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Experimental study on flocculation and dewatering of shield waste slurry from underground construction

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ABSTRACT

With the advancement of the urbanization process, numerous urban construction projects have led to a surge in the discharge of construction waste in China. The slurry directly produced in underground engineering and the wastewater indirectly produced in the sand washing procedure account for the majority of construction waste, but their utilization rate is less than 5%, and they have poor sustainability. Therefore, developing technology for dewatering and volume reduction of waste slurry and wastewater with high water content is of great practical significance for promoting green construction and environmental protection. In this paper, organic and inorganic flocculants were designed to identify the characteristics and efficiency of slurry-water separation. A set of tests, including sedimentation, particle size distribution, turbidity, zeta potential, and scanning electron microscopy (SEM) tests were conducted to study the effect and mechanisms of flocculants on slurry settlement. The results show that the use of flocculants can quickly agglomerate small particles into larger ones, and there is an optimal dosage for each type of flocculant. The order of the effect of flocculants from strongest to weakest is APAM > PAM > CPAM > PAC > $AlCl_3$. Compared to inorganic flocculants, the conditioning effect of organic flocculants is better. The flocculation process is generally the result of the combined action of four types of agglomeration mechanisms. The results provide theoretical guidance and a reference for formulating the optimal design for slurry treatment.

Keywords: Shield slurry; Flocculants; Slurry-water separation; Micro-mechanism

1 INTRODUCTION

With the rapid development of urbanization, a significant number of construction projects in cities have resulted in an increase in the discharge of construction waste. In China, slurry and wastewater produced by underground projects, such as shield tunnelling, pile installation, and cut-off walls, account for the majority of construction waste, but their utilization rate is less than 5%, which is not sustainable.

The slurry produced by underground projects is a suspension system comprising water, bentonite, cohesive soil, and foaming agents. Currently, large particles in the slurry are generally separated by vibrating screening, sand washing, and cyclones. The remaining slurry and wastewater with high water content are then dewatered for further utilization, which is a challenging and time-consuming process due to the high water content and fine particle content. Therefore, it is of great practical significance to develop an eco-friendly and suitable technology for dewatering and volume reduction of waste slurry.

Currently, slurry dewatering treatment mainly includes flocculation dewatering, mechanical dewatering, seepage dewatering, evaporation dewatering, natural drying/heat drying, and combined dewatering. Among them, flocculation dewatering is widely used due to its advantages of high efficiency, low cost, and small footprint. Furthermore, mechanical and seepage dewatering are often combined with flocculation dewatering in engineering to improve efficiency and reduce costs.

Existing studies have focused on the development of flocculant experiments and the benefits of adding flocculants. However, a systematic analysis and comparison of various organic and inorganic flocculants on the dewatering of waste slurry still needs further investigation. Moreover, the slurry-water separation process and mechanism need to be adequately explored from both macroscopic and microscopic perspectives. Therefore, this study aims to identify the effects and mechanisms of flocculants on slurry settlement through a set of tests, including sedimentation, particle size distribution, turbidity, zeta potential, and scanning electron microscopy (SEM) tests.

2 MATERIALS

2.1 Shield waste slurry

The slurry tested in the experiment was taken from a settling pond of shield waste slurry after sand washing, which originated from an underground construction project site in Xian city, China. This project includes an underground shield section that passes through a water-rich sand layer. During shield excavation, modifiers are commonly used to enhance the shield muck's flow plasticity and prevent consolidation while reducing the likelihood of segregation. As a result, the shield slurry produced by washing the shield muck via the sand washing process is a combination of soil particles, water, and modifiers. Due to the high water content of the shield slurry, there is a significant risk of direct landfilling, which can lead to poor anti-slide ability of the piled slope and landslide accidents. Furthermore, there are no adequate pollution prevention and treatment measures in place for ordinary landfill sites, resulting in the contamination of groundwater and rock and soil by harmful substances like amendments. However, the solid particles in the shield slurry are primarily composed of silt and clay, which have a certain recycling value. For example, they can be used as building materials to replace natural resources and promote resource utilization.

The basic characteristics of the waste slurry were determined in accordance with the Chinese standard Test Methods of Soils for Highway Engineering [JTG E40-2007 (Chinese Standard 2007)], and are summarized in Table 1.

The original slurry was tested for some basic physical properties, as follows: the density of the test slurry was found to be 1.04g/cm^3, and its pH value was 9.75, indicating it was weakly alkaline. The slurry exhibited a high initial water content of 93.9% by weight, denoting the proportion of water present in the semi-liquid mixture comprised of solid material. The initial Zeta potential was measured at -22.42mV. The particle size distribution analysis indicated that the sand, silt, and clay-sized fractions comprised 5.79%, 89.5%, and 4.70% of the slurry, respectively. The high content of fine particles made it difficult for the slurry to settle and separate naturally.

Table 1. *Basic physical characteristics of shield waste slurry*

Density (g/cm^3)	Initial water content (%)	Initial Zeta potential (mV)	pH	Sand (%)	Silt (%)	Clay (%)
1.04	93.9	-22.42	9.75	5.79	89.5	4.70

2.2 Flocculants

During the flocculation process, stable colloidal particles or fine particles in slurry collide, aggregate, bond with each other and precipitate large-size flocs.

Flocculants generally refer to agents that can destabilize solutes, colloids, or suspended particles in aqueous solutions to produce flocs or flocculent precipitates. According to the types of their compounds, flocculants can be divided into four categories: inorganic flocculants, organic polymer flocculants, composite flocculants and microbial flocculants. The typical representatives of various flocculants, their advantages and disadvantages are shown in Table 2.

Colloids and fine particles are dispersed in the suspension system. After adding flocculants, the particles will destabilize and collide with each other, destroy the stability of the colloidal structure and the system. They gradually adhere to each other, agglomerate and form larger flocculated sediment. The flocculation process can in fact be divided into two parts. First, unstable particles in water collide with each other and are brought together by Van der Waals forces. These particles are usually small in size and form "micro flocs". After that, the micro flocs collide and further aggregate to form "big flocs" due to interactions such as chemical bonds and adsorption bridging.

In this study, a range of flocculants, including APAM, CPAM, PAM, $AlCl_3$, and PAC, were utilized in the experiments.

Table 2. *The category, advantages and disadvantages of flocculants*

Category	Typical flocculants	Advantages and disadvantages
Inorganic flocculant	Aluminum chloride ($AlCl_3$), aluminum sulfate ($Al_2(SO_4)_3$),ferric sulfate ($Fe_2(SO_4)_3$), ferric chloride ($FeCl_3$), polyaluminum chloride (PAC), polyaluminum sulfate (PAS), polymer Silicic acid (PS), etc.	1. Cheap and easy to use 2. Mainly used for water purification, removal of heavy metals, sulfides, etc. 3. High dosage but normal effect 4. A lot of corrosive sludge produced by flocculation
Organic polymer flocculant	Natural material: chitosan, chitin flocs, etc. Synthetics: polyacrylamide (anionic APAM, cationic CPAM, nonionic PAM), sodium polyacrylate, polyethylene,etc.	1. Excellent flocculation effect, less dosage and wide application range 2. Good stability; almost not affected by temperature and pH; less sludge is produced; flocs are easy to separate 3. The flocculant itself or the product of hydrolysis has certain toxicity
Composite flocculant	Polyaluminum iron nitride (PAFC), polyphosphorus ferric chloride (PPFC), starch-polyacrylamide graft copolymer, etc.	1. Wide range of application 2. The synthesis process is complex and requires high cost
Microbial flocculant	AHU 7165 Aspergillus parasitica, KJ201 Alcaligenes symphysis, etc.	1. Self-decomposition; low pollution; obvious flocculation effect 2. Few related studies, and the conditions for culturing are strict

3 EXPERIMENTAL PROCEDURE

3.1 Testing plan

To improve the efficiency of the environmental protection treatment process for shield waste slurry, we conducted flocculation and dewatering experiments on slurry with high water content using a variety of commonly used engineering flocculants such as APAM, CPAM, PAM, $AlCl_3$, and PAC. Based on the experimental results, we identified the most suitable flocculant type and dosage, and analyzed the micro-mechanisms of flocculation. The testing plan includes the following:

Conduct sedimentation tests to compare and evaluate the settling effect of various flocculants. We will identify the optimal dosage by comparing particle size distribution (measured using a Laser Particle Size Analyzer, Eye Tech 17353004) and the turbidity change of the slurry supernatant (measured using a Turbidimeter, ZD-10A).

Observe the microscopic changes of particles and analyze the microscopic changes in particle structure after flocculation and sedimentation using analysis of Zeta potential (measured using a Nanoparticle Size Analyzer, NanoBrook 90Plus Zeta 240065), microscope scanning (using a Zeiss Field Emission Scanning Electron Microscope, SEM Merlin 14013319), and other methods.

Analyze the micro-mechanisms of different types of flocculants in reducing suspension stability and exerting a flocculation effect, based on the results of the sedimentation tests and microscopic experiments.

3.2 Testing methods

As slurry is not convenient for long-distance transportation, the shield waste slurry used in the experiment was prepared by mixing original dry sand with deionized water to match the moisture content of the original slurry. The mixture was then stirred for over 24 hours to ensure complete hydration and homogeneity of the samples. Preliminary experiments were conducted to determine the effective concentration ranges of several flocculants for further experiments. The PAM-class flocculants were first dissolved in deionized water to achieve concentrations of 1g/L and then diluted to 100, 300, 500, 700, and 900 mg/L. On the other hand, $AlCl_3$ and PAC were easily dissolved in water and were directly dissolved in deionized water to achieve concentrations of 400, 800, 1200, 1600, and 2000 mg/L. The flocculant solution was then mixed with the slurry and evenly distributed by stirring with a stirrer at 100 r/min for 1 min. To analyze and compare the flocculation effect of different flocculants, a control sedimentation test was conducted on the original slurry without any flocculant. Static sedimentation tests were carried out by filling 1L cylinders with 1L mixed slurry one by one, settling naturally for 24 h, and observing and recording the final height of the slurry-water separation interface.

To measure the effect of flocculation and sedimentation, changes in slurry particle gradation and Zeta potential before and after adding flocculants were measured. Additionally, the turbidity changes of the supernatant after the static sedimentation test were analyzed. Based on the results, the optimal type and dosage of flocculant were selected.

After determining the optimal amount of flocculant, sediment samples of the original slurry and those treated with flocculants were taken out for low-temperature drying for 24h. The samples were sputter-coated with a thin layer of gold on the fresh surface to enhance the conductivity and were tested for microscopic morphology changes of the sediment by scanning electron microscopy (SEM).

4 RESULTS

4.1 Flocculation-Induced change in slurry particle size

Figures 1 illustrates the particle size distribution of sediments in slurry after treatment with different concentrations of flocculants. The results reveal that the flocculation effect of $AlCl_3$-treated shield waste slurry is poor, with almost no significant effect on the particle size. Determining the optimal dosage of $AlCl_3$ requires additional tests such as turbidity experiments.

In comparison, the flocculation effect of PAC is more apparent, and the aggregation effect of flocculants on slurry particles initially increases and then decreases with the rise in flocculant concentration.

The flocculation effects of organic flocculants APAM, CPAM, and PAM on treating shield waste slurry are significantly better than those of inorganic flocculants.

During the stirring process, the slurry rapidly precipitates large particles, resulting in very high slurry-water separation efficiency. As the concentration of organic flocculants increases, the flocculation effect appears to increase initially and then weaken, indicating an optimal dosage. The long-chain bridging is the primary method of flocculation in organic polymer flocculants. The appropriate dosage density promotes the formation of a flocculation molecular network between the molecular chains of the flocculant, which can effectively trap fine particles and colloids in the slurry.

This results in a continuous increase in the gravitational volume of the flocculation, leading to the significant acceleration of flocs settling. If the density of the flocculant is too high, its long molecular chain structure will be seriously curled, forming a coil wrapped by flocs, which results in a decrease in the bridging effect. The results in Figure 1(c) show that the treatment of APAM has the most significant deviation to the left in the particle size curve of the slurry when added at a concentration of 500mg/L. Additionally, the particle size distribution changes significantly.

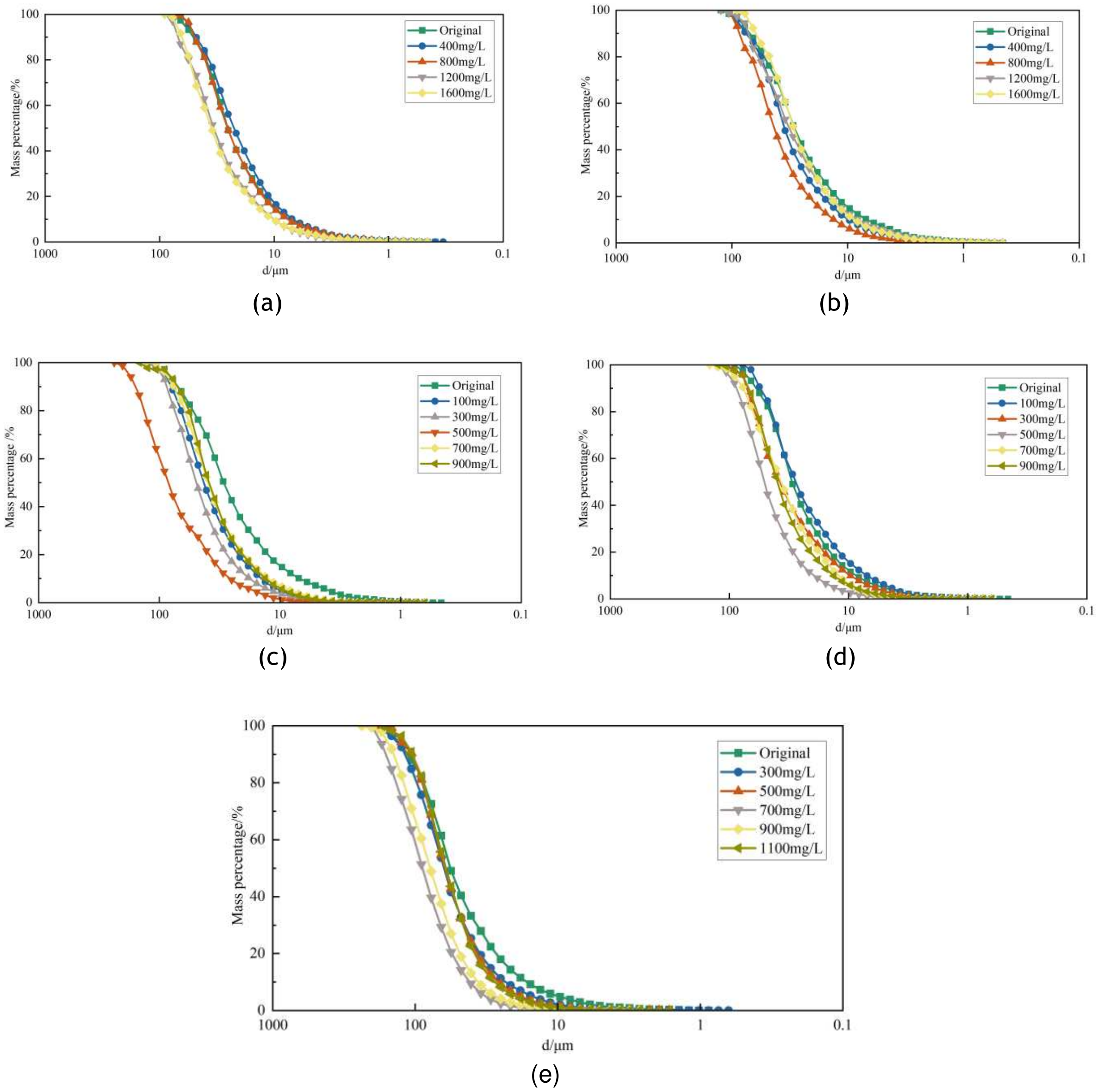


Figure 1. *Change in particle size distribution curves of sediments in slurry caused by flocculation: (a) The original slurry (b) $AlCl_3$-treated slurry (c) PAC-treated slurry (d) APAM-treated slurry (e) CPAM-treated slurry (f) PAM-treated slurry*

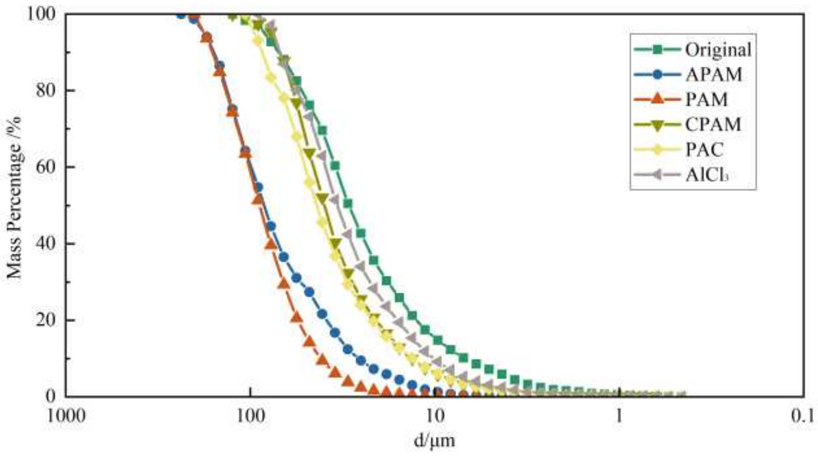
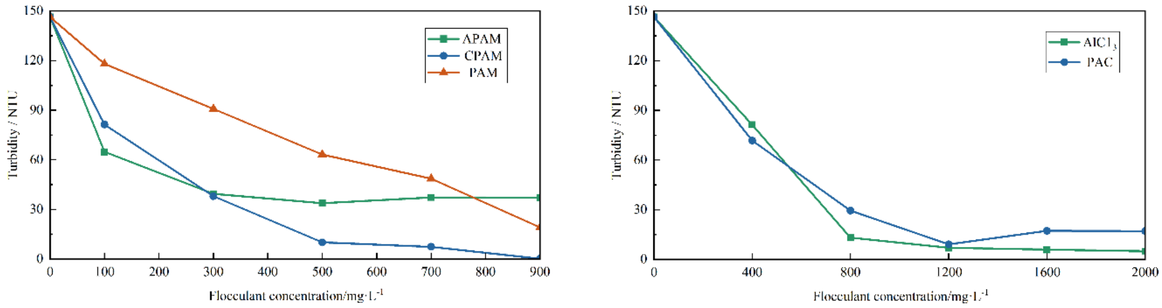


Figure 2. *Comparison of particle size distribution curves between flocculants in optimal dosage*

4.2 Flocculation-Induced change in turbidity of slurry supernatant after settlement

After settling for 24 hours, the supernatant turbidity of the original slurry was measured at 146.4 NTU. Figure 3 shows the change in turbidity of the supernatant of the flocculant-treated slurry with the amount of flocculant added. The turbidity of the various slurry supernatants decreased rapidly at first with the increase of flocculant, and then stabilized, indicating that there was an optimal dosage of flocculant. When an excess amount of flocculant was added, its molecular chain could not be fully opened to absorb more fine particles and act as a bridging agent in flocculation and sedimentation, resulting in no significant improvement in slurry flocculation and sedimentation.

Among the organic flocculants, the supernatant turbidity of the slurry treated with APAM, CPAM, and PAM did not change significantly at dosages above 500, 500, and 700 mg/L, respectively. The turbidity of the slurry treated by $AlCl_3$ and PAC decreased slowly between 800 and 1200 mg/L, indicating that the optimal dosage was between 800 and 1200 mg/L. This finding is consistent with the conclusion based on the change in the particle size distribution curve mentioned earlier.



(a) (b)

Figure 3. *Change in the supernatant turbidity of slurry caused by various flocculants: (a)slurry treated by organic flocculants (b)slurry treated by inorganic flocculants*

4.3 Selection and analysis of flocculants based on static sedimentation tests

To quantitatively analyze the influence of flocculants on the particle size distribution of shield waste slurry, the slurry particle size is divided into 4 intervals with nodes at 8.5 μm, 50 μm, 75 μm, and 250 μm in Figure 4.

For the original slurry, the proportions of particles in the 0-8.5 μm and 8.5-50 μm ranges were 12.3% and 63.9%, respectively, accounting for 76.2% of the total mass of all particles. This indicates that the original slurry contains a high content of fine particles, making it difficult to settle under natural conditions.

After adding flocculants, the proportion of particles in the 0-50 μm range in the treated slurries was smaller than that of the original slurry, indicating that the rapid aggregation of some colloidal and fine particles reduced the mass ratio of small particles and effectively increased the ratio of large particles.

By comparing the adjustment effects of different types of flocculants, the proportion of large particles with a size of 50-250 μm increased by 3.0% and 12.4% in $AlCl_3$ and PAC-treated slurries, respectively. In contrast, the proportion of large particles with a size of 50-250 μm in the CPAM, APAM, and PAM-treated slurries increased by 20.1%, 48.8%, and 68.0%, respectively. Additionally, particles with a size of 75-250 μm in the APAM and PAM-treated slurries respectively accounted for 55.5% and 60.4% of the total mass, which is much higher than the slurry treated with inorganic flocculants.

These results indicate that the effect of polyacrylamide organic flocculants on the aggregation of fine particles is significantly stronger than that of traditional inorganic flocculants.

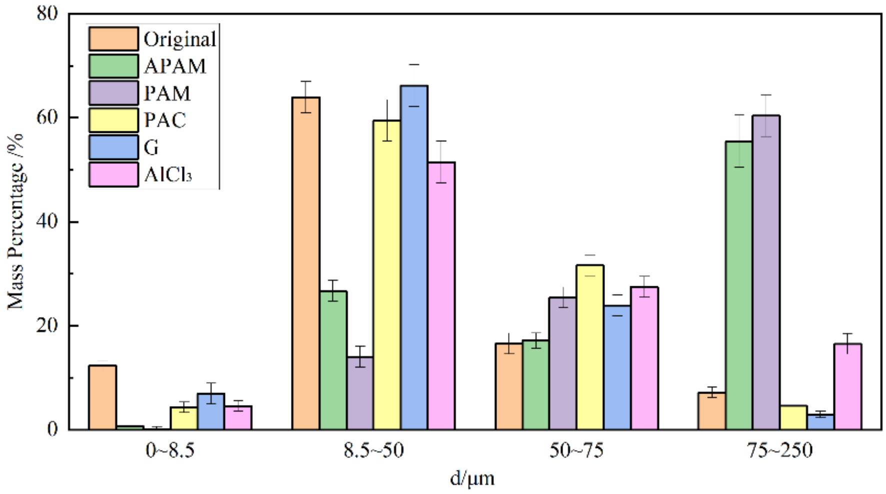


Figure 4. *Comparison of particle size distribution of sediments in slurry treated with optimal amount of flocculants*

Table 3. *Average particle size of slurry sediments treated by various test flocculants in optimal dosage*

Category	Average particle size of slurry sediments in optimal dosage (μm)
PAM-treated slurry	88.3
APAM-treated slurry	83.1
PAC-treated slurry	43.4
CPAM-treated slurry	39.4
$AlCl_3$-treated slurry	33.7
Original slurry	29.0

5 MICRO-MECHANISMS OF FLOCCULATION

5.1 Flocculation-Induced microscopic morphology changes of slurry by SEM

Figure 5 shows the SEM images of the original slurry sample and the slurry treated with $AlCl_3$, PAC, APAM, CPAM, and PAM.

The original slurry contained a high proportion of fine particles, which had a flake-like morphology and tended to accumulate parallel to each other. After treatment with flocculants, the content of fine particles decreased, and they agglomerated to form larger flocs with a disordered arrangement and random contact mode, leaving gaps between the flocs.

The flocs had a multi-level structure and were composed of many large flocs, which contained many small flocs or single sediment particles formed by active groups such as hydrogen bonds. The small flocs or particles were connected to form a large floc network. The complex structure of the floc network helped capture and sweep more flocs or colloidal particles, resulting in a larger floc structure with a complex arrangement. The flocs formed by inorganic flocculants were generally smaller in volume and simpler in hierarchical structure than those formed by PAM polymer flocculants.

The flocs had three contact modes, namely surface-surface, edge-surface, and edge-edge. Surface-surface contact was dominant, and the contact and connection between flocs were relatively loose and easy to compress.

Compared with the shield slurry treated with inorganic flocculants, the flocs formed by the flocculation of polyacrylamide polymer organic flocculants had smaller gaps between them, denser connections between particles, and were more likely to form large-sized floc networks. Moreover, fewer small particles were exposed on the floc surface.

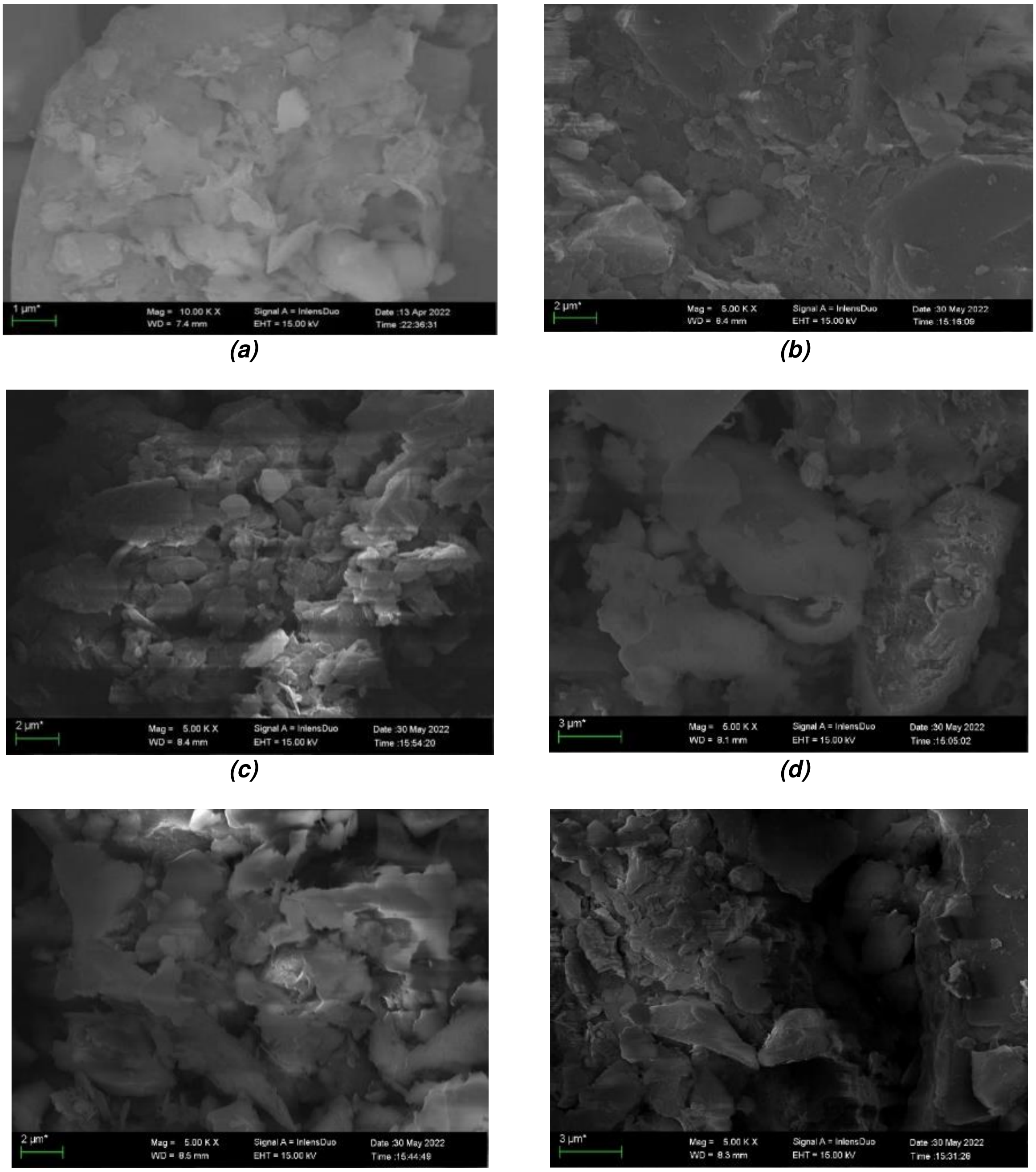
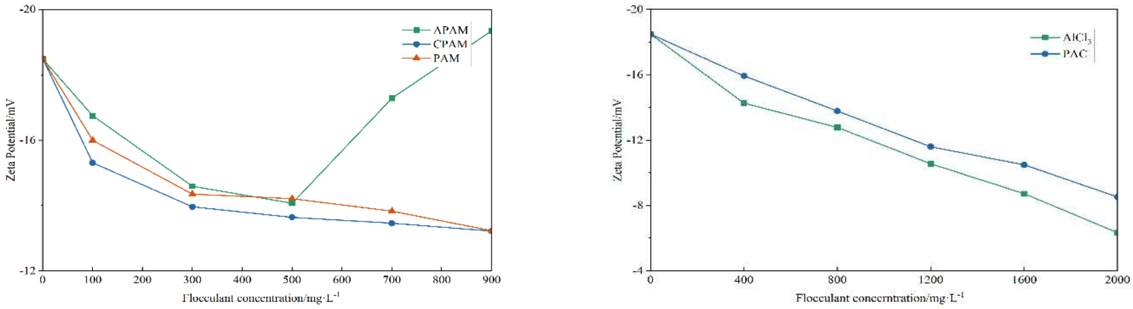


Figure 5. *Comparison of particle size distribution between various test flocculants in optimal dosage* ***(a)*** *The original slurry (b) $AlCl_3$-treated slurry (c) PAC-treated slurry (d) APAM-treated slurry (e) CPAM-treated slurry (f) PAM-treated slurry*

5.2 Flocculation-Induced Zeta potential changes of slurry

Figure 6 shows the changes in Zeta potential of the slurry treated with different flocculants. The Zeta potential of the APAM-treated slurry initially decreases and then increases with the increase of flocculant content. On the other hand, the Zeta potential of the CPAM and PAM-treated slurries decreases with the increase of flocculant content and then remains stable. However, the change range is small, indicating a minimal impact of the compressed double layer in the flocculation mechanism.

In comparison, the effect of inorganic flocculants, such as $AlCl_3$ and PAC, on the Zeta potential of the slurry is much more pronounced. As their content increases, the Zeta potential of the slurry continuously decreases, indicating that the compression electric double layer effect plays a dominant role in their mechanism. Overall, the results suggest that the organic flocculants have a weaker effect on the Zeta potential of the slurry than the inorganic flocculants.



(a) (b)

Figure 6. *Zeta potential changes of slurry treated by various flocculants: (a)slurry treated by organic flocculants (b)slurry treated by inorganic flocculants*

6 CONCLUSIONS

In this study, we conducted a series of tests to determine the appropriate type and optimal dosage of flocculants for the flocculation treatment module in the environmental protection treatment process of shield waste slurry and wastewater indirectly produced in the sand washing procedure. The tests included sedimentation, particle size distribution, turbidity, Zeta potential, and scanning electron microscopy (SEM) tests. The results allowed us to analyze the microcosmic mechanism of the flocculants destabilizing the suspension system and exerting the flocculation effect to provide a scientific basis for the flocculation treatment of shield waste slurry in engineering practice.

The main conclusions of this study are as follows:

Firstly, the addition of flocculants can accelerate the settling and aggregation of particles, reduce particle dispersion, and promote the aggregation of fine particles to form large floc structures.

Secondly, compared to inorganic flocculants, the conditioning effect of organic flocculants is better. The optimal concentrations of various flocculants for slurry-water separation are 500mg/L (APAM), 500mg/L(CPAM), 700mg/L(PAM), 1200mg/L($AlCl_3$), and 800mg/L(PAC), respectively. Further addition of flocculant would prevent the separation of water from slurry. The order of the effect of flocculants from strong to weak is: APAM > PAM > CPAM > PAC > $AlCl_3$.

Finally, the flocculation process is generally the result of the combined action of four flocculation mechanisms, including compressing the thickness of the electric double layer, characteristic adsorption neutralization, adsorption bridging, and net trapping and sweeping. Depending on the different flocculants and conditions of use, a certain flocculation mechanism dominates. Among the tested flocculants, the micro-mechanisms of $AlCl_3$ and PAC are dominated by compressed electric double layer, the micro-mechanism of APAM is dominated by adsorption bridging and net trapping and sweeping, and the micro-mechanisms of CPAM and PAM are dominated by characteristic adsorption neutralization and net trapping and sweeping.

7 ACKNOWLEDGEMENTS

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