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Articles

Evaluation conditions to treat slime water with coagulant-bioflocculant by orthogonal design

Condiciones de evaluación para el tratamiento del agua de fango de carbón con coagulante-biofloculante por prueba ortogonal

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Abstract

In this study, a new type of microbial flocculant combined with Poly-aluminum ferric chloride (PAFC) coagulant was utilized in the coagulation-flocculation experiment of slime water. The removal effects of PAFC dosage, the bioflocculant dosage, and pH on the turbidity and Chemical Oxygen Demand (COD) of the coal slime water were investigated. The results illustrated that the best conditions for removing slime water turbidity and COD by single factor test were as follows: the dosage of PAFC was 37.5 mg/l, the dosage of bioflocculant was 0.75 mg/l, and the pH was 7.0. The most influential factors for turbidity and COD removal were identified through orthogonal test methodology as pH and PAFC dosage, respectively. The coagulation-flocculation of the best condition was a dosage of PAFC of 50.0 mg/l, the dosage of bioflocculant was 0.5 mg/l, and the pH was 7.0. Under this condition, the turbidity removal efficiency of 93.2 % and the COD removal efficiency of 47.2 % were obtained.

Keywords: Coal slime water, bioflocculant, single factor, orthogonal design.

Resumen

En este estudio se utilizó un nuevo tipo de floculante microbiano combinado con coagulante de cloruro férrico de polialuminio (PAFC) en el experimento de coagulación-floculación de agua de limo de carbón. Se investigaron los efectos de la dosis de PAFC, la dosis de biofloculante y el pH sobre la turbidez y la demanda química de oxígeno (COD) del agua de limo de carbón. Los resultados mostraron que las mejores condiciones para eliminar la turbidez del agua de limo y la COD mediante la prueba de factor único fueron las siguientes: la dosis de PAFC fue de 37.5 mg/l, la dosis de biofloculante fue de 0.75 mg/l y el pH fue de 7.0. A través de la prueba ortogonal se identificó que los factores más influyentes para la turbidez y eliminación de COD son el pH y la dosis de PAFC, respectivamente. La mejor condición de la coagulación-floculación fue con una dosis de PAFC de 50.0 mg/l, una dosis de biofloculante de 0.5 mg/l y el pH de 7.0. Bajo esta condición, se obtuvo una eficiencia de remoción de turbidez del 93.2 % y una eficiencia de remoción de COD del 47.2 %.

Palabras clave: agua de limo de carbón, biofloculante, factor único, diseño ortogonal.

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Introduction

Water resources are one of the most indispensable and vital energy sources on the earth. However, with rapid economic development, various industrial wastewater discharges increase, and water pollution is becoming increasingly severe (Chiban, 2011; Chiban, Soudani, Sinan, Tahrouch, & Persin, 2011). Among these, coal mine wastewater is one (Faustino *et al*., 2019).

Coal slime water from coal washing consists of water and coal powder. Its main characteristics are high turbidity, fine powder, and its strong negative charge on the surface of solid particles. These particles are dispersed in water because the exact charges repel each other (Lin, Li, Hou, Kuang, & Wang, 2017). Furthermore, due to the surface interaction (adsorption, dissolution, combination, etc.) of solid particles in coal slime water (Chiban, Soudani, Sinan, & Persin, 2012; Chiban, Soudani, Zerbet, & Sinan, 2013), its removal becomes more complex, not only because of its suspension properties but also because of its colloidal characteristics (Sun, Xu, Nie, & Zheng, 2013; Lin *et al*., 2020; Liang, Baoping, & Hong, 2010). Consequently, it is very difficult to control these conditions, which have become the main conditions in restricting the removal of pollution sources in the coal industry (Guo, Zhang, & Gao, 2013). However, coal slime water must be clarified and recycled to save water resources and reduce environmental pollution (Barraza, Guerrero, & Pineres, 2013;

Zhang, Chen, Fu, Yan, & Kim, 2016). Therefore, it is crucial to purify the slime water and meet the discharge standard.

In coal preparation plants, methods of coagulation-flocculation and aggregation usually strengthen the process of coal slime water to achieve wash water closed-circuit circulation. As a result, we must focus on this problem in applying coagulant and flocculant. Flocculants are increasingly widely used in coal preparation plants because they are high efficiency and low cost (Wang, Gan, & Zhang, 2019; Xia *et al*., 2018; Gumfekar & Soares, 2018). Among them, the inorganic polymer coagulant PAFC has the advantages of various applications. For example, it can condense high turbidity water, coal mine water, and lake water as drinking water sources. Notably, the effect is better than Polyaluminum chloride and ferric chloride (Faraji *et al*., 2019). The reagent has higher stability, shorter solidification time, and faster-settling speed, so it is often used to treat wastewater from the coal mine. However, it faces large dosage and secondary pollution problems, so the research and development dosage is small, and the flocculant without secondary pollution is incredibly concerning. Among them, microbial flocculants are interesting because of their excellent flocculation effect, and they are biodegradable and non-toxic, and harmless to the human body and environment (Yang, Wang, & Liu, 2017; Nguyen, Klai, Nguyen, & Tyagi, 2016; Singh *et al*., 2017). Microbial flocculants are natural organic macromolecules produced and secreted by

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microorganisms during active metabolism and cell lysis. These molecules can flocculate suspended solids (SS), cells, and colloid materials (Lian *et al*., 2008). Previous studies have primarily focused on screening new isolates and optimizing culture conditions for high yield production (Xia *et al*., 2008; Aljuboori, Idris, Abdullah, & Mohamad, 2013; Liu *et al*., 2013). Consequently, it is essential to use the separated microbial flocculants in the actual industrial wastewater treatment.

In this study, the bioflocculant developed in the laboratory combined with PAFC coagulant was used for the coagulation-flocculation purification process of coal slime water. The removal effects of bioflocculant dosage, PAFC dosage, and pH on turbidity and COD of coal slime were studied. The degree of the influencing coagulation-flocculation effect was determined by conducting an orthogonal experiment, and a good result was achieved. This may help develop new flocculation agents and improve the clarification process of coal.

Materials and methods

Materials

Poly-aluminum Ferric Chloride (PAFC), which contains 30.4 % of alumina (Al_2O_3) and 1.5 % of iron oxide (Fe_2O_3); potassium dichromate ($K_2Cr_2O_7$); Silver sulfate (Ag_2SO_4); Mercuric sulfate ($HgSO_4$); 1,10-Phenanthroline ($C_{12}H_8N_2 \cdot H_2O$).

Raw water in this study was collected from the coal slime water of the coal mine water purification plant in Heilongjiang province, China. After sampling the water from the coal mine, the samples were quickly transported to the laboratory. The turbidity and COD of the water samples were determined within 12 hours. The raw water characteristics were as follows: the temperature was 20.2 °C, pH was 7.6, turbidity was 2440.0 NTU, and COD was 2037.1 mg/l.

The bioflocculant is primarily composed of polysaccharides (90.6 %) and protein (9.3 %) with a molecular weight of 10^5-10^6 Da (Li, Ma, & Zuo, 2016). The fermentation conditions were shaking at 140 rpm/min, 30 °C, and fermentation for 24 hours. Fermentation medium: glucose 10 g/l, K_2HPO_4 5 g/l, $MgSO_4 \cdot 7H_2O$ 0.2 g/l, urea 0.5 g/L, KH_2PO_4 2 g/l, NaCl 0.1 g/l, yeast extract 0.5 g/l, pH 7.5. The medium applied in the expansion culture was the seed culture medium prepared according to 10 % of the required fermentation liquid. The finished product was the liquid bacterial solution.

Jar-test

Coagulation experiments were performed in 1.0 L beakers using conventional Jar-test apparatus at room temperature. Each experiment was repeated three times to ensure experimental accuracy. The experiments of a single factor of PAFC dosage (12.5-62.5 mg/l), bioflocculant dosage (0.25-1.25 mg/l), and pH (5.0-9.0) were implemented (Li *et al.*, 2020). After adding PAFC, the solution was subjected to rapid mixing at 300 rpm (an equivalent of the average mixing gradient of 241.7 s^{-1}) for 1 minute; bioflocculant was added again, followed by slow mixing at 50 rpm (an equivalent of the average mixing gradient of 23.8 s^{-1}) for 5 minutes. The mixing was then stopped to allow the aggregated flocs to settle for 20 minutes. When each jar test was over, the supernatant water samples from approximately 3 cm below the water surface were taken for measurement.

Turbidity was measured by an SGZ-2P turbidimeter (Tomperi, Isokangas, Tuuttila, & Paavola, 2022); COD was determined using the potassium dichromate method (Zhang, Chen, Liu, Luo, & Gu, 2018); pH was measured by Sartorius PB-10 acidity meter.

Results and discussion

Effect of PAFC dosage on turbidity and COD removal

As depicted in Figure 1, the turbidity of coal slime water was significantly reduced by increasing the dosage of PAFC from 12.5 mg/l to 37.5 mg/l. When the dosage was 37.5-62.5 mg/l, the difference in turbidity removal efficiency was small, and the highest removal efficiency was able to reach 88.2 % at 62.5 mg/l. The reason for this is attributed to the fact that the cations provided by PAFC in the water reduced the negative charges on the surface of microbial flocculants and colloidal particles and promoted

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the adsorption capacity of flocculants to the suspended particles in water (Zhang *et al*., 2019). A close and stable large floc was formed under the combined action of electric neutralization and adsorption bridging, which enhanced the flocculation effect (Wang, Jiang, Tan, Wang, & Wang, 2018). The results demonstrated that charge neutralization was one of the most crucial coagulation mechanisms.

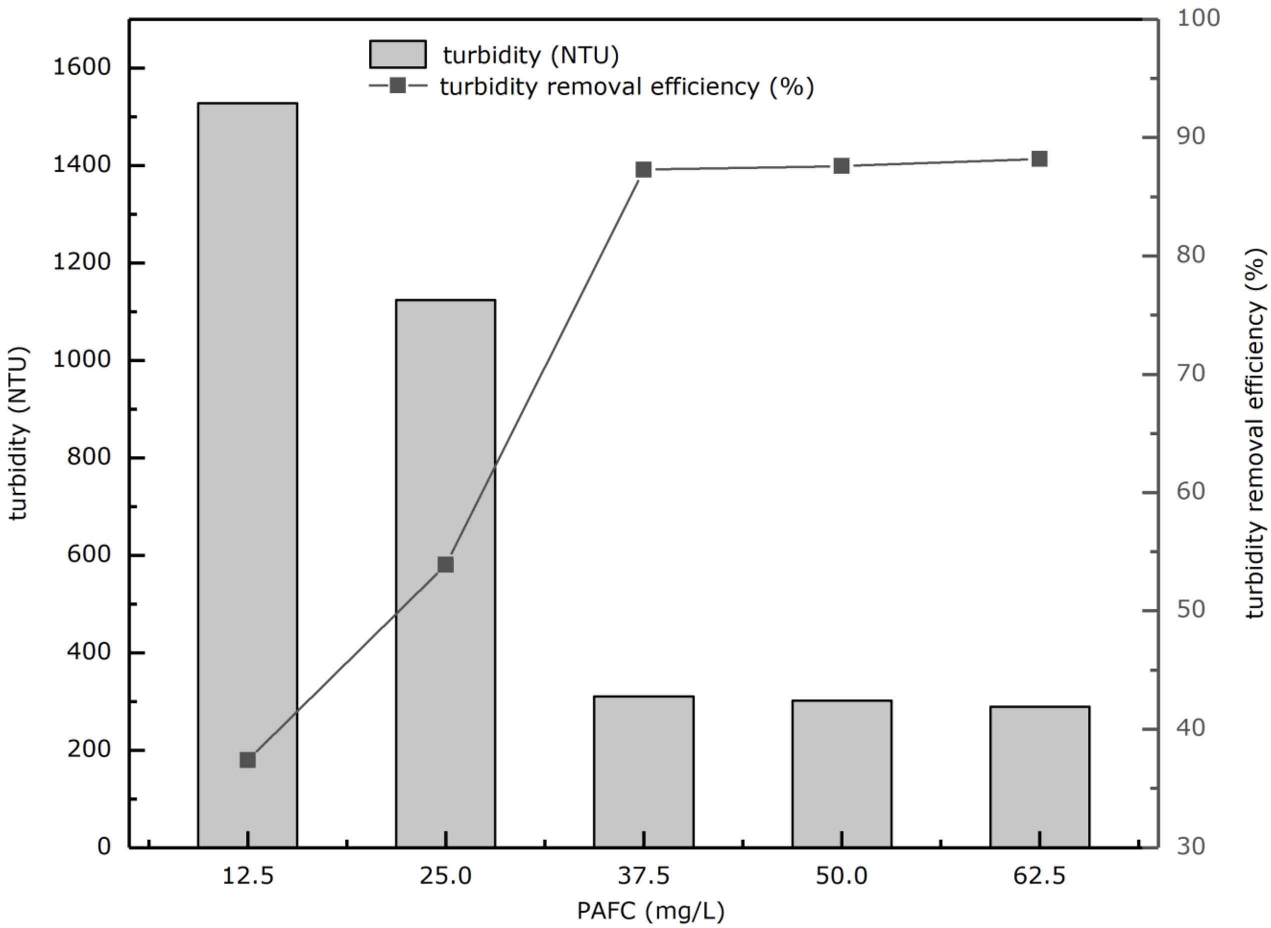

Figure 1. Effects of PAFC on removal efficiency of turbidity.

As illustrated in Figure 2, the COD of coal slime water firstly decreased and then increased with the increase of PAFC dosage from 12.5 mg/l to 62.5 mg/l. It was noted that the highest COD removal efficiency reached 46.2 % at the dosage of 50 mg/l, and then the COD removal efficiency began to decline with the increase of the dosage. The COD removal efficiency was slightly different when the dosage was 37.5-62.5 mg/l. This is because PAFC can bridge with particles in coal slime water and neutralize the electric charge, which is beneficial to adsorption and flocculation (Gao, Liu, Wang, & Huang, 2013). Specifically, COD removal efficiency was similar when the PAFC dosage was 37.5 mg/l and 50 mg/l, respectively. Therefore, under the comprehensive consideration of flocculation efficiency and economic cost, the optimum dosage of PAFC was 37.5 mg/l.

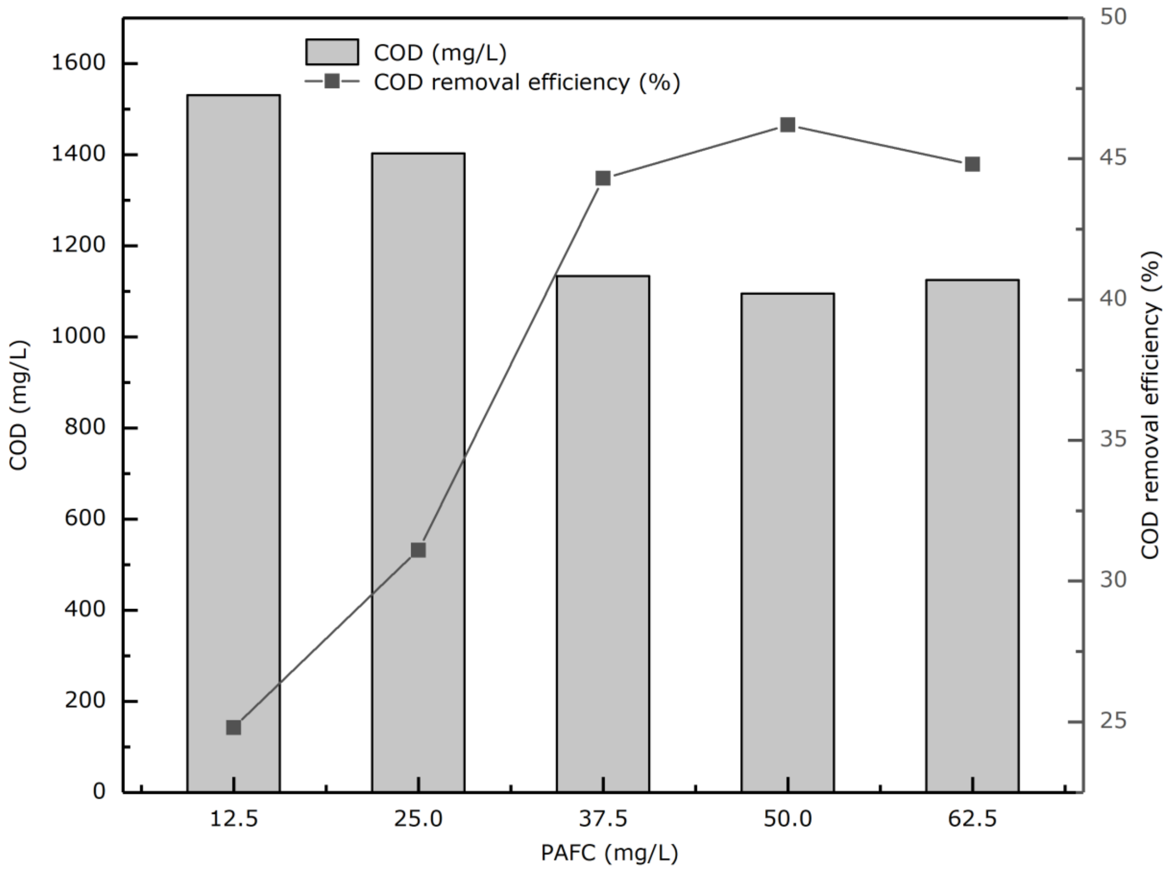

Figure 2. Effects of PAFC on removal efficiency of COD.

Effect of bioflocculant dosage on turbidity and COD removal

As depicted in Figure 3, with the increase in bioflocculant dosage, the turbidity removal efficiency increased, and the highest turbidity removal efficiency reached 40.6 % at 1.25 mg/l. However, there was a slight difference in the effect of turbidity removal at a dosage of 0.75-1.25 mg/l. With the dosage increase, the removal of COD by bioflocculant initially increased and then decreased in Figure 4. It can be seen from the figure that when the dosage was 1.0 mg/l, the highest COD removal efficiency was 30.0 %, and with the later increase in the dosage, the COD removal efficiency began to decrease.

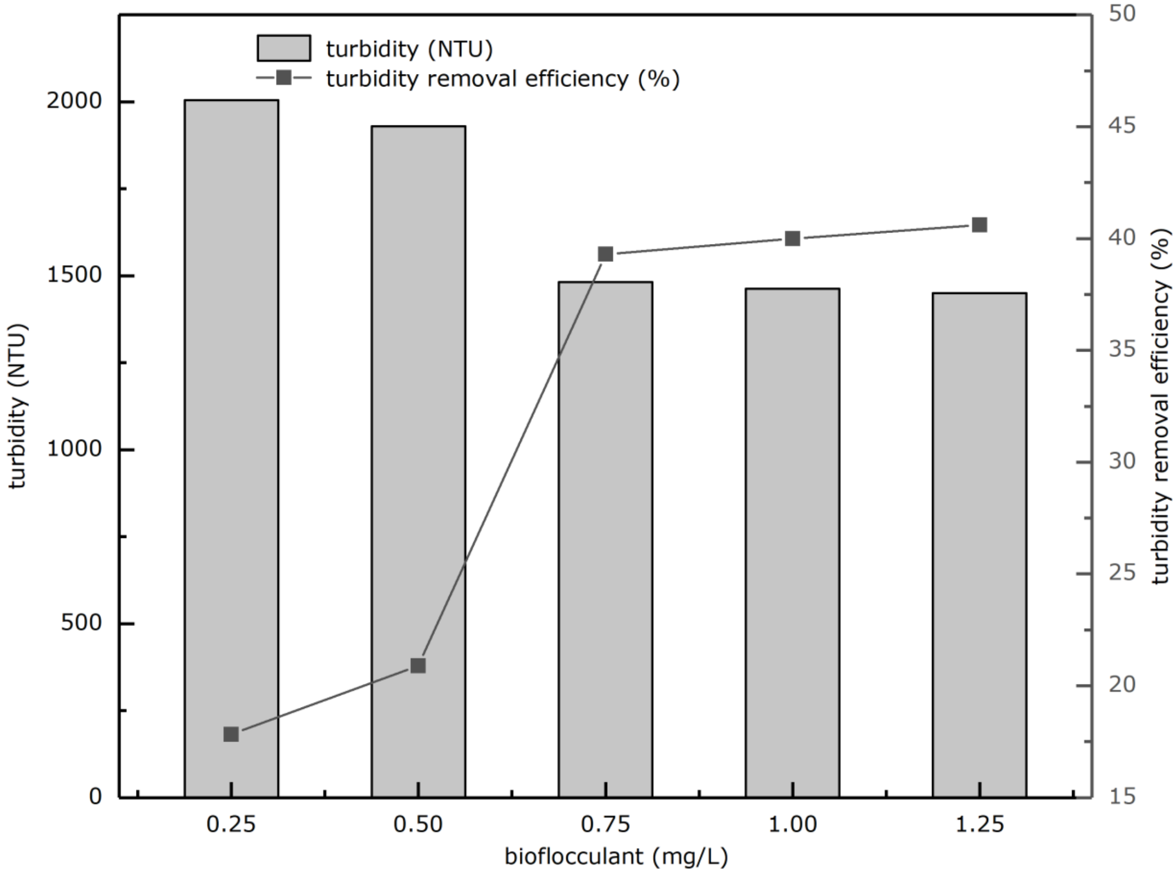

Figure 3. Effects of the bioflocculant on removal efficiency of turbidity.

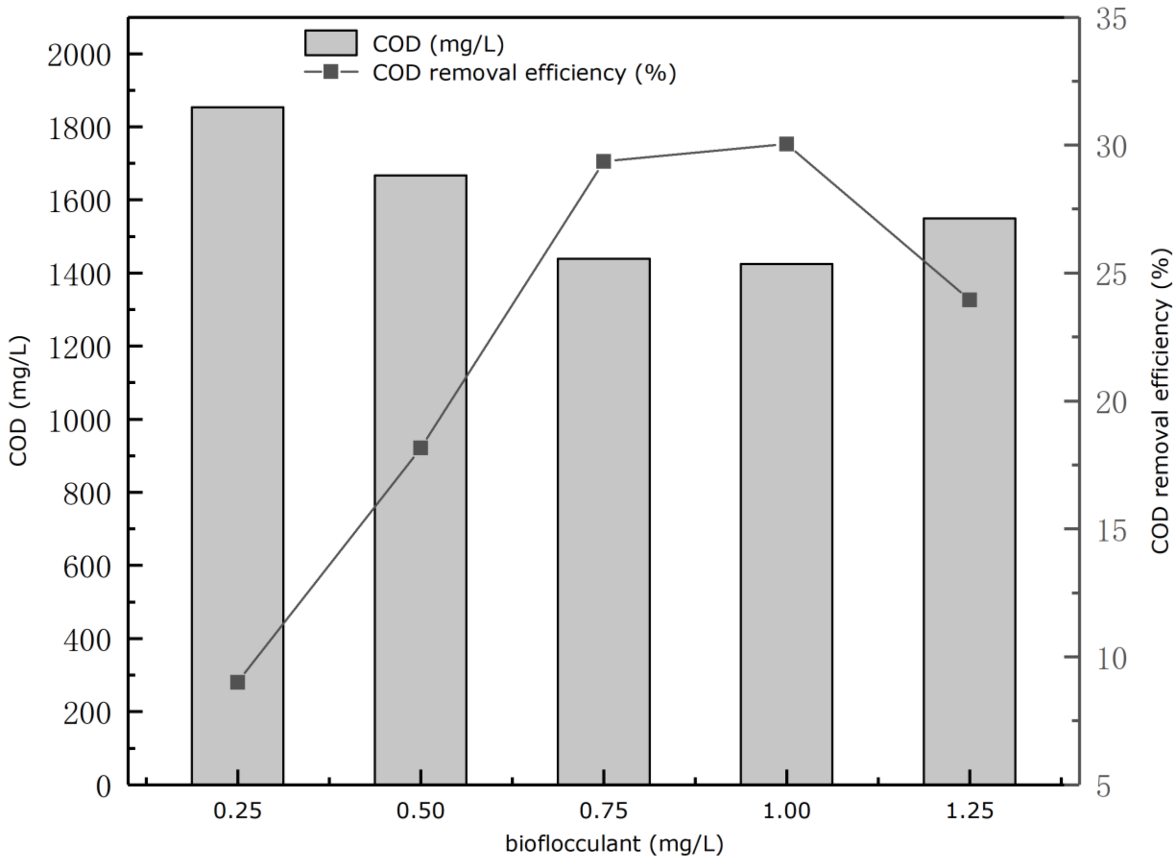

Figure 4. Effects of the bioflocculant on removal efficiency of COD.

Notably, there was a slight difference in COD removal efficiency at a dosage of 0.75-1.25 mg/l. When the dosage of the bioflocculant was more than 1.0 mg/l, there was excessive polysaccharide in the bioflocculant, which led to low COD removal efficiency (Liang *et al*., 2010). The above results also reflect that the addition of microbial reagent

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disrupted the charge balance of the coal water, and the polymer flocculant produced efficient flocculation settlement. Thus, the two reagents synergize slime water, and the ideal effect was achieved. A similar finding was documented by Gong *et al*. (2016), which reported that both the microbial reagent and polymer flocculant interacted with $CaCl_2$, and the three reagents have a synergistic effect on slime water.

Effect of pH on turbidity and COD removal

As shown in Figure 5, the turbidity of coal slime water firstly decreased and then increased with the pH increase from 5.0 to 9.0. When the initial pH was 7.0, the turbidity removal efficiency reached a peak (85.7 %). At the pH range of 5.0-9.0, the lowest turbidity removal efficiency was 39.2 %. The optimum pH value was 7.0, so it was necessary for the amount of acid and alkaline solutions to adjust the pH to be saved. This pH value was the same as the initial pH. When the pH was more than 7.0, the negative charge of impurities in coal slime water increases the electrode potential and repulsion between particles, which is not conducive to particle aggregation. When the pH was less than 7.0, it was not conducive to the hydrolysis of polyaluminium chloride to aluminum hydroxide polymer, affecting the coagulation of coal slime water. These results were similar to the previous results reported by Li *et al*. (2016) and Niu, Li, Zhao, Ren, and Yang (2011).

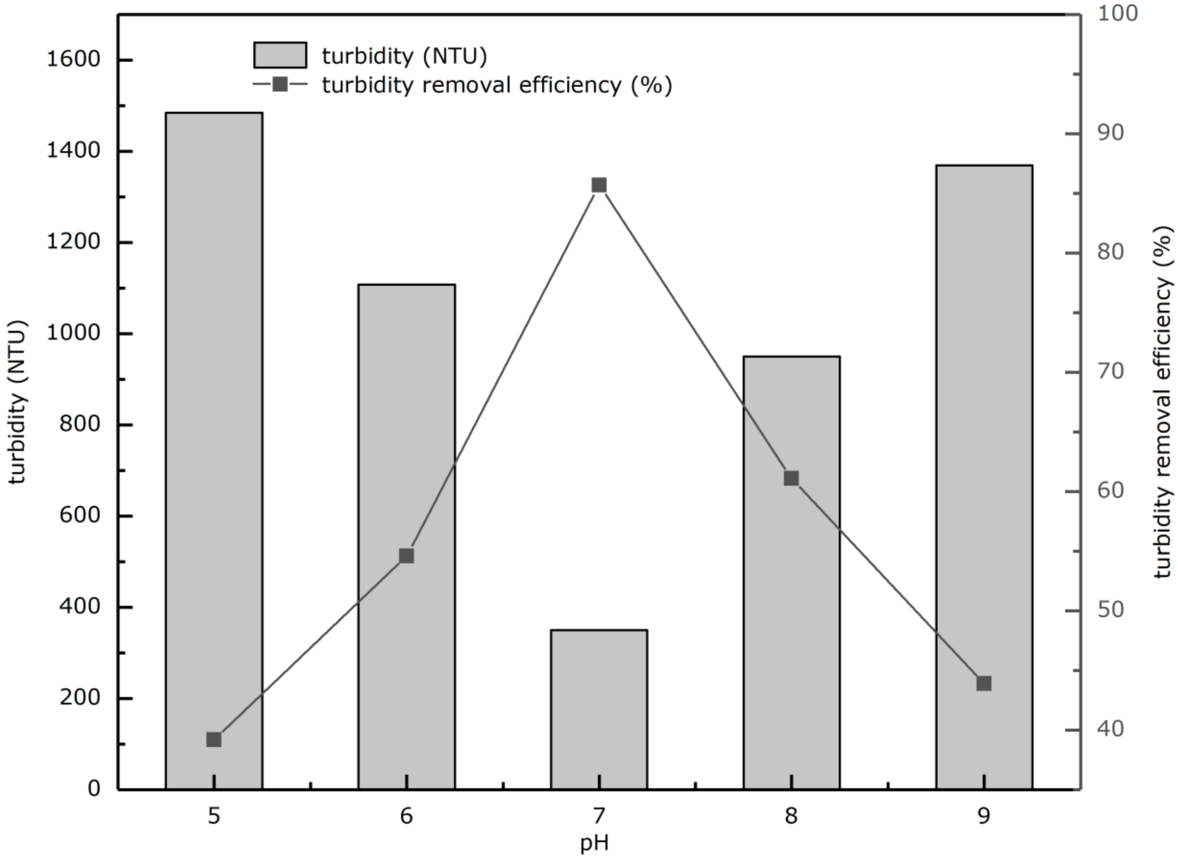

Figure 5. Effect of pH on removal efficiency of turbidity.

In Figure 6, with the increase of pH value, COD removal initially increased and then decreased. Under pH 7.0 conditions, the highest COD removal efficiency reached 44.2 %, and with the later increase in pH value, the COD removal efficiency decreased. Zeta potential is directly affected by the pH of slime water, an index affecting the stability of the colloidal dispersion system. With the increase of the absolute value of zeta potential, the stability of the colloid is enhanced. H^+ can neutralize some negative ions on slime's surface at a lower pH value and reduce the repulsion force between particles and zeta potential value. Meanwhile, with the increase of pH value, H^+ decreases, the HO^- ion content increases, the repulsion between particles increases, and zeta potential increases, so it can be seen that lower or higher pH is unfavorable to COD removal.

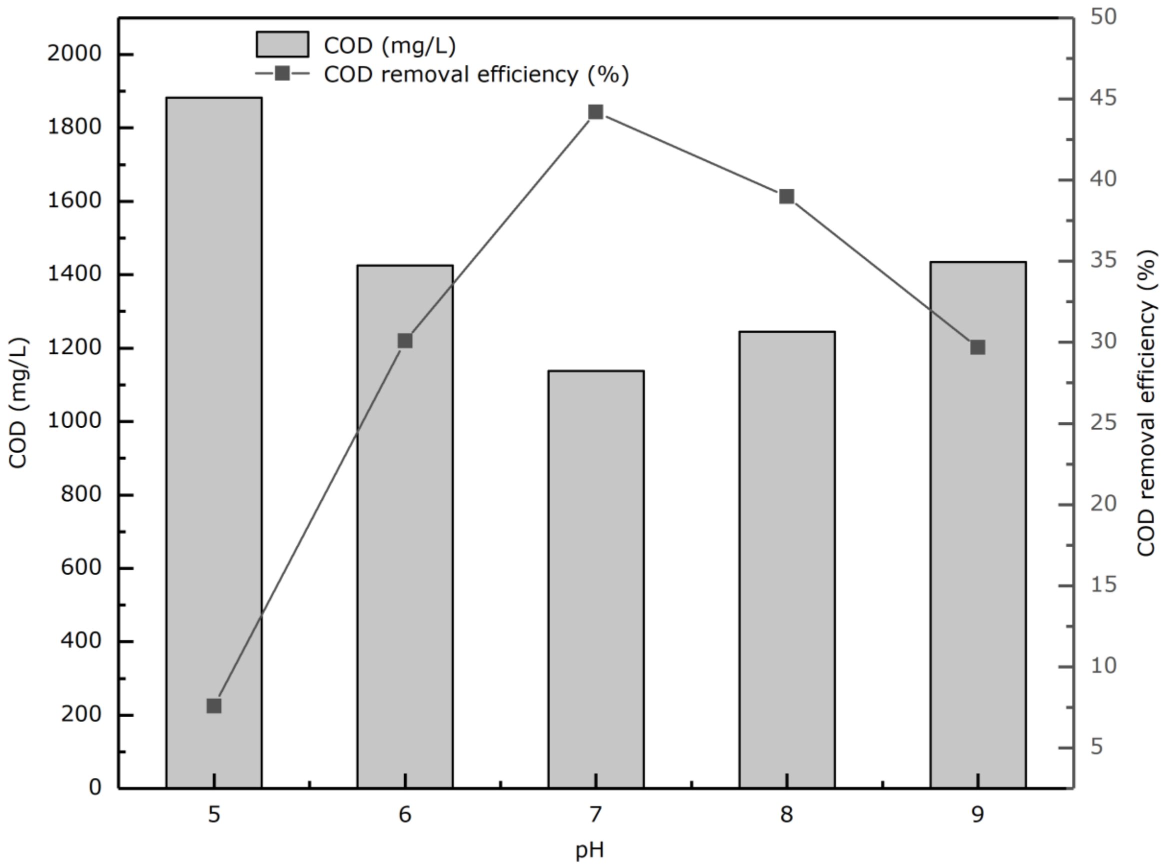

Figure 6. Effect of pH on removal efficiency of COD.

Orthogonal test on turbidity and COD removal efficiency

Orthogonal experiment design involves selecting some representative points from the overall test according to the orthogonality. This indicates that the variables are evenly distributed and independently evaluated in an orthogonal array. This method is an efficient and economical multi-factor experimental design method that can determine each variable's optimal conditions through limited experiments (Shi, Liu, Deng, & Yang, 2019; Peng *et al*., 2013). According to the experimental results of a single factor, three groups of data were selected for an orthogonal experiment with three factors and three levels (Significance level a = 0.05). See Table 1 for parameter selection.

Table 1. Design form of the orthogonal test L9 (3^3).

Levels	**Factor A**	**Factor B**	**Factor C**
	PAFC (mg/l)	**Bioflocculant (mg/l)**	**pH**
1	25.0	0.50	6.0
2	37.5	0.75	7.0
3	50.0	1.00	8.0

It can be seen from Table 2 that the combination of PAFC and bioflocculant can obtain a higher turbidity removal rate. By comparing the range(R), pH was considered the more significant factor in the coagulation-flocculation process. The influence degree of each factor on the turbidity removal rate was C > A > B. In other words, the effect of pH value on turbidity removal rate was the most significant, followed by PAFC dosage and bioflocculant dosage, and this was consistent with the variance result (see Table 3). This result can be attributed to the dissociation of coagulants in a specific pH range. An appropriate pH value can improve the dissociation degree and charge density of coagulant, which is conducive to coagulant molecules' diffusion and facilitates the bridging effect of bioflocculant. Consequently, it can be seen here that pH value plays an important role (Li *et al*., 2016).

Table 2. L9(3^3) orthogonal tests range analysis on the removal efficiency of turbidity and COD.

No	Factors			Removal Efficiency	
	A	**B**	**C**	**Turbidity (%)**	**COD (%)**
1	1	1	1	53.8	29.8
2	1	2	2	82.1	37.0

3	1	3	3	60.1	31.7
4	2	1	2	86.5	43.5
5	2	2	3	73.9	44.1
6	2	3	1	71.3	40.8
7	3	1	3	90.4	44.5
8	3	2	1	73.0	44.6
9	3	3	2	86.1	44.4
The removal efficiency of turbidity					
k1	65.333	76.900	66.033	/	/
k2	77.233	76.333	84.900	/	/
k3	83.167	72.500	74.800	/	/
R	17.834	4.400	18.867	/	/
The removal efficiency of COD					
k1	32.833	39.267	38.400	/	/

k2	42.800	41.900	41.633	/	/
k3	44.500	38.967	40.100	/	/
R	11.667	2.933	3.233	/	/

Note: k1, k2, and k3 were the average values of the sum of removal rates of different categories at various levels. R was the range, indicating the degree of influence of each factor on the results.

Table 3. Analysis of variance of turbidity and COD removal efficiency.

Index	**Factor**	**S_A**	**f**	**F**	**F critical value**	**Significance**
Turbidity	A	494.842	2	7.174	5.143	***
	B	34.376	2	0.097	5.143	*
	C	534.816	2	7.756	5.143	***
COD	A	238.336	2	13.639	5.143	***
	B	15.629	2	0.895	5.143	*
	C	15.696	2	0.900	5.143	*

Note: S_A is the sum of squares of deviations; *ƒ* is freedom; An F value that exceeds the F critical value is significant. ***indicates considerable effect, * suggests no significant effect and significance level a=0.05.

According to the k value and variance analysis, the best coagulation-flocculation condition that affects the turbidity removal effect was the combination of A3B1C2. Simply put, the best conditions for turbidity removal are listed as follows: the dosage of PAFC was 50 mg/l, the bioflocculant was 0.50 mg/l, and the pH was 7.0. Under these conditions, the turbidity removal efficiency of 93.2 % was obtained.

The R and variance (see Table 3) analysis results suggested that the order of factors affecting COD removal efficiency was A > C > B. The amount of PAFC had the most significant impact on COD removal efficiency, followed by the pH value and the amount of bioflocculant. The order of COD removal efficiency slightly differs from turbidity. From the above results, it can be inferred that the best combination of factors affecting the removal efficiency of COD in slime water is A3B2C2, which was the PAFC dosage of 50 mg/l, bioflocculant of 0.75 mg/l, and pH 7.0. Under these conditions, the removal efficiency of the COD was 47.5 %. The results illustrate that the water quality was obviously improved under the best treatment conditions of A3B2C2. So, adding bioflocculant into PAFC could enhance the flocculation performance due to the additional bridging bonds between bioflocculant and Al/Fe-particles. Under the effect of van der Waals force, charge neutralization, and strong bridging, the colloidal particles with COD formed a whole and finally precipitated from the water with gravity (Li *et al*., 2016).

There was little difference observed between the mean values of B1 and B2 under the best conditions of turbidity and COD removal efficiency. This is because the B factor has the lowest effect on turbidity and COD removal efficiency, and variance tests were not significant. Under the comprehensive consideration of flocculation efficiency and economic cost, the best condition for turbidity and COD removal efficiency were determined as A3B1C2. Under these conditions, the removal efficiency of turbidity and COD were 93.2 % and 47.2 %, respectively.

Conclusions

The single factor experiments reflect that the best removal effect of turbidity and COD of the coal slime water was: PAFC dosage 37.5 mg/l, bioflocculant dosage 0.75 mg/l, pH 7.0. The orthogonal test indicated that

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the pH value and the dosage of PAFC of the solution significantly impact the removal efficiency of turbidity and COD. Thus, the best turbidity and COD removal efficiency conditions were A3B1C2 and A3B2C2, respectively. Considering flocculation efficiency and economic cost, the best condition for turbidity and COD removal efficiency was finally determined as A3B1C2. The results demonstrate that the bioflocculant enhanced the treatment effects of PAFC for the coal slime water. Under this condition, the turbidity removal efficiency of 93.2 % and the COD removal efficiency of 47.2 % were obtained.

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