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 conditioners for sewage sludge dewatering. The effects of these two types of modified diatomite on the dewaterability and settling performance of the activated sludge were studied. The mechanisms of the two modified diatomite types in the activated sludge system were elucidated. The efficiency of the CPAM-modified diatomite was better than that of the CTAB-modified diatomite in improving the settleability and dewaterability of sludge. The results indicated that specific resistance to filtration (SRF) was decreased from $8.52 \times 10^{12}$ to $0.92 \times 10^{12}$ 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 resulted in excellent sludge settling of activated sludge. Further studies showed that the polymer/surfactant adsorbed in diatomite increased sludge dewaterability and improved the sedimentation rate owing to stripping extracellular polymeric substances (EPS) and damaging the internal structure of the sludge, leading to sludge conduce bound water release. According to scanning electron microscope (SEM) images, the two types of modified diatomite powder maintained the porous structure and showed a more complete and uniform structure compared to natural diatomite.

Keywords Polymer · Surfactant · Sludge dewatering · Settling performance · Specific resistance to filtration (SRF) · Extracellular polymeric substances (EPS)

Introduction
With the rapid development of urbanization and industrialization resulting in a surge in 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). Environmental pollution caused by large amounts of sludge cannot be ignored. The treatment and disposal of sludge are key technologies and cost controls of the sewage treatment process (Peng et al. 2021). The difficulty of dewatering due to the high compressibility and low permeability of sludge filter cake is a bottleneck in sludge treatment systems (Huang et al. 2015).

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

Chemical conditioning is another important means applied to benefit the mechanical dewatering method. Traditional chemical conditioners, such as surfactants (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). Extracellular polymeric substances (EPS) are necessary components of activated sludge, having a high affinity for water and thus being highly hydrated. Surfactants can alter the settleability and dewaterability of activated sludge by changing the lease of EPS (Fu et al. 2009; Yuan et al. 2011). However, massive
amounts of surfactants or flocculants have disadvantages in terms of follow-up treatment and disposal, and some polymers increase the cost of sludge treatment. For example, a typical flocculant ferric chloride is highly acidic and toxic and easily corrodes equipment. This increases the concentration of dioxins in incineration flue gas (Yang et al. 2016).

Natural diatomite (SiO₂·nH₂O) is a low-cost, environment-friendly, and natural micro/nanostructured material that has stable acid performance, high porosity, high specific surface area, and low cost, making it suitable as a filter aid, catalytic support, and adsorbent (Liu et al. 2018). Its rich mesoporous/microporous structure accelerates moisture extraction between sludge particles and the loading of modified materials to improve sludge dewatering performance. However, natural diatomite used directly as a conditioner cannot fully meet the demands of sludge dewatering and the environment. Pore size distribution, impurity content, and bulk density are the main factors affecting the quality of the catalyst carrier; therefore, it is necessary to improve further the physical and chemical properties of diatomite (Danilde-namor et al. 2012; Dong and Liu 2020). Surfactants and polymers are effective diatomite modifiers that can improve the pore structure and surface charge, while the subsidence performance of the entire system is enhanced in water samples (Yang et al. 2011). Such as polyvinyl chloride (Wu et al. 2018), polyethylene glycol (Qian et al. 2016), polyethyleneimine (Gao et al. 2005), polyacrylamide (PAM) (Yang et al. 2011), and cetyl trimethyl ammonium bromide (CTAB) (Reham et al. 2017; Sprynskyy et al. 2010) have been widely used in the surface modification of diatomite. For instance, Sprynskyy et al. (2010) prepared CTAB-modified diatomite to remove uranium ions from water. The maximum adsorption capacity of the natural diatomite was 25.63 μmol/g, while the corresponding value of 667.40 μmol/g (158.8 mg/g or 15.9 wt. %) was obtained for the CTAB-diatomite. Yu et al. (2015) prepared phenyltriethoxysilane-modified diatomite (PTES-Dt) to treat the organic matter. The resulting PTES-Dt exhibited quadrupled maximum benzene adsorption compared to isolated diatomite. Previous studies have focused on the adsorption of heavy metals from water using modified diatomite (Liu et al. 2018; Huang et al. 2020).

Yang et al. (2011) modified raw diatomite by PAM and applied it to treat the simulated wastewater with an intensive concentration of lead ions. The results indicated that modification could enhance the diatomite and acidic heavy metal wastewater’s compatibility. Observed by scanning electron microscopy (SEM), the PAM molecule arrayed on the diatomite’s surface smoothly that improved its interior structure and the charge on the surface, even enhanced the sedimentation performance of the diatomite in heavy metal wastewater. Few studies have investigated the role of modified diatomite as a conditioner for sludge dewatering. Considering the application range, cost, and preparation conditions of the modified diatomite, combined with the results of our group’s previous experiments, cationic polyacrylamide (CPAM) and CTAB were selected as representative surfactants and polymers, respectively.

This study aims to investigate the role of modified diatomite particles in the dewatering performance of an activated sludge system. A typical polymer (CPAM) and surfactant (CTAB) were used to modify diatomite, respectively. The sedimentation and dewatering efficiency of the sludge was evaluated by taking the dosage of these two types of modified diatomite and pH as control factors, 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 and the morphology of dewatering sludge were investigated by SEM.

### Materials and methods

#### Test materials

Sewage sludge samples were collected from the Changsha Guozhen Wastewater Treatment Plant in Changsha, China. The physical and chemical characteristics of the sludge are listed in Table 1. All the chemicals used were of analytical grade. A natural diatomite sample was obtained from Chenzhou (Hunan Province, China). Deionized water was used in this study.

#### Preparation of modified diatomite

The preparation process of modified diatomite was according to the following procedure. Natural diatomite was roasted in a muffle furnace at 450–550 °C for 1–2 h and then cooled to room temperature (20–25 °C). The roasted diatomite (40 g) and 5–10% HCl (200 ml) were mixed in a 500 ml beaker and reacted at room temperature (20–25 °C) for 1–2 h. Then, the mixture was filtered, washed with deionized water, and dried in a vacuum oven at 100 ± 2 °C for 24 h.

| Table 1 Characteristics of raw sludge |
|---------------------------------------|
| Mixed liquor suspended solids (MLSS) (g/L) | 4.83 ± 0.026 |
| Volatile solids (MLVSS) (g/L) | 2.52 ± 0.015 |
| Water content (%) | 99.4 ± 0.4 |
| Specific resistance (m/Kg) | 8.5 × 10^{12} |
| Turbidity of supernatant (NTU) | 28.5 ± 0.5 |
| pH | 6.7 |
| Protein in supernatant (mg/L) | 41.4 ± 1.7 |
| Polysaccharide in supernatant (mg/L) | 8.3 ± 0.24 |
then placed in a water bath at 50–65 °C and was stirred for 2 h at a stirring speed of 150–300 r/min. The sample was repeatedly washed and filtered with ethanol and deionized water until the pH was neutral and then dried to a constant weight (Yang et al. 2011). Then, 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 a 100-mesh screen.

**Sludge settling and dewatering**

A specific dosage of the modified diatomite and 150 ml activated sludge mixture was added to a 200 ml beaker and blended violently with a glass rod. After the mixture was stirred, 50 ml of the sludge was centrifuged and the supernatant was prepared for EPS testing. The mixture was then poured into a 100 ml measuring cylinder. The volume of settling sludge was recorded every 5 min for 60 min to obtain the settling volume of sludge (SV60). SRF was measured using a vacuum extraction and filtration device; 100 ml of sludge was poured into a standard 9 cm Buchner funnel with pre-wetting quantitative filter paper at a constant vacuum pressure of 0.03 Mpa for 10–15 min. SRF was determined according to Baskerville and Gale (1968). The water content of the sludge cake trapped by filter paper was measured according to standard methods (APHA, 1998). Finally, dewatered sludge samples and modified diatomite powders were prepared for SEM (JSM-6380LV Japan).

**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). The 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**

SV30 is an important index for evaluating settling sludge performance (Kim et al. 2010). Thirty minutes was extended to 60 min in this study to investigate the sludge sedimentation process more thoroughly. The settling sludge volume after adding CPAM-modified diatomite over time is shown in Fig. 1. Natural diatomite, CPAM, and CPAM-modified diatomite with sludge weight ratios of 0%wt, 0.2%wt, 0.4%wt, and 0.8%wt were added. Serial numbers of samples with different sludge conditioners and dosages are shown in Table 2. For the sludge sample with modified diatomite
added, the height of sludge settling reached stability before 40 min.

In contrast, the settling speed of the sludge sample without conditioning was significantly lower, which required more than 60 min to complete the entire 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 conditioners might increase the volume of sludge. Meanwhile, it could be observed that there were clusters of sludge flocs floating on the surface of the liquid supernatant with 0.8%wt addition.

It was clear that the sludge settling performance by adding CPAM-modified diatomite was better than that of natural diatomite and CPAM. Diatomite addition increased the internal binding strength of the sludge flocs and prevented floc crushing. CPAM was adsorbed on the surface of the negatively charged sludge particles. The thickness of the hydration film on the surface of the particles was reduced by compressing the double electric layer, and the repulsive force between the sludge particles was reduced to form larger flocs. When the flocs grew to a certain extent, the additional external and internal binding forces reached a balance and no longer increased. At this time, the apparent density of the flocs also reached a maximum, and the settlement performance was the best. If there is excessively modified diatomite in the sludge, under the action of strong adsorption and bridge building, the formed flocs will combine the free water around them into larger aggregates. These aggregates contained a large amount of free water, which was loose and had poor performance, resulting in the breaking of the sludge settlement. Adding CPAM alone was slightly better than adding natural diatomite, and adding CPAM-modified diatomite realized the synergistic effect of these two.

The settling sludge volume after adding CTAB-modified diatomite over time is shown in Fig. 2. Natural diatomite, CTAB, and CTAB-modified diatomite with sludge weight ratios of 0%wt, 0.2%wt, 0.4%wt, and 0.8%wt were selected, respectively. The sludge settling curve was similar to that of CPAM. The higher the amount of modified diatomite or natural diatomite, the faster is the sludge settling. Overall, the CPAM-modified diatomite was better than the CTAB-modified diatomite in improving the settling performance of the sludge. Different from adding CPAM-modified diatomite, the sludge settlement rate and efficiency of adding 0.8%wt CTAB-modified diatomite were better than those of 0.4%wt, which was embodied that the final sludge volume was the same, but the sludge settling speed was faster. Surfactant addition increased the internal binding strength of the sludge flocs and prevented floc crushing. Simultaneously, by compressing the double electric layer, the thickness of the hydration film on the surface of the particles decreased, and the repulsive force between the sludge particles was reduced to form larger flocs. It is worth noting that a few clusters of sludge flocs 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. The addition of 0.8%wt was suitable for CTAB-modified diatomite. A dosage of 0.4%wt was determined as the feasible dosage, considering the cost of the modified diatomite.

**Effect of modified diatomite on sludge dewatering performance**

The water present in sewage sludge is classified into four categories: free water (approximately 70%, not directly combined with sludge), interstitial water (approximately 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).

The effects of the dosage of natural diatomite, CPAM, and CPAM-modified diatomite on the SRF and cake moisture content are shown in Fig. 3. It is well known that SRF has been widely used to gauge sludge dewatering. However, for sludges with low SRF and ease of filtration, the cake moisture content does not necessarily decrease because of the presence of chemically bound water. Therefore, the SRF represents the difficulty level of filtration performance, and the moisture content of the sludge represents the efficiency.

| Number | Natural diatomite | CPAM | CPAM-modified diatomite | CTAB | CTAB-modified diatomite |
|--------|------------------|------|-------------------------|------|-------------------------|
| A1     | 0.2%             |      |                         |      |                         |
| B1     | 0.2%             |      |                         |      |                         |
| C1     | 0.2%             |      |                         |      |                         |
| D1     |                  | 0.2% |                         |      |                         |
| E1     |                  |      |                         | 0.2% |                         |
| A2     | 0.4%             |      |                         |      |                         |
| B2     | 0.4%             |      |                         |      |                         |
| C2     | 0.4%             |      |                         |      |                         |
| D2     |                  | 0.4% |                         |      |                         |
| E2     |                  |      |                         | 0.4% |                         |
| A3     | 0.8%             |      |                         |      |                         |
| B3     | 0.8%             |      |                         |      |                         |
| C3     | 0.8%             |      |                         |      |                         |
| D3     |                  | 0.8% |                         |      |                         |
| E3     |                  |      |                         | 0.8% |                         |

Table 2 Serial numbers of samples with different sludge conditioners and dosage
of filtration. To comprehensively evaluate the effect of modified diatomite on sludge dewatering performance, both the SRF and moisture content were considered evaluation parameters in this study. The effects of the two types of modified diatomite on the SRF and water content of the sludge are shown in Fig. 3 and Fig. 4. Raw sludge contains many fine sludge particles that enter the pores of the filter cake, resulting in the reduction of porosity in the filtration process, leading to poor permeability of the filter cake. It was confirmed that CPAM could enhance the flocculation effect of sludge through electrical neutralization and particle adsorption bridging (Ma et al. 2018).

In conclusion, after CPAM pretreatment, the settling sludge performance was significantly and continuously
improved with increasing CPAM dosage. Under the same conditions, CPAM was better than natural diatomite in reducing cake moisture content. Therefore, CPAM and natural diatomite have advantages and disadvantages in reducing the SRF and cake moisture content. The CPAM-modified diatomite can effectively neutralize the advantages of these two, resulting in a synergistic effect. As shown in Fig. 3, when the dosage of CPAM-modified diatomite was 0.6%wt, SRF sharply decreased from $8.52 \times 10^{12}$ (raw sludge) to $0.90 \times 10^{12}$ m/kg, the cake moisture content decreased from 92.2 to 68.5%. The addition of modified diatomite can increase the sludge floc size by flocculating fine sludge particles, thus improving the porosity of the filter cake, which was similar to the addition of polymeric flocculant. However, when the CPAM-modified diatomite dosage increased from 0.6 to 0.8%wt, SRF and the filter cake moisture did not change significantly. The excess long-chain molecules squeezed each other and could not extend freely, weakening the adsorption and bridging effects (Yang et al. 2016). After careful consideration, 0.6%wt was the most suitable dosage for CPAM-modified diatomite.

The effects of the dosage of natural diatomite, CTAB, and CTAB-modified diatomite on the SRF and cake moisture content are shown in Fig. 4. As shown in Fig. 4, CTAB was better than natural diatomite in reducing SRF and cake moisture content when the addition was below 0.6%wt. When the dosage increased from 0.2 to 0.8%wt, the SRF and cake moisture content exhibited a stable decrease with natural diatomite. However, the SRF was no longer reduced, and cake moisture content was rebounded with CTAB or modified diatomite when the dosage was above 0.6%wt. In the suction filtration process, when the amount of CTAB exceeded 0.4%wt, a large amount of foam appeared in the filtrate, which negatively affected sludge dewatering. Owing to the association between the hydrophobic components of the surfactant, it acted as a bridge and net to re-flocculate the suspended sludge particles, forming a larger sludge floc. This re-flocculated sludge floc structure was loose, and free water could pass quickly, greatly improving the filtration speed. However, excess CTAB re-adsorbed part of the free water and wrapped it in the sludge flocs. The suitable dosage of the CTAB-modified diatomite in this study was approximately 0.6%wt, but considering the cost, 0.4% was determined as the recommended dosage.

**Effect of pH on sludge dewatering performance**

Acid addition to sludge has also been beneficial 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 decreased from $1.24 \times 10^{12}$ to $0.92 \times 10^{12}$ m/Kg for CPAM-modified diatomite and from $2.37 \times 10^{12}$ to $1.92 \times 10^{12}$ m/Kg for CTAB-modified diatomite on decreasing the pH in the range of 6.8–3.5. However, the water content and SRF showed a drastic increase when the pH was less than 3.5, demonstrating that a suitable pH value for vacuum extraction and filtration dehydration was 3.5. The dominant reason acidification treatment can significantly reduce the SRF is that it can stimulate EPS to
dissociate from the activated sludge surface, making sludge aggregates easy to accumulate. However, when the pH was less than 3.5, dewaterability did not improve with a further increase in acidity because excessive EPS was released in a short period and blocked the filter paper. The effect of pH on the SRF and cake moisture content with CPAM-modified diatomite was more significant. Overall, a pH value of 3.5 was considered appropriate.

**Effect of modified diatomite on EPS in the supernatant**

Many factors affect sludge dewatering performance, such as EPS concentration, particle size distribution, pH value, and organic concentration. Among them, the EPS concentration has been comprehensively investigated and is considered one of the key factors affecting sludge dewatering performance (Liu and Fang 2003).

EPS is a macromolecular polymer, such as polysaccharides, proteins, and nucleic acids, which are secreted by microorganisms in vitro under certain environmental conditions and widely exist in the interior and surface of activated sludge flocs. These compounds help retain moisture and significantly improve the ability of sludge flocs to bind to water. Therefore, the concentrations of polysaccharides and proteins in EPS were measured to explore the effects of these compounds on dehydration performance (Fig. 5).

The effect of the modified diatomite on the concentration of proteins and polysaccharides is shown in Fig. 6. Proteins and polysaccharides were 41.4 mg/L and 8.3 mg/L, respectively, in the raw sludge sample. Both proteins and polysaccharides increased with increasing amounts of the modified diatomite. At 0.8%wt CPAM-modified diatomite dosage, the increases in the levels of proteins and polysaccharides infiltrate were 169% and 329%, respectively; meanwhile, 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 consistently decrease sludge dewaterability. EPS has a high affinity for water and is thus highly hydrated. Adding the modified diatomite, EPS on the surface of sludge particles can quickly separate from sludge and dissolve in water under a surfactant/coagulant, thus reducing the interstitial water, thereby reducing SRF and moisture content.

Nevertheless, high EPS concentrations increase sludge viscosity (Wang et al. 2010) and a decrease in dewaterability (Chen et al. 2001). In vacuum filtration, the EPS gathers on the surface of the filter paper to form a membrane, which blocks the passage of water. With an increase in modified diatomite, the sludge surface released more EPS into the water, resulting in the obstruction of filtration.

**SEM of modified diatomite and dewatering sludge**

The natural and modified diatomite SEM images are shown in Fig. 7. According to the SEM images shown in Fig. 7, natural diatomite breaks easily, the structure is not intact, and more impurities cover the surface. It is evident that both CPAM-modified and CTAB-diatomite were finer than natural diatomite, especially the impurities were significantly reduced, and the micropores were clearer and more uniform. This shows that the cleaning and roasting of the modified diatomite played a purification role, but the introduction of modified additives did not change the microporous structure.
of natural diatomite. According to the SEM images, there is little structural difference between the CPAM-modified diatomite and CTAB-modified diatomite. It has been guaranteed that CPAM smoothly covers the surface of diatomite, and the weak acid condition is conducive to protonation on the surface of diatomite (Yang et al. 2011). SEM images of the dewatered sludge are shown in Fig. 8. By comparing the apparent morphology of the dewatered sludge with that prepared with CPAM-modified diatomite, it was evident that the latter had better cohesion and formed larger holes, which was more conducive to water release. Acid and CPAM caused the release of sludge EPS, reduced sludge viscosity, and increased sludge settleability. Consequently, the sludge appeared more compact, and more water could be moved during filtration dewatering. Sanin and Vesilind (1994) also observed that EPS removal reduced sludge viscosity. Thus, the sludge appeared more compact, which improved mechanical dewaterability.

**Conclusion**

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 polymer absorption, charge neutralization, particle bridging, decreasing cake blinding, and increasing cake permeability. The experimental data indicated that the efficiency of CPAM-modified diatomite was better than that of CTAB-modified diatomite in improving the settleability.
and dewaterability of sludge. SEM showed that the modified diatomite did not change the microporous structure of the natural diatomite. However, more complete and uniform powders were obtained, which were more conducive to water passage. In addition, the change characteristics of sludge floc structure (microstructure, fractal structure, and floc size) and types of EPS (proteins and polysaccharides) should be investigated in greater depth. The interaction mechanism between EPS and organic pollutants during the treatment process should also be researched more extensively. Overall, this research proves the potential of CPAM/CTAB-modified diatomite for sewage sludge pretreatment in mechanical dewatering. This could provide reference debugging information for the sludge dewatering process and promote the development of efficient and environmentally friendly sludge dewatering technology.

**Fig. 8** SEM images of dewatering raw sludge sample (a) and dewatering sludge-added CPAM-modified diatomite conditioning (b)

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

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**Data availability** All data generated or analyzed during this study are included in this published article.

**Declarations**

**Ethics approval and consent to participate** Not applicable.

**Consent for publication** Not applicable.

**Competing interests** The authors declare no competing interests.

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