Settleability and Dewaterability of Sewage Sludge with Modified Diatomite

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Abstract

In this study, cationic polyacrylamide (CPAM) modified diatomite and cetyl trimethyl ammonium bromide (CTAB) modified diatomite were synthesized and used as conditioner in sewage sludge dewatering. The effects of these two types of modified diatomite on the dewaterability and settling performance of activated sludge were studied. The mechanism of the two types of modified diatomite in the activated sludge system was elucidated. The efficiency of CPAM-modified diatomite was better than that of CTAB-modified diatomite in improving the settleability and dewaterability performance of sludge. The results indicated that specific resistance to filtration (SRF) was decreased from $8.52 \times 10^{12} \text{ m/Kg}$ to $0.92 \times 10^{12} \text{ m/Kg}$, and the water content in the remaining sludge cake after pumping filtration was decreased from 92.2% to 68.1%. by adding 0.4% of CPAM-modified diatomite and pH=3.5, which result in optimal sludge settling of activated sludge. Further studies showed that the polymer/surfactant adsorbed in diatomite increased sludge dewaterability and improved the sedimentation rate as a result of stripping extracellular polymer substances (EPS) and damaging the internal structure of sludge conduce bound water releasing. According to scanning electron microscope(SEM) images, two types of modified diatomite powder not only kept the porous, but also shown more complete and uniform structure in comparison to nature diatomite.

Introduction

With the rapid development of urbanization and industrialization brought about the surge of sewage treatment, it is expected that by 2021, the annual output of excess activated sludge (wet sludge) in China will reach approximately 40 million tons (Ruan et al. 2021). The environmental pollution caused by a large amount of sludge cannot be ignored. The treatment and disposal of sludge is one of the key technologies and cost control of sewage treatment process (Peng et al. 2021). The difficulty of dewatering due to the high compressibility and low permeability of sludge filter cake is the bottleneck in sludge treatment system (Huang et al. 2015).

Nowadays, mechanical filter press is the most widely employed efficient method for sludge dewatering, which requires pretreatment to improve its dewaterability (Wu et al. 2020). Such as gypsum (Zhao 2002), zeolite (Liao et al. 2014), diatomite (Li et al. 2018) which can reduce the compressibility meanwhile improve the mechanical strength and permeability of the sludge cake, are regarded as the typical physical conditioners (Yang et al. 2016).

Chemical conditioning is another important means applied to benefit mechanical dewatering. The traditional chemical conditioner such as surfactant (Liu et al. 2020; He et al. 2020), flocculants and polymers (Hu and Chen 2021) promote the agglomeration of fine sludge particles into large flocs through charge neutralization or adsorption bridging, which is more conducive to precipitation and compression(Rao et al. 2019). EPS are necessary components of activated sludge which have a high affinity for water and thus are highly hydrated. It has been reported that surfactants can stimulate the release of EPS of to improve the settling capacity and dewaterability of sludge. (Fu et al. 2009). But the

massive surfactants or flocculants have disadvantages to the follow-up treatment and disposal, besides, some polymers increase the cost of sludge treatment. For example, ferric chloride as a typical flocculant is highly acidic and toxic, which easy to corrode equipment. It will increase the concentration of dioxin in incineration flue gas (Yang et al. 2016).

Nature diatomite ($\text{SiO}_2 \cdot n\text{H}_2\text{O}$), is a low-cost, environment-friendly and natural micro/nanostructured material, has stable performance in acid, high porosity, high specific surface area, and low cost which make it applicable as filter aid, catalytic support and adsorbent (Liu et al. 2018). Its rich mesoporous/microporous structure is good for the accelerating the moisture extraction between sludge particles and loading of modified materials for improving the sludge dewatering performance. However, natural diatomite directly used as a conditioner cannot fully meet the demand of the sludge dewatering and the environment yet. Pore size distribution, impurity content and bulk density are the main factors affecting the quality of catalyst carrier, so it is necessary to further improve the physical and chemical properties of diatomite (Danildenamor et al. 2012; Dong et al. 2020).

Previous studies focused on the adsorption of heavy metals from water by modified diatomite (Liu et al. 2018; Huang et al. 2020). Few research on the role of modified diatomite as a conditioner in sludge dewatering. The purpose of this study is to research the role of modified diatomite particles in the dewatering performance of activated sludge system. A typical polymer, cationic polyacrylamide(CPAM) and a typical cationic surfactant, cetyl trimethyl ammonium bromide(CTAB) were selected to modify diatomite. Taking the dosage of these two types of modified diatomite and pH as the control factors, the sedimentation and dewatering efficiency of sludge were evaluated from the sedimentation volume (SV), specific resistance to filtration (SRF), cake moisture content and EPS concentration. The structural differences between natural diatomite and modified diatomite as well as the morphology of dewatering sludge by adding them were investigated by scanning electron microscope (SEM).

Materials And Methods

Test materials

Sewage sludge samples were collected from Changsha Guozhen wastewater treatment plant, Changsha, China. The physical and chemical characteristic parameters of sludge are shown in Table 1. All chemicals were of analytical grade. Natural diatomite sample came from Chenzhou (Hunan Province, China). The water used here was deionized water.

Preparation of modified diatomite

The preparation process of modified diatomite according to the following procedures: nature diatomite was roasted in a muffle furnace at 450°C-550°C for 1-2h and then cooled to room temperature. The roasted diatomite (40g) and 5%-10% HCl (200 ml) were mixed 500ml beaker and then placed in a water bath at a temperature of 50°C-65°C and stir it for 2 h at a stirring speed of 150-300r/min. The sample was repeatedly washed and filtered with ethanol and deionized water until pH was neutral, and then dried

to constant weight. Weighed 20 g acidifying diatomite into a beaker, added 100 mL of 2 % CPAM/CTAB solution and then stirred for 2 h at the speed of 100 r/min at room temperature, followed by suction filtration, the sample was dried in a thermostatic oven for 2 h. After cooling, it was ground and passed through 100 mesh screen.

Sludge settling and dewatering

A certain dosage of the modified diatomite and 150 ml activated sludge mixture were added to a 200 ml beaker and blended violently by glass rod. After the mixture was completely stirred, 50ml of the sludge was taken for centrifugation, and the supernatant was prepared for EPS test. After that, the mixture was poured into 100 ml measuring cylinder. The volume of settling sludge was recorded every 5 min for 60 mins, that the settling volume of sludge (SV_{60}) was obtained. SRF was measured by vacuum extraction and filtration device. 100ml of sludge was poured into a standard 9cm Buchner funnel with pre-wetting quantitative filter paper at a constant vacuum pressure of 0.03 Mpa for 10-15 minutes. Determinations of SRF was performed according to Baskerville and Gale (Baskerville and Gale 1968). The water content of the sludge cake trapped by the filter paper was measured in accordance with standard methods (APHA 1998). Finally, dewatering sludge samples and modified diatomite powders were prepared for SEM.

Determination of EPS

Coomassie Brilliant Blue G-250 was used to determine the extracellular protein content in the filtrate with bovine serum protein as the standard protein, and its absorbance was measured at 595 nm (Marion 1976). Polysaccharide was stained with anthrone, and its absorbance was measured at 625 nm (Riesz et al., 1985).

Results And Discussion

Effect of modified diatomite on sludge settling performance

SV_{30} is an important index to evaluate sludge settling performance. In order to investigate the sludge sedimentation process more completely, 30 mins was extended to 60 mins in this study. Sludge settling volume by adding CPAM-modified diatomite with time was shown in Fig. 1. Nature diatomite, CPAM and CPAM-modified diatomite with sludge weight ratios of 0, 0.2%, 0.4% and 0.8%wt were added, respectively. For the sludge sample with modified diatomite added, the height of sludge settling basically reached stability before 40 min, while the settling speed of the sludge sample without conditioning was significantly lower, which had more than 60 mins to complete the whole settling process. The settling performance was best with adding 0.4% wt CPAM-modified diatomite. When the dosage of CPAM-modified diatomite was below 0.4% wt, the more added amount, the better sludge settling was obtained. Compared with 0.4%wt and 0.8% wt, there was no significant difference in the settling rate of the sludge, but the volume of settled sludge was lower at 0.4% wt because more conditioner might increase the volume of sludge. Meanwhile, it could be clearly observed that there were clusters of sludge floc floating on the surface of the liquid supernatant with 0.8% wt addition.

It was obvious that the sludge settling performance by adding CPAM-modified diatomite were all better than that of nature diatomite and CPAM. The addition of diatomite could increase the internal binding strength of sludge floc and prevent floc crushing. Meanwhile, CPAM could adsorb on the surface of negatively charged sludge particles. By compressing the double electric layer, the thickness of the hydration film on the surface of particles was reduced, and the repulsive force between sludge particles was reduced to form larger flocs. When the flocs grew to a certain extent, the additional external force and internal binding force reached a balance and no longer increased. At this time, the apparent density of the flocs also reached the maximum, and its settlement performance was the best. If there was excessive modified diatomite in the sludge, under the action of strong adsorption and bridge building, the formed flocs would combine the free water around them into larger aggregates. These aggregates contained a lot of free water, which was loose and had poor settlement performance, resulted in breaking sludge settlement. The effect of adding CPAM alone was slightly better than that of adding natural diatomite, and adding CPAM-modified diatomite realized the synergistic effect of these two.

Sludge settling volume by adding CTAB-modified diatomite with time was shown in Fig. 2. Nature diatomite, CTAB and CTAB-modified diatomite with sludge weight ratios of 0, 0.2%, 0.4% and 0.8%wt were selected, respectively. The sludge settling curve was similar to that of CPAM. Whether modified diatomite or natural diatomite, the more added amount, the faster the sludge settling. On the whole, CPAM-modified diatomite was better than CTAB- modified diatomite in improving the settling performance of sludge. Different from adding CPAM-modified diatomite, the sludge settlement rate and efficiency of adding 0.8%wt CTAB-modified diatomite were better than that of 0.4%wt, which was embodied that the final sludge volume was same, but the sludge settling speed was faster. The addition of surfactant increased the internal binding strength of sludge floc and prevent floc crushing. At the same time, by compressing the double electric layer, the thickness of the hydration film on the surface of particles was decreased, and the repulsive force between sludge particles was reduced to form larger flocs. It's worth noting that there were a few clusters of sludge floc floating on the surface of the liquid supernatant with 0.8% wt CTAB-modified diatomite addition, was much less than that of 0.8% wt CPAM-modified diatomite. An addition of 0.8% wt was optimal for CTAB-modified diatomite. Taking into consideration the cost of the modified diatomite, the dosage of 0.4% was determined as the feasible dosage.

Effect of modified diatomite on sludge dewatering performance

The water existed in sewage sludge is classified into four categories: free water (about 70%, not directly combined with sludge), interstitial water (about 20%, which is trapped inside cracks in the solid particle), surface water (adhered to the surface of fine sludge particles) and chemically bound water (inside microbial cells) (Feng et al. 2009).

Effect of dosage of nature diatomite, CPAM and CPAM-modified diatomite on SRF and cake moisture content were shown in Fig. 3. It is well known that SRF has been widely used as means of gauging sludge dewatering. However, for sludges with low SRF and easy to filter, the cake moisture content does not necessarily decrease due to the presence of chemically bound water. Therefore, SRF just represents the

difficulty level of filtration performance, moisture content of sludge represents the efficiency of filtration. In order to comprehensively evaluate the effect of modified diatomite on sludge dewatering performance, both SRF and moisture content were taken as evaluation parameters in this study. The effects of two modified diatomite on the SRF and water content of sludge were shown in Fig. 3 and Fig. 4. The raw sludge contains a large number of fine sludge particles, which enter into the pores of the filter cake, resulting in the reduction of porosity in the filtration process, lead to a poor permeability of the filter cake. It was confirmed that CPAM can enhance the flocculation effect of sludge through electrical neutralization and particle adsorption bridging. In conclusion, the sludge settling performance after CPAM pretreatment was significantly improved, and was continuously improved with the increase of CPAM dosage. Under the same conditions, CPAM was better than natural diatomite in reducing SRF, but not as good as natural diatomite in reducing cake moisture content. Therefore, CPAM and natural diatomite had their own advantages and disadvantages in reducing SRF and cake moisture content. CPAM-modified diatomite can effectively neutralize the advantages of these two, obtained a synergistic effect. As shown in Fig. 3, when the dosage of CPAM-modified diatomite was 0.6%wt, SRF sharply decreased from 8.52×10^{12} m/Kg(raw sludge) to 0.90×10^{12} m/Kg, the cake moisture content decreased from 92.2% to 68.5%. The modified diatomite added can increase the sludge floc size by flocculating fine sludge particles, thus improve the porosity of the filter cake, which was similar with adding polymeric flocculant. However, when the CPAM-modified diatomite dosage increased from 0.6%wt to 0.8%wt, SRF and the filter cake moisture did not change as significantly as before. Because the excess long-chain molecules squeezed each other and cannot extend freely, which also weakens its adsorption and bridging effect (Yang et al. 2016). Comprehensive consideration, 0.6%wt was the most suitable dosage for CPAM-modified diatomite.

Effect of dosage of nature diatomite, CTAB and CTAB-modified diatomite on SRF and cake moisture were shown in Fig. 4. As shown in Fig. 4, CTAB was better than nature diatomite both in reducing SRF and cake moisture content when addition was below 0.6%wt. When the dosage increased from 0.2%wt to 0.8%wt, the SRF and cake moisture content exhibited a stable decreased with nature diatomite. But the SRF no longer reduced and cake moisture content was rebounded with CTAB or modified diatomite when the dosage above 0.6%wt. In the process of suction filtration, when the amount of CTAB exceeded 0.4%, a large amount of foam appeared in the filtrate, which brought a negative effect on sludge dewatering. Due to the association between the hydrophobic components of the surfactant, it played the role of bridge and net to re-flocculate the suspended sludge particles, forming a larger sludge floc. This re-flocculated sludge floc structure was loose, free water can pass quickly, thus greatly improving the filtration speed. But excessive CTAB re-adsorbed part of the free water and wrapped it in the sludge flocs. The optimum dosage of the CTAB- modified diatomite in this study was about 0.6%wt, but taking into consideration the cost, the dosage of 0.4% was determined as the optimum dosage.

Effect of pH on sludge dewatering performance

Addition of acid to sludge was also demonstrated be benefit for filtration dewatering (Chen et al. 2001). It was observed that the water content was decreased from 75.3% to 68.1% and 81.5% to 70.0% for CPAM-

modified diatomite and CTAB-modified diatomite, respectively, on decreasing the pH in the range of 6.8–3.5. Meanwhile, SRF was decreased from 1.24×10^{12} m/Kg to 0.92×10^{12} m/Kg for CPAM-modified diatomite and 2.37×10^{12} m/Kg to 1.92×10^{12} m/Kg CTAB-modified diatomite on decreasing the pH in the range of 6.8–3.5. However, the water content and SRF showed that there was a drastic rise when pH was less than 3.5, which demonstrated that the optimum pH value for vacuum extraction and filtration dehydration was 3.5. The dominant reason why acidification treatment can significantly reduce SRF was that it can stimulate EPS to dissociate from activated sludge surface, making sludge aggregates easy to accumulate. However, in the case of pH less than 3.5, the dewaterability did not improve with the further increase of acidity because excessive EPS was released in a short period of time and blocked the filter paper. The effect of pH on SRF and cake moisture content with CPAM-modified diatomite was more significantly. Overall, pH value 3.5 was appropriate.

Effect of modified diatomite on EPS in supernatant

There are many factors affecting sludge dewatering performance, such as EPS concentration, particle size distribution, pH value, organic concentration and so on. Among them, EPS concentration has been comprehensive investigated and is considered as one of the key factors affecting sludge dewatering performance (Liu and Fang 2003).

EPS is a kind of macromolecular polymer, such as polysaccharide, protein and nucleic acid, which is secreted by microorganisms in vitro under certain environmental conditions, widely exists in the interior and surface of activated sludge floc. These compounds help to retain moisture and significantly improve the ability of sludge floc to bind to water. Therefore, we measured the concentration of polysaccharides and proteins in EPS to explore the effect of these compounds on dehydration performance.

Effect of modified diatomite on concentration of protein and polysaccharides were shown in Fig. 6. Proteins and polysaccharides were 41.4 and 8.3 mg/L, respectively, in the raw sludge sample. Both proteins and polysaccharides increased with increasing modified diatomite. At 0.8%wt CPAM-modified diatomite dosage, the increases in the levels of proteins and polysaccharides in filtrate were 169% and 329%, respectively, meanwhile, that for CTAB-modified diatomite were 119% and 270%, respectively. As observed in the present work, increases in EPS led to rapid increases in SRF, but did not always decrease sludge dewaterability. EPS have a high affinity for water and thus are highly hydrated. Adding the modified diatomite, EPS on the surface of sludge particles can quickly separate from sludge and dissolve in water under the influence of surfactant/coagulant, thus reducing the interstitial water, so that SRF and moisture content reduction. Nevertheless, high EPS concentrations lead to the viscosity of sludge increased (Wang et al. 2010) and dewaterability decreased (Chen et al. 2001). In vacuum filtration, these EPS gathered on the surface of filter paper to form a membrane, which blocks the passage of water. With the increase of modified diatomite, the sludge surface released more EPS into the water, resulting in obstruction of filtration.

SEM of modified diatomite and dewatering sludge

SEM images of natural and modified diatomite were shown in Fig. 7. According to the SEM shown in Fig. 7, natural diatomite was easy to break, the structure was not intact, and more impurities covered the surface. It obvious that both CPAM-modified and CTAB-diatomite were finer than natural diatomite, especially the impurities was significantly reduced, and the micropores were clearer and more uniform. This showed that the cleaning and roasting of the modified diatomite play a purification role, but the introduction of modified additives did not change the microporous structure of natural diatomite. According to SEM images, there is little difference between CPAM-modified diatomite and CTAB-modified diatomite in structure.

SEM images of dewatering sludge were shown in Fig. 8. By comparing the apparent morphology of the dewatering sludge with that prepared with CPAM-modified diatomite, it was obvious that the latter had better cohesion and forms larger holes, which was more conducive to water release. The raw sludge showed dense structure which obviously increased the resistance of water migration.

Conclusions

Two types of organic modified diatomite (CPAM/CTAB modified diatomite) were synthesized by roasting and acid modification. In summary, the settleability and dewaterability of sewage sludge can be improved significantly after conditioning with CPAM/CTAB modified diatomite. By adding modified diatomite, the sludge settling rate was increased and reached stability before 40 min, 0.4% wt were the optimal dosage both for CPAM-modified diatomite and CTAB-modified diatomite. The SRF and water content of sludge decreased the most when the modified diatomite dosage was from 0.2% wt to 0.6% wt. For CPAM-modified diatomite, when dosage was 0.4%wt, SRF of sludge was decreased from 8.52×10^{12} m/Kg to 0.92×10^{12} m/Kg, the cake moisture content of sludge dropped from 92.2–68.1%, which was better than that of CTAB-modified diatomite. When the dosage was more than 0.6% wt, the SRF value changed little, but the moisture content of filter cake increased. Addition of acid to sludge was also proven to be favorable to filtration dewatering. pH value 3.5 was the optimal in this study. SEM showed that the modified diatomite did not change the microporous structure of natural diatomite, but more complete and uniform powders were obtained. Meanwhile, SEM showed that the dewatering sludge with modified diatomite presented a structure more conducive to water passage. Overall, this research proved the potential of CPAM/CTAB modified diatomite for sewage sludge pretreatment in mechanical dewatering.

Declarations

Acknowledgments

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Data availability

The datasets used and/or analyzed during the current study are available from the corresponding author on reasonable request.

Compliance with ethical standards

Conflict of interest: The authors declare that they have no conflict of interest.

Ethical approval: This article does not include any studies involving human participants or animals. The authors declare that the manuscript has not been published previously.

Consent to participate: All authors voluntarily to participate in this research study.

Consent to publish: All authors consent to the publication of the manuscript.

Author contribution

LYW conceived and conducted experiment, performed, and analyzed data. XL initiated and supervised the project, drafted the manuscript. KL and YHS helped analyze the data and prepare the manuscript. RGL helped material preparation, data collection. All authors read and approved the final manuscript.

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Tables

Table 1 Characteristics of raw sludge

Mixed liquor suspended solids (MLSS) / g/L	Volatile solids (MLVSS) / g/L	Water content /%	Specific resistance /m/Kg	Turbidity of supernatant /NTU	pH	Protein in supernatant /mg/L	Polysacchari de in supernatant /mg/L
4.83 ± 0.026	2.52 ± 0.015	99.4 ± 0.4	8.5 × 10 ¹²	28.5 ± 0.5	6.7	41.4 ± 1.7	8.3 ± 0.24

Figures

Figure 1

Effect of dosage of nature diatomite, CPAM and CPAM-modified diatomite on sludge settling volume (N: nature diatomite; M: CPAM-modified diatomite)

Figure 2

Effect of dosage of nature diatomite, CTAB and CTAB modified diatomite on sludge settling volume (N: nature diatomite; M: CTAB-modified diatomite)

Figure 3

Effect of dosage of nature diatomite, CPAM and CPAM-modified diatomite on SRF and cake moisture content (r : specific resistance to filtration; w : cake moisture content; N: nature diatomite; M: CPAM-modified diatomite; pH3.5)

Figure 4

Effect of dosage of nature diatomite, CTAB and CTAB-modified diatomite on SRF and cake moisture content (N: nature diatomite; M: CTAB-modified diatomite; pH3.5)

Figure 5

Effect of pH on SRF and cake moisture content with modified diatomite (MCPAM: CPAM-modified diatomite; MCTAB: CTAB-modified diatomite. 0.4%wt dosage)

Figure 6

Effect of modified diatomite on concentration of EPS (MCPAM Pr: Protein concentration with CPAM-modified diatomite; MCTAB Pr: Protein concentration with CTAB-modified diatomite; MCPAM Ps: Polysaccharides concentration with CPAM-modified diatomite; MCTAB Ps: Polysaccharides concentration with CTAB-modified diatomite; 0.4%wt dosage; pH3.5)

Figure 7

SEM images of the natural diatomite(a), CPAM-modified diatomite(b) and CTAB-modified diatomite(c) samples

Figure 8

SEM images of dewatering raw sludge sample (a) and dewatering sludge added CPAM modified diatomite conditioning(b)