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Comparison of polysilicic acid (PSiA) and magnesium sulfate modified polysilicic acid (PMSiS) for effective removal of Congo red from simulated wastewater

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Abstract−Polymagnesium silicate sulfate (PMSiS) was prepared by using magnesium sulfate modified polysilicic acid (PSiA). The PMSiS's functional group and morphology were analyzed by FTIR and SEM, respectively. The coagulation performance of PMSiS and PSiA was studied and compared in terms of the Congo red (CR) dye removal efficiency. After modification, PMSiS was a complexation polymeric species with the compact rod-like structure. Compared with PSiA, PMSiS was more efficient and stable. In the process of treating simulated wastewater containing CR dye, the dye removal efficiency of PMSiS could achieve above 80% at the lower coagulant dosage of 0.4 g/L, while the dye removal efficiency of PSiA could only reach about 70% at the higher coagulant dosage of 1.2 g/L. Moreover, PMSiS had wider applicable range of dosage and temperature than PSiA at optimal conditions of pH 12 and once addition of feeding mode. Therefore, PMSiS will be a promising coagulant in the process of treating practical wastewater containing dyes.

Keywords: Polymagnesium Silicate Sulfate, Coagulation Efficiency, Congo Red Dye, Wastewater Treatment

INTRODUCTION

With the accelerated development of agriculture/industry, there is no denying that water pollution is becoming more and more serious [1,2]. A great many industries, including leather, textile, pharmaceutical, food, cosmetic and paper, should use organic dyes in the process of production [3]. Because of the complex molecular structures, stable nature and low biodegradability, organic dye is one of the most hazardous pollutants in wastewater. There is a tendency that the dye pollutants accumulate in living and food chain transmission [4,5]. And then they can do great harm to human beings. Therefore, various methods, including biological approach [6,7], oxidation [8,9], adsorption [3,10], and coagulation and flocculation (CF) [11,12] are used for the treatment of wastewater containing dyes. Among these processes, CF is more widely used, which is proved to be easy operation, economical competence and low environmental impact [13,14].

The feasibility and efficiency of the CF process mainly depends on coagulants. The performance of coagulant is usually relying on its own chemical composition and structure [15-18]. Hence, coagulants with high performance have been widely studied. For example, polyaluminum chloride was applied for the treatment of the oil sands process-affected water. The removal efficiency of turbidity can achieve more than 96% at the dosage range of 0.5-3.0 mM Al [19]. Polyferric sulfate (PFS) was prepared using a novel method which was coupled with bipolar membrane electrodialysis. The results show that the removal efficiency of turbidity increased to 93.43%, and energy consumption decreased to 2.72 kW·h/(kg H_2SO_4) [20]. Naturally, these inorganic polymer coagulants (IPC), due to their higher cationic charge and higher molecular weight, can achieve greater coagulation performance compared to conventional coagulants [21]. However, the coagulation efficiency of IPC is still low in comparison with organic polymeric coagulants [22]. To improve the coagulation efficiency of IPC, some new composite coagulants are produced by adding other additive components to inorganic coagulant. PSiA is one of the most widely used additive components. By adding PSiA, the molecular size of the new composite coagulants increases and its ability of aggregating is improved [23, 24]. For instance, polyferric silicate sulfate (PFSiS) and polyaluminum silicate chloride (PASiC) were synthesized through polysilicic acid (PSiA) combined with polyferric sulfate (PFS) and polyaluminum chloride (PAC) [25,26]. According to the results, PFSiS shows higher molecular size, stronger ability of aggregating and weaker ability of charge neutralization than that of PFS. PASiC has higher polymeric Al species fraction, more efficiency and lower stability than that of PAC. However, CF processes, which use Al and Fe coagulants for wastewater treatment, produce a great deal of sludge. Nothing can be recycled or reused from the sludge, which needs to be disposed either at landfill or ocean dumping [27]. Moreover, there is dispute about the possible adverse impacts of Al on human health and organisms [28]. The synthesized ferric coagulants may lead to metallic color and secondary pollution because of the excessive iron [29].

To overcome the problems caused by Al-based and Fe-based coagulants, Mg-based coagulants were proposed and studied for purifying dye wastewater [30,31]. One of the most attractive features of Mg-based coagulant is that the functional MgO photocatalyst can be recovered from the PMSiS coagulated sludge by incineration; and another one is that the magnesium ion is non-toxic. Thus the problem of sludge disposal may be solved and the chemical costs can be reduced [32]. Furthermore, the results in previous studies demonstrate that the magnesium-based coagulant can gain

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high dye removal efficiency. Thus, we were encouraged to develop and report the synthesis of polymagnesium silicate in earlier studies [12,33]. Poly-magnesium-silicate-chloride (PMSC) was prepared using $MgCl_2$ salts combined with PSiA. The emphasis in this research [12] is the effect of the molar ratio of Mg/Si on coagulation performance of PMSC, and the results have proved PMSC to be an efficient coagulant for dye simulated wastewater purification. Meanwhile, the focus of this research [33] is the effects of acid medium on the coagulation performance of PSMC. The results show that SO_4^{2-} has strong influences on the coagulation performance. Thus, the $MgSO_4$ as the modifier is expected to produce more efficient and stable poly-magnesium-silicate coagulants. Following our successful development of polymagnesium silicate, the objective of this work was to develop efficient and stable polymagnesium silicate sulfate (PMSiS).

In this study, PMSiS samples were synthesized using $MgSO_4$ salts combined with PSiA. Its coagulation performance and stability were studied for treatment of CR dye simulated wastewater and compared with PSiA. In the first stage, the functional group and surface morphology of PMSiS were investigated, supported by FTIR spectrometer and scanning electron microscopy (SEM). In the second stage, the coagulation performance of PMSiS for treatment of CR dye simulated wastewater was evaluated. To get an optimal operational parameter, these parameters including the coagulant dosage, feeding mode, pH and temperature in CR dye simulated wastewater were also investigated.

Table 1. All influence factors in CF experiment

Types of factors	Conditions
Dosage of coagulant (g/L)	0.4
	0.6
	0.8
	1.0
	1.2
	1.4
Feed mode	Once addition
	Interval addition
	Dropwise addition
pH in simulated wastewater	11.00
	11.50
	12.00
	12.50
	13.00
Temperature in simulated wastewater (°C)	20
	25
	30
	35
	40
	45
	50

MATERIALS AND METHODS

1. Materials

Magnesium sulfate ($MgSO_4$) and sodium silicate (Na_2SiO_3) were obtained from Sinopharm Chemical Reagent Co., sulfuric acid (H_2SO_4, purity≥98 wt%) was purchased from West long Chemical Co., Ltd. Conge red (CR) ($C_{32}H_{22}N_6Na_2O_6S_2$) was obtained from Tianjin Damao Chemical Factory. Other inorganic reagents were also purchased from Sinopharm Chemical Reagent Co. All chemicals used for the preparation of PMSiS were analytical reagent. Distilled water was used in all the experiments.

2. Methods

2-1. Synthesis of PMSiS Coagulant

PMSiS coagulants were fabricated according to our previous researches [33]. First, the pH of 6.3 wt% Na_2SiO_3 solution (dissolving $Na_2SiO_3 \cdot 9H_2O$ particles in water at room temperature) was regulated by the H_2SO_4 solution (the volume ratio of H_2SO_4 and H_2O was 1 : 1) until the pH of solution was 1.6-2.0. Then, the above solution was stirred for 1 h using a magnetic stirring apparatus (79HW-1, Jiangsu Jintan Jincheng Guosheng Experimental Instrument Factory, China). The polysilicic acid (PSiA) solution was obtained. Under normal stirring, 22.5 mL of 1 M $MgSO_4$ solution (1.25 mL/min) was dripped into the fresh PSiA solution, and then the solution was stirred for 60 minutes. Afterwards, the product of PMSiS was collected.

2-2. Characterization of PMSiS and PSiA

To obtain dried powder, the prepared liquid PMSiS and PSiA coagulants were dehydrated in oven at 50 °C for several days and then ground into powder. The morphology of PMSiS and PSiA was observed with SU1510 scanning electron microscopy (SEM). The functional groups in PMSiS and PSiA were evaluated by FTIR (Nicolet-380). The powders of PMSiS and PSiA were mixed with the conventional potassium bromide pellets (mass ratio of KBr and sample: 100 : 1) and then pressed into tablets. The wave-numbers were in the range of 4,000-400 cm^{-1}.

2-3. Coagulation and Flocculation (CF) Experiments

The effects of coagulant dosage, feeding mode, pH and temperature on coagulation performance of PMSiS and PSiA were investigated in the treatment of CR dye simulated wastewater. Table 1 shows all of influence factors in this research. While controlling these factors, the overall process of coagulation-flocculation is shown in Fig. 1. Congo red dye (CR) was dissolved in deionized water to obtain the simulated wastewater. The CR concentration in the wastewater samples was 100 mg/L. The maximum wavelength of Congo red is 498 nm. A certain volume (250 mL) of CR simulated wastewater was placed in a beaker and the pH regulated as needed. Then the coagulants were added into the CR dye simulated wastewater. All the CF experiments were done by using stirring equipment (Model RH-6, Changzhou Renhe Instrument factory, China) at room temperature. First, the wastewater was stirred at 300 rpm for 2 minutes and then slow mixing at 60 rpm was applied for 10 minutes. Afterwards, the mixture was maintained for 20 minutes under static conditions. Owing to the tiny amount of suspended matter, the water samples were taken from 2 cm below the surface of water for detecting residual dye concentration. The color analyses of samples were made by spectrophotometer (Model 722E, Shanghai Yuanxi Instrument Ltd., China).

2-4. Calculation of CR Dye Removal Efficiency

At the maximum absorption wavelength (λ_{max}), the absorbance

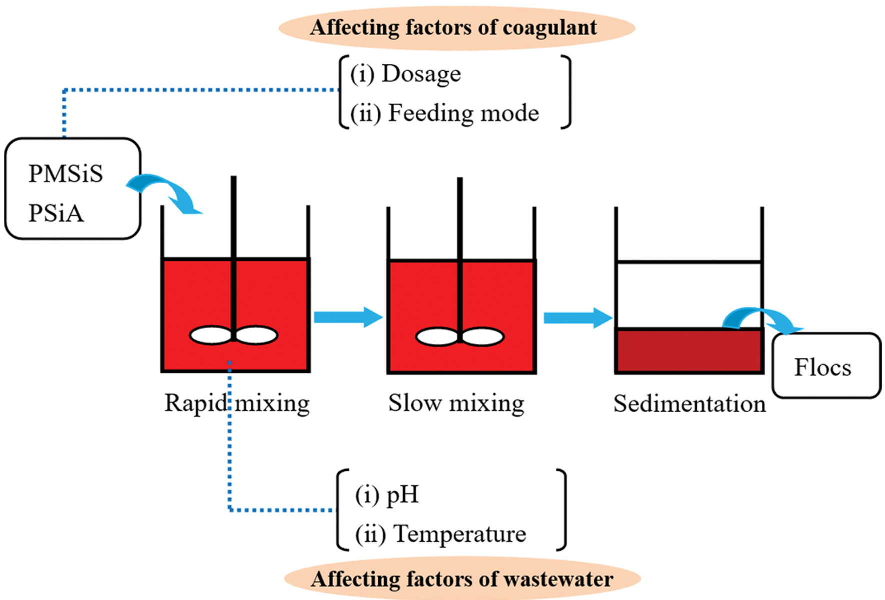

Fig. 1. The overall coagulation-flocculation process in this study.

of the water samples was linear with the CR dye concentration. As a result, the CR dye removal efficiency could be clearly identified. The CR dye removal efficiency (RE%) was determined according to Eq. (1) [13,34-36]

$$RE\% = \frac{A_0 - A_t}{A_0} \times 100 \quad (1)$$

where A_0 is the absorbance of initial CR dye simulated wastewater and A_t is the absorbance of water samples after coagulation run.

All the experimental data were average values determined through three independent experiments. The calculation error was about ±5%.

RESULTS AND DISCUSSION

1. Characterization of PMSiS and PSiA

1-1. FT-IR Spectroscopy

The IR spectra of PMSiS and PSiA are recorded in Fig. 2. The absorption peaks which are located at 3,425 and 3,459 cm^{-1} in PMSiS and PSiA spectra are intermolecular association stretching vibration of -OH [37]. The medium absorption peaks at range of 1,645-1,632 cm^{-1} of PMSiS and PSiA are bending vibration of water [38], while the peak of PMSiS is at 1,643 cm^{-1} and the peak of PSiA is at 1,634 cm^{-1}. It implies that PMSiS and PSiA comprise structural and adsorbed water. Furthermore, the absorption peaks at 1,141 cm^{-1} and 1,125 cm^{-1} can be attributed to stretching vibration of Si-O [12]. There is an obvious difference in absorption peaks between spectra of PMSiS and PSiA. The peaks at 1,091, 981, 636, and 460 cm^{-1} can be seen only in PMSiS. Among these, the peaks at 1,091 and 981 cm^{-1} are stretching vibration of Si-O-Mg [12], the other peaks at 636 and 460 cm^{-1} are bending vibration of Mg-O bond. Overall, the above IR analysis results prove that PMSiS coagulant is a complex compound of Mg and Si.

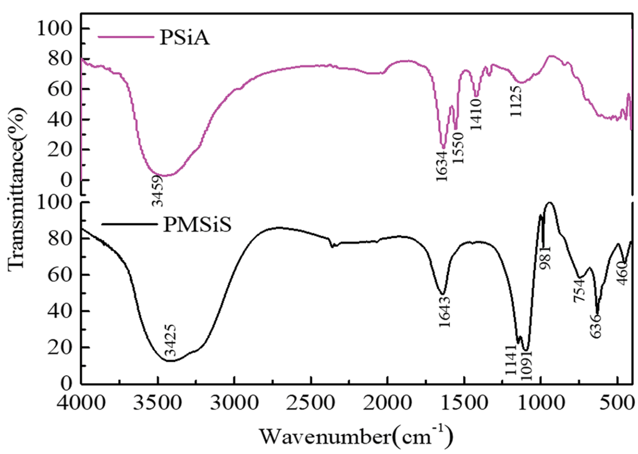

Fig. 2. IR spectra of PMSiS and PSiA.

1-2. SEM and EDS Results

The surface morphology of PMSiS which was prepared with magnesium sulfate is shown in Fig. 3. It is evident that PMSiS presents a compact rod-like structure. At the same time, PSiA displays a type of scattered minute particle structure. Because PSiA can produce a wide variety of silica species including $Si_4O_8(OH)_4^{4-}$, $Si_3O_5(OH)_5^{3-}$, $Si_2O_3(OH)_4^{2-}$ and finally polymeric species. As determined by EDS, the elements of Mg and S are presented in PMSiS. It shows that major elements of PMSiS are Mg, Si, O, S. And then $MgSO_4$ is beneficial to the polymerization between PSiA and Mg^{2+}. Due to the molecular compact rod-like structure of the components in PMSiS, charge neutralization and bridge-aggregation will be greatly strengthened when PMSiS is used for treatment of CR dye simulated wastewater.

2. Evaluation of PMSiS and PSiA

For the sake of investigating the stability and coagulation performance of PMSiS and PSiA, they were applied for treatment of CR dye simulated wastewater. The results of PMSiS were compared with that of PSiA.

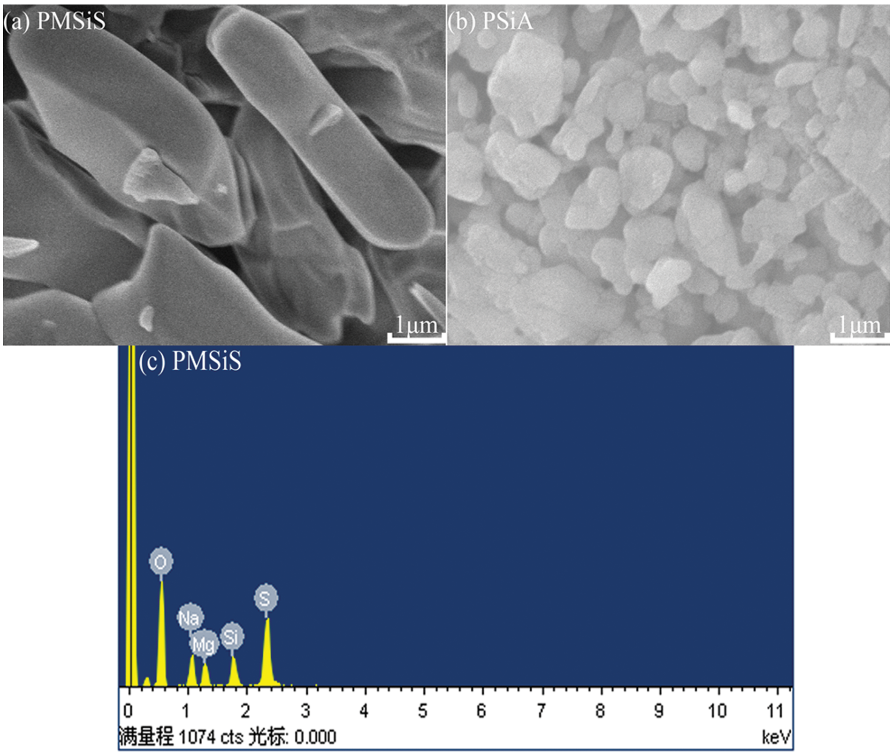

Fig. 3. SEM photographs of (a) PMSiS and (b) PSiA, (c) EDS analysis of PMSiS.

Table 2. The stability of PMSiS and PSiA

Coagulants	pH	Storage time (day)	Removal efficiency (%)	Appearance
PSiA	1.98	10	70.22	Transparent liquid
PSiA	1.98	15	-	Gelation
PMSiS	2.14	45	95.93	Transparent liquid
PMSiS	2.14	55	-	Precipitation

2-1. The Stability of PMSiS and PSiA

At room temperature, the stability of the samples was observed. The results are shown in Table 2. In comparison with PSiA's stability, the sample of PMSiS was more stable. On one hand, PSiA sample had become gelation after 11 days. On the other hand, PMSiS samples showed transparent liquid and high performance after 45 days. However, some precipitations had begun to be produced in samples of PMSiS after 55 days. This can be attributed to the fact that metal ions are generally considered as polymerization retardants [38]. The addition of metal ions may reduce the amounts of H_4SiO_4 and thus decrease the polymerization rate of PSiA. Accordingly, the stability of PMSiS was improved.

2-2. Effect of the PMSiS and PSiA Dosage

The removal of dye with PMSiS and PSiA coagulants is shown in Fig. 4. In the curve of PMSiS, the dye removal efficiency increased with the increase of dosage at the beginning. Subsequently, the dye removal efficiency had almost no change as dosage increased. The main reason is that high dosage will lead to strong ability of charge neutralization and adsorption-bridging. However, more flocs with charges are produced as the dosage of PMSiS coagulant further increases. As a result, the surface of aggregates is wrapped in the redundant flocs with charges. The more similar the charge is, the stronger charge repulsion is. And then the dye removal efficiency

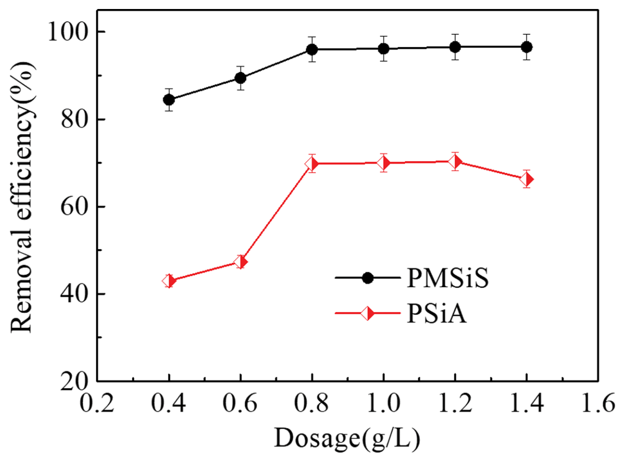

Fig. 4. Effect of PMSiS and PSiA dosage on the dye removal efficiency. The other factors were pH=12.00, the temperature= 20±2 °C and once addition of feeding mode.

had no obvious increase in the latter part of the dosage range. When it comes to the curve of PSiA, as the dosage increased, there was increase in the dye removal efficiency at the beginning. Afterwards, the dye removal efficiency showed a slight trend of decrease. At the coagulant dosage of 0.8 g/L, the highest dye removal efficiency of PMSiS could achieve 95.98%, but the highest dye removal efficiency of PSiA could only achieve 70.35% at the coagulant dosage of 1.2 g/L. In conclusion, the coagulation performance of PMSiS was better than PSiA's within the range of dosage. The Mg^{2+} can strengthen the ability of charge neutralization, adsorption and bridge-aggregation of PMSiS, which improves the coagulation performance of PMSiS.

2-3. Effect of the PMSiS and PSiA Feeding Mode

The particle size distribution in CF process relies on many factors. It is crucial that the supersaturation should be produced partially or whole in the tank at the feed point [39]. The CF experiments were done to study different feeding modes on properties of PMSiS and PSiA. The "once addition" feeding mode is a method of adding in a flash. The "interval addition" feeding mode is a method of half to half adding in 4 minutes. And the "dropwise addition" feeding mode is a method of slowly adding dropwise in 4 minutes. The dye removal efficiency with different feeding modes is presented in Fig. 5. The results showed that the highest the dye removal efficiency was the feeding mode of once addition. Moreover, the dye removal efficiency of PMSiS was all above to 80% under the three feeding modes. On the contrary, the dye removal efficiency of PSiA could only reach about 70% under the optimum feeding mode of once addition. The main reasons are as follows: PMSiS shows a stronger capacity for compressing the double layer electric field and enhancing charge neutralization. In general, the coagulant is the essential element which is made up for the floc formation structure and the specific surface area of the particles. Furthermore, the subsequent experiment process will benefit from the initial floc size. It is quite a common phenomenon that the high supersaturation will emerge at the feed point if the coagulant is added to wastewater with once addition feeding mode. In early stages of rapid mixing, more polymerization extent is formed and particles will be enlarged in a very short time. Compared with interval addition and dropwise addition, the initial floc size by once addition is relatively larger. It is revealed that the bigger flocs have resistance to shear. Consequently, the coagulant and the bigger flocs size have strong influence on the dye removal efficiency.

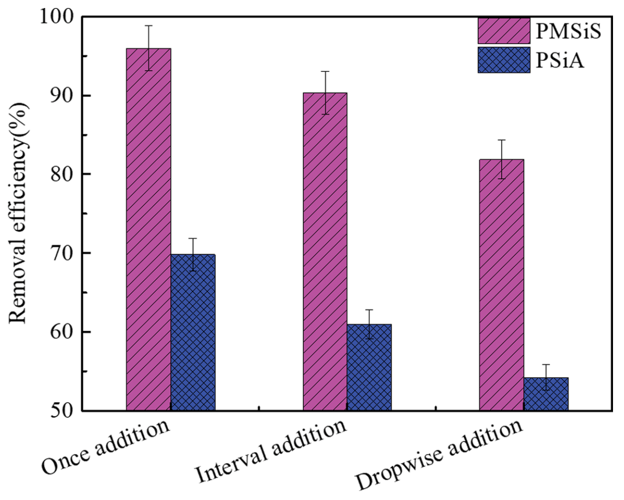

Fig. 5. Effect of PMSiS and PSiA feeding mode on the dye removal efficiency. The other factors were the dosage of coagulant= 0.8 g/L, pH=12.00, and the temperature=20±2 °C.

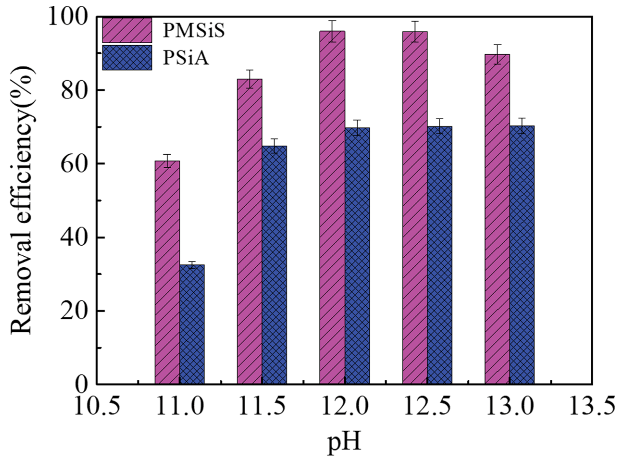

Fig. 6. Effect of pH in simulated wastewater on the dye removal efficiency. The other factors were the dosage of coagulant= 0.8 g/L, the temperature=20±2 °C and once addition of feeding mode.

2-4. Effect of pH in Wastewater

The dye removal efficiency was detected to determine the effects of the pH in simulated wastewater, as shown in Fig. 6. The solution of NaOH and HCl was applied to regulate the pH in simulated wastewater. The PMSiS coagulant showed that the dye removal efficiency increased within the pH range 11-12.5. As further increase of pH, the dye removal efficiency presented a slight decreasing trend. The main reasons are as follows: Because of the increase of pH, the more OH^- is, the more magnesium polymers will be generated, and this causes the ability of bridge-aggregation to enhance. Moreover, the colloids particle charges reduce under the mechanism of charge neutralization. And then the destabilization of the colloidal particles in wastewater will be strengthened. However, due to the high alkaline environment, the coagulant will be vulnerable to hydrolysis. So the bridging flocculation of coagulant may be inhibited. At the same time, the dye removal efficiency of PSiA could only gain about 70% at the pH range 12-13. The low dye removal efficiency of PSiA is because it will generate silica species with negative charge under the higher pH.

2-5. Effect of Temperature in Simulated Wastewater

Textile scouring and bleaching are usually performed at high temperature. So the temperature of the textile wastewater can reach 45±5 °C in summer [14]. PMSiS and PSiA were applied to treating CR dye simulated wastewater at temperature range of 20-50 °C. The results are shown in Fig. 7. The effect of temperature on the coagulation performance of PMSiS and PSiA was significantly different. The temperature had no apparent impact on the dye removal efficiency when PMSiS coagulant was used. When the coagulant of PSiA was applied to treating CR dye simulated wastewater, the dye removal efficiency decreased sharply at temperature range

Table 3. Comparison of the dye removal efficiency by Fe and Al coagulants, natural coagulants and the coagulant of PMSiS

Coagulant	The initial concentration of Congo red in wastewater (mg/L)	Optimum coagulant dosage (mg/L)	Optimum pH	The dye removal efficiency (%)
$Al_2(SO_4)_2$ [40]	187.92	80	8.26	84.37
PAC [41]	500	150	3-9	99.82
PFS [40]	77.82	141.66	6.35	98.28
Natural coagulants [42,43]	60	25	4	74-85
PMSiS	100	800	12	95.98

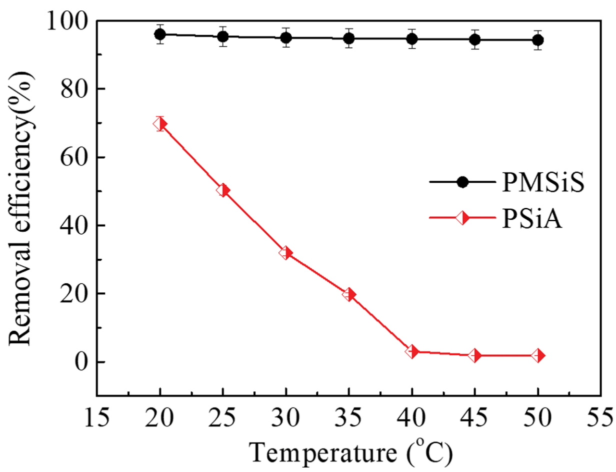

Fig. 7. Effect of temperature in simulated wastewater on the dye removal efficiency. The other factors were the dosage of coagulant=0.8 g/L, pH=12.00, and once addition of feeding mode.

20-40 °C and reached almost to zero at 40-50 °C. Therefore, the coagulation performance of PMSiS is proved to be undamaged at high temperatures. The primary reason of decreased dye removal efficiency of PSiA is that the main coagulation mechanism is adsorption [38]. The adsorption ability of PSiA gets worse and worse with temperature increase.

Moreover, the coagulation performance of PMSiS was compared with natural coagulants and traditional Fe, Al coagulants which were reported in these references [40-43]. The results are shown in Table 3. It was clearly indicated that the coagulation performance of PMSiS was more efficient than traditional $Al_2(SO_4)_2$ coagulants and natural coagulants. However, there were slight differences in the dye removal efficiency among coagulants of PMSiS, PFS and PAC.

CONCLUSIONS

A composite coagulant of PMSiS was fabricated by using $MgSO_4$ modified PSiA coagulant. The results of FTIR and SEM demonstrated that PMSiS is different from PSiA in their different morphology. According to the FTIR results, PMSiS is a kind of polymeric complex. SEM images showed that PMSiS presents compact rod-like structure while PSiA exhibits scattered minute particles structure. The colloidal particles are easily coagulated and the ability of bridge-aggregation among flocs is strengthened due to PMSiS's structure and morphology. Therefore, PMSiS showed more efficient coagulation performance and better stability than that of PSiA. At the lower coagulant dosage of 0.4 g/L, the dye removal efficiency of PMSiS could achieve above 80%, while that of PSiA could only reach about 70% at the higher coagulant dosage of 1.2 g/L. At optimal conditions of pH and feeding mode, PMSiS showed a wide applicable range of dosage and temperature. Moreover, SO_4^{2-} is a counter anion to magnesium. The hydroxylated magnesium polymers are linked together by SO_4^{2-} because of its high valence. Thus, the coagulation performance of PMSiS could be improved. In a word, PMSiS is a promising coagulant which is used to treat wastewater containing dyes. However, all experiments were done by treating Congo red dye simulated wastewater. Undoubtedly, there are still many studies, such as treatment of two or more dyes of wastewater, real wastewater and pilot experiments, to be done before PMSiS coagulant is to be put to practical use.

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