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A study on the deep dewatering of urban dewatered-sewage sludge by aluminum chloride

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ABSTRACT

Due to its composition and biological nature, dewatered sludge (water content was 80%) is usually highly compressible and known to be difficult to dewater. In this paper, deep dewatering of urban dewatered-sewage sludge (DD-UDSS) using different dosages of inorganic flocculant AlCl₃ was investigated. Dewaterability of the DD-UDSS was determined in a series of sludge dewatering tests. Their physicochemical characteristics were evaluated in terms of thermogravimetry, zeta potential, transmission electron microscope, and particle size analysis. Results revealed that the moisture content (MC) of DD-UDSS was decreased, at the same time the positive electricity on surface of DD-UDSS gradually increased with the increase in AlCl₃ dosage. The minimum MC was observed at 54.03% (8 g AlCl₃/300 g UDSS), which was relatively low as compared with the urban dewatered-sewage sludge (UDSS) of 81.2%. Additionally, the structure of the microstructure as well as the particle size was also changed by the addition of AlCl₃. These results suggested that with the addition of AlCl₃, the physicochemical characteristics of the DD-UDSS were improved; thus, its dewaterability was improved.

Keywords: Sludge; Dewatering; AlCL₃; TG; Zeta potential; Particle size; TEM

1. Introduction

In wastewater treatment processes, large amounts of sludge commonly containing water over 95% were produced [1]. In China, the total volume of urban sewage treatment across the country reached 0.136 billion m^3/d , and the production of dewatered sludge (water content 80%) reached 30 million t/a. Sludge with as

high as 80% moisture content (MC) was not treated properly and it was very difficult to perform further deep dewatering, thus posing a potential threat to the environment [2]. During the wastewater treatment process, waste-activated sludge dewatering usually adopted the mechanical dewatering conditioned with dehydrant like cationic polyelectrolyte polyacrylamide (PAM) in small and medium-sized sewage treatment plants in China [3]. The MC of urban dewatered-sewage

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sludge (UDSS) is still as high as 80%, which causes great difficulties for further disposal, and recycling of dewatered sludge. Therefore, further reduction of sludge MC to decrease sludge volume and quantity is significant to sludge treatment, disposal, and recycling [4]. The large quantity of UDSS is a serious problem to urban sewage treatment plants as well as the whole community. In order to achieve further disposal and recycling, it is necessary to study the deep dewatering (further drying) of urban dewatered-sewage sludge (DD-UDSS).

Sludge is a colloidal system in a stable suspension state, which is formed by large numbers of tiny sludge particles in water. Because UDSS performed like shiny stuff and water molecules were encapsulated by sludge colloid particles, it was extremely difficult to separate sludge particles from the water phase of UDSS [5]. At present, sewage sludge drying methods mainly include natural drying, thermal drying, and mechanical dewatering. Natural drying is the simplest and the most energy-efficient method, while it requires a long operation period and large space, and its secondary pollution is hard to control [6]. Similarly, the thermal drying method can also cause severe secondary pollution. Large amounts of different waste gases are emitted in the thermal drying process [7]. Additionally, energy consumption of the thermal drying is the highest among the three methods [8]. In contrast, the mechanical dewatering method involves lower operating cost and energy consumption than the thermal drying method [9]. However, due to the sludge MC of about 75-80%, it would be difficult to perform further dewatering and to select a suitable dehydrating agent for the sludge [10]. Currently, researches are mostly focused on the dewatering of sewage waste-activated sludge. There are few studies on deep dewatering of urban dewatered-sewage sludge (DD-UDSS). Liu et al. [11] studied the influence of the lime treatment on the deep dewatering of dewatered sludge, and they found that the moisture of dewatered sludge was significantly influenced by the lime treatment, and the MC could be reduced to 60% by conditioning 25% of lime dosage. Although the moisture of the sludge decreased significantly, 25% of lime dosage greatly increased the cost and the quantity of the waste. Therefore, it is necessary to find a dehydrating agent with low dosage and low cost.

In this paper, the dewaterability of UDSS at various AlCl₃ dosages was studied by MC test of DD-UDSS. Furthermore, the physicochemical properties of UDSS were characterized by TG test, micro electrophoresis, Malvern laser particle size analyzer, and transmission electron microscope (TEM) to study the influence of various AlCl₃ dosages on moisture distribution, zeta potential, particle size distribution, and morphological structure. Based on the results, the dewatering mechanism was also clarified.

2. Materials and methods

2.1. Materials

The UDSS employed was obtained from the Guangda Waste Water Treatment Factory, Jinan (Shandong, China). The UDSS was waste-activated sludge after flocculation treatment and dewatering process by cationic polyelectrolyte polyacrylamide. The sludge samples were kept in a sealed storage at room temperature about at 20°C and used within two-weeks. The characteristics of UDSS are presented in Table 1. The sludge was mixed with a suitable amount of water. After stirring the sludge mixture at the same mixing speed until it was well blended, particle size was measured by Malvern laser particle size analyzer.

Inorganic flocculants aluminum chloride was used to enhance the dewaterability of DD-UDSS. Six different dosages (0, 2, 4, 6, 8, and 10 g) of aluminum chloride solution about 20 mL were mixed fully with 300 g of UDSS. After 15 min of settling, the MC of DD-UDSS, thermogravimetry (TG), zeta potential, particle size, and TEM were measured, respectively.

2.2. Sludge dewatering

The experimental equipment for DD-UDSS is shown in Fig. 1. Filter cartridge was used to hold the dewatered sludge encased by filter cloth. The filtrate flowed out through holes on the filter cartridge under the pressure of jack. The filtrate was received by water box. The dewatering performance of DD-UDSS was studied by measuring the MC of filter cake (the sludge cake after dewatering).

The MC of filter cake is determined by using the following equation.

$$MC(\%) = \left(\frac{M_{\rm CT} - M_{\rm ad}}{M_{\rm CT}}\right) \times 100 \tag{1}$$

where $M_{\rm CT}$ is the weight of filter cake and $M_{\rm ad}$ is the weight of filter cake after drying for 1 h in the drying oven of 105°C [12].

2.3. TG test

The UDSS conditioned with different dosages of AlCl₃ was used to study its moisture distribution by TG test (SDT Q600 V8.3 Build 101) with carrying gas

Table 1 Characteristics of the UDSS

Sample	pН	Moisture content (%)	Ash content (% db)	Organics content (%)	Zeta potential (mV)	Mean particle size (µm)
Sludge	7.95	81.2	17.67	8.65	-41.5242	62.7



Fig. 1. Schematic diagram of the deep dewatering of dewatered sludge.

of pure N₂. Cell temperature was increased from 10 to 110° C at a rate of 15° C/min and the temperature of 110° C was maintained for 60 min.

 $M_{\rm sn}$ is the moisture weight of product at each moment. It is determined by using the following equation.

$$M_{\rm sn} = G_n - G_N \ (n = 0, 1, \ldots)$$
 (2)

 G_n is the weight of sludge at each moment and G_N is the equilibrium weight.

The dry-basis MC at each moment X_n is determined by using the following equation.

$$X_n = M_{\rm sn}/M_g \ (n = 1, 2, \ldots)$$
 (3)

 M_g is the equilibrium weight of sludge product.

The drying rate of sludge V_n is determined by using the following equation.

$$V_n = \frac{(X_n - X_{n-1})}{\Delta t} \ (n = 1, 2, ...)$$
(4)

 Δt is the span of time during the TG test [13].

2.4. Zeta potential measurements

The zeta potentials of UDSS were measured by microelectrophoresis (JS94H, Zhongchen Digital Technology Equipment Ltd, Shanghai). The sludge sample (0.229 g) was added to 0.01 M NaCl solution of 50 mL, stirred for 15 min, and its zeta potential was measured at last. The zeta potential values presented were obtained from the average of at least five measurements and the standard deviation was considered as the error range.

2.5. Particle size analysis

Particle size analysis was performed by Malvern laser particle size analyzer (Mastersizer 2000, Malvern Instruments Ltd, England), based on laser diffraction principles. The sludge sample of 2 g was used to measure the particle size and mixed with 250 mL deionized water. The mixture was stirred for 10 min by magnetic stirrer and was used to measure the particle size of the sludge sample. For analysis, each sludge sample was diluted to meet the requirements of instrument measurement. Each sample was measured at least 10 times and only the mean values were shown. The experiment results revealed the particle size distribution of the UDSS conditioned with AlCl₃ of different dosages. The recorded values were median diameter d (0.5), which means those smaller or greater than its particles each account for 50% in the sample.

2.6. TEM tests

A suitable amount of UDSS conditioned with AlCl₃ was mixed with absolute ethyl alcohol. The sample of mixture was used for TEM tests (TEM, JEM-1011).

3. Results and discussion

3.1. Dewatering of the sludge

The effect of different $AlCl_3$ dosages on sludge dewaterability of DD-UDSS was investigated. The MC of filter cake at various $AlCl_3$ dosages is shown in Fig. 2.

Within the range of AlCl₃ dosage smaller than 8 g AlCl₃/300 g UDSS, the MC of filter cake decreased as the dosage of AlCl₃ increased from 73.41 to 54.03%. When the dosage of AlCl₃ was 8 g AlCl₃/300 g UDSS, the dewaterability of DD-UDSS reached the maximum (the MC of filter cake was 54.03%). And then within the range of AlCl₃ dosage greater than 8 g AlCl₃/ 300 g UDSS, the MC of filter cake increased with the increase in AlCl₃ dosage. The results above indicated that AlCl₃ was a suitable dehydrant in the deep dewatering process with a good dewatering performance. The quantity of AlCl₃ at the optimum dosage (8 g AlCl₃/300 g UDSS) was relatively small; thus, it is feasible in practical dewatering process.

3.2. TG test

The TG method is most commonly used to measure the moisture distribute of sludge [14]. The dry rate curve of the sludge is shown in Fig. 3. The drying process of sludge consisted of the accelerated stage, the constant velocity stage, and the decelerated stage: (1) the accelerated stage: the sludge was heated up and the free water of sludge was evaporated at this stage; (2) the constant velocity stage: the surface of sludge was kept wet and the temperature was constant. The internal water migrated to the surface first



Fig. 2. The MC of filter cake at various AlCl₃ dosages.



Fig. 3. The dry rate curve of the sludge.

and then evaporated from the surface into the air. The external water evaporation rate of sludge was nearly equal to the rate of water diffusion from the interior to the surface of sludge. This stage was mainly for the evaporation of interstitial water; and (3) the decelerated stage: with the ongoing drying process, the rate of water diffusion from the interior to the surface of sludge was less than the external water evaporation rate of sludge at this stage. The surface water and the bound water of sludge were mainly evaporated here [15]. From the analysis above, it can be seen that when the drying rate reached the maximum, a larger portion of free water had less dry-basis MC.

The drying rate curves of DD-UDSS at various AlCl₃ dosages are given in Fig. 4. It can be seen that the drying rate increased continuously with the decrease in dry-basis MC, and then decreased quickly. Kopp and Dichtl [16,17] and Vaxelaire and Cézac [18] also studied the moisture distribution of sewage sludge, used thermo-gravimetric measurement to distinguish four different types of water (free water, interstitial water, surface water, and bound water), and found that the decrease in drybasis MC with a rapidly increasing drying rate approximately represented the free water content of the sludge. The drying rate of DD-UDSS without AlCl₃ reached the maximum value at greater drybasis MC than the DD-UDSS with AlCl₃. However, after the addition of AlCl₃, the dry-basis MC at the maximum drying rate of DD-UDSS decreased. This result indicated that DD-UDSS conditioned with AlCl₃ had more free water than DD-UDSS without AlCl₃. The addition of AlCl₃ changed the moisture distribution of UDSS, transforming other forms of water into free water.





Fig. 4. The drying rate curves of DD-UDSS at various AlCl₃ dosages.

3.3. Zeta potential of the sludge

The relationship between MC of filter cake, the zeta potential, and AlCl₃ dosage is shown in Fig. 5.

The surface of the urban sewage sludge was negatively charged [19]. The zeta potential of UDSS was -41.52 mV. The positive electricity on the surface of DD-UDSS gradually increased as the dosage of AlCl₃ increased, and the particle charge of the sludge was reversed at the dosage of about 5.5 g/300 g UDSS. Al³⁺ released by electrolyte AlCl₃ were adsorbed on the surface of sludge particles and its negative charge was neutralized; thus, the positive electricity of sludge particles was enhanced with the addition of AlCl₃ and the sludge particle charge may be reversed with excess electrolyte adsorption [20]. When the magnitude of zeta potential was between ± 10 and ± 30 mV, the particle of the sludge would become unstable, and when the magnitude of zeta potential was between 0 and $\pm 5 \text{ mV}$, the particle of the sludge would be condensed rapidly. As a whole, with the increase in positive electricity on the surface of DD-UDSS, the instability of the sludge particles was strengthened at first and then weakened. Thus, the dosage of AlCl₃ should be moderate.

Compared with the MC of filter cake at various AlCl₃ dosages in Fig. 5, some positive correlations were observed between the magnitude of the zeta potential and the MC of filter cake. The MC of filter cake decreased with the rise of the positive electricity on the surface of sludge particles. When zeta potential was 13.5 mV, the MC of filter cake reached the minimum value (54.03%). Luo et al. [21] found that the optimum flocculation was related to the decline of the magnitude of zeta potential and occurred when zeta potential value decreased to the isoelectric point by



Fig. 5. The relationship between MC of filter cake, the zeta potential and $AlCl_3$ dosage.

charge neutralization, but the optimum dewatering performance did not occur here. It was probably because the improvement of the dewaterability was not only affected by charge neutralization but also by other factors.

3.4. Analysis of TEM and particle size

Fig. 6 shows the TEM micrograph of UDSS. As indicated by the arrows, the dispersed cloudy matter was extracellular polymeric substance (EPS) [22]. Large numbers of EPS dispersed homogeneously and the whole microstructure of dewatered sludge presented the structure of reticular formation. It was because UDSS was conditioned by cationic polyelectrolyte polyacrylamide and dewatered before, and the sludge particles had already flocculated during the first dehydration. The interior of the floc structure still had numerous voids visible and large amounts of water were wrapped. Due to this structure of the sludge floc, the sludge still showed relatively low-compressive strength [23].

The TEM micrograph of UDSS conditioned with AlCl₃ (8 g/300 g UDSS) is given in Fig. 7. Compared with the TEM micrograph of UDSS, the agglomerate of UDSS conditioned with AlCl₃ flocculating with a large number of microcosmic sludge particles was larger, more compact, and better flocculated. The microstructure of sludge particles turned into globular structure and EPS scattered in UDSS were all flocculated as well. The phenomenon above was the reason why the negative charge on the surface of sludge particles was neutralized and the negative charge repulsion in sludge particles decreased with the addition of AlCl₃. Therefore, sludge particles flocculated together



Fig. 6. TEM micrograph of UDSS.



Fig. 7. TEM micrograph of UDSS conditioned with $AlCl_3$ (8 g/300 g UDSS).

and formed bigger agglomerate. Because of the further flocculation of sludge particles, the interstitial water among sludge particles turned into free water and finally changed the moisture distribution of the sludge.

With the addition of AlCl₃, the microstructure of the sludge changed a lot as well as the particle size. Fig. 8 shows the particle size of UDSS and the MC of filter cake at various AlCl₃ dosages. The floc size was an important factor with respect to the dewatering of the sludge, because increased floc size led to the exposure of fewer surfaces, weakening the hydrophilicity of flocs of the sludge and thus, contributing to the obvious improvement in the dewaterability [24]. The particle size (median diameter) of UDSS was $62.7 \,\mu$ m. With the increase in AlCl₃ dosage, the particle size of



Fig. 8. The particle size of UDSS and the MC of filter cake at various $AlCl_3$ dosages.

sludge decreased first when the dosage of AlCl₃ was 2 g/300 g UDSS and then increased. When the AlCl₃ dosage was 8 g/300 g UDSS, the particle size reached the maximum value (299.56 µm). Nevertheless, the particle size of the sludge decreased again with the increase in the AlCl₃ dosage after 8 g/300 g UDSS. Considering the microstructure change of the sludge for analysis, the decrease in the sludge particle size in the beginning might be due to the shrinkage of the net structure after the addition of AlCl₃. With further flocculation of sludge particles with the increase in AlCl₃ dosage, the agglomerate was becoming larger and larger, and reached the maximum value (299.65 µm) at last. Floc size increased with the increase of the AlCl₃ dosage, which was mainly due to bridging and charge neutralization [25]. With continuous addition of AlCl₃, excess Al³⁺ absorbed on the surface of sludge particles enhanced its positive charge repulsion and the particle size of sludge finally reduced slightly.

Compared with the MC change of the filter cake at various AlCl₃ dosages, it is obvious that the MC of filter cake were proportional to the particle size of sludge. The dewatering performance of sludge was significantly influenced by sludge particle size. With the addition of AlCl₃, the positive electricity on the surface of DD-UDSS increased, and its negative charge was neutralized gradually. Accordingly the stability of sludge particles was weakened; the particle size of sludge increased and large amount of water wrapped by sludge particles turned into free water. Thus the free water content of sludge increased after the addition of AlCl₃. The dewaterability of sludge reached the maximum value at 8 g AlCl₃/300 g UDSS. When the dosage of AlCl₃ was excessive, the positive charge repulsion between sludge particles increased with the increase in AlCl₃ dosage, and the stability of sludge particles enhanced. Therefore, the particle size of sludge decreased and the dewatering performance of sludge reduced.

4. Conclusion

 $AlCl_3$ was selected for the deep dewatering of the sludge and the dewaterability of the sludge conditioned with $AlCl_3$ was investigated. The moisture distribution, zeta potential, morphological structure, and particle size of UDSS were characterized to provide a thorough understanding of dehydration mechanism.

The addition of $AlCl_3$ significantly improved the dewaterability of dewatered sludge. The MC of filter cake decreased from 73.41 to 54.03%. The results

indicated that AlCl₃ as dehydrating agent was feasible. The UDSS conditioned with AlCl₃ produced large amount of free water and changed the water distribution of sludge; thus, improved the dewatering performance of UDSS. The zeta potential of the sludge was changed and the stability of sludge particles was enhanced. The changes above were all beneficial to the deep dewatering of UDSS.

The UDSS presented the structure of reticular formation, and the agglomerate of UDSS conditioned with AlCl₃ was larger and more compact. The dewaterability of sludge was also significantly influenced by sludge particle size. The bigger particles of the sludge had better dewatering performance. Thus, the increase in the particle size was conducive to the deep dewatering of UDSS.

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