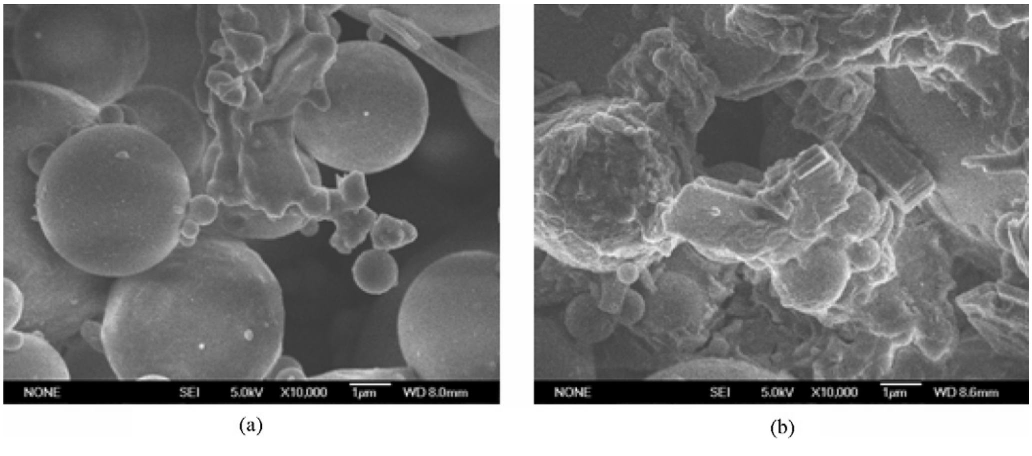

Fig. 8. FESEM images of RCFA (a) and MCFA (b).

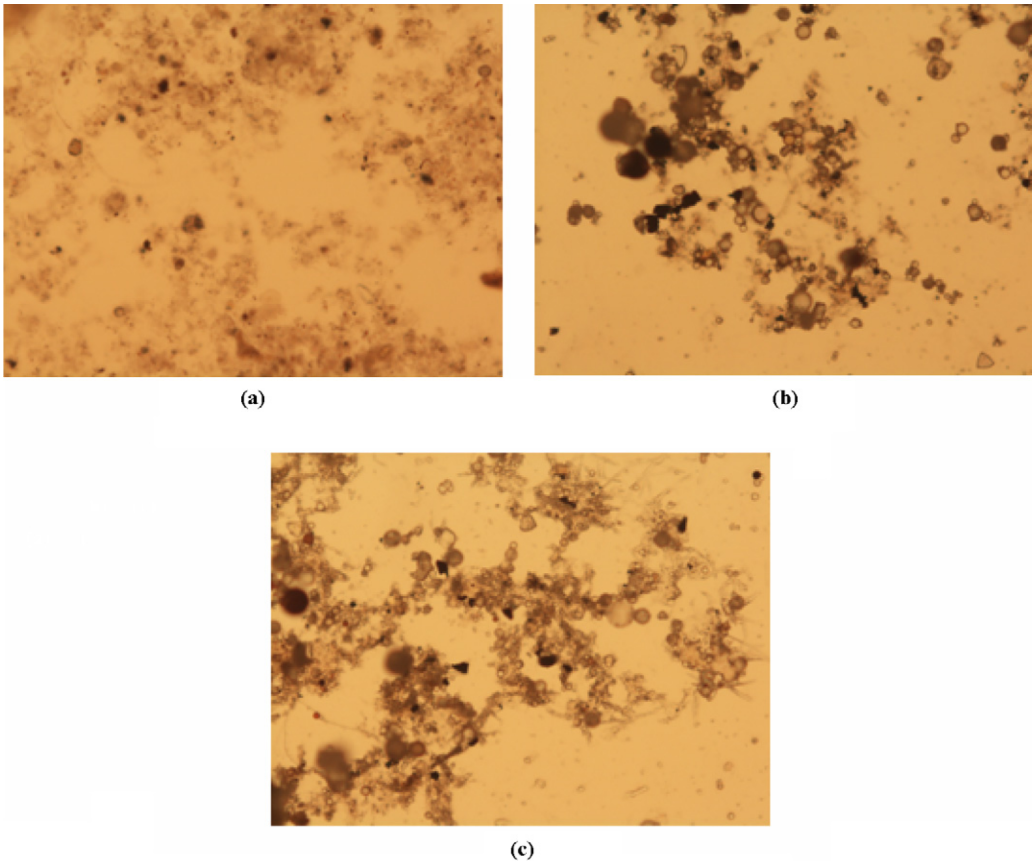

Fig. 10. Micrographs of the raw sludge (a), sludge conditioned with RCFA (b) and with MCFA (c) (magnification: 10 × 10).

floc framework aggregation. Micrographs of the raw sludge, the sludge conditioned with RCFA and MCFA were shown in Fig. 10. The raw sludge particles presented a dispersion state, and there were few flocs (Fig. 10a). After conditioned with RCFA, the sludge particles congregated around coal fly ash microspheres, and some flocs were formed. The floc size was small, and the flocs were dispersed (Fig. 10b). The best result appeared in Fig. 10c, where the sludge was conditioned with MCFA. We observed floc network with MCFA as cores, furthermore, the floc size was bigger. When sludge particles congregated with each other, MCFA microspheres adsorbed and bridged the sludge particles or the flocs, and enhanced the particle collision and floc growth. Besides, the strength of the new flocs was enhanced, and floc breakage induced by shear stress weakened. So the block of filter cake pores by fine particles was inhibited.

When the sludge was compressed and the sludge cake grew, a highly compressible sludge might deform under positive pressure, thus the size of the micro-passages reduced, through which water was released. After the sludge was conditioned with coal fly ash, the coal fly ash acted as skeleton builders, formed a permeable and rigid lattice structure and altered the sludge compressibility that remained porous under high pressure [29–32]. When the coal fly ash was modified, the surface structure and characteristics of the MCFA changed (as Fig. 8b), but the permeable and rigid lattice structure structural framework formed was unchanged. Moreover, the rough surface of MCFA might provide more passages, which retained solid particles and allowed the water to be transmitted, further improving the water release.

Therefore, the main mechanisms of sludge conditioning with MCFA might include improving floc formation through charge neutralization and adsorption bridging and providing the water transmitting passages by skeleton builder. The more detailed mechanisms need to be explored further.

4. Conclusion

The coal fly ash was modified with sulfuric acid in order to improve the sewage sludge dewaterability. The orthogonal experiments (L9(3^4)) with SRF as the target index showed that acid concentration and soaking time played important roles in coal fly ash modification and the optimal conditions were: acid concentration, 4 mol l^{-1}; ration of acid to coal fly ash, 5:1 ml g^{-1}; soaking time, 3 h. The coal fly ash modified with sulfuric acid (MCFA) showed a much stronger conditioning capacity than raw coal fly ash (RCFA). With a MCFA dosage of 273%, SRF of the sludge decreased from 1.86×10^{13} to 4.23×10^{11} m kg^{-1}, and the filter cake moisture decreased from 86.90% to 56.52%. The centrifugal dewatering and settling tests showed that MCFA improved the centrifugal dewaterability and settleability of the sludge. The main sludge conditioning mechanisms with MCFA might be improving floc formation through charge neutralization and adsorption bridging and providing the water transmitting passages by skeleton builder.

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