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Research Article

Treatment and Recycling of the Process Water in Iron Ore Flotation of Yuanjiacun Iron Mine

Wen-li Jiang[1] and Hai-feng Xu[2]

[1]*School of Resources and Environmental Engineering, Wuhan University of Technology, Wuhan 430070, China*
[2]*Changsha Research Institute of Ming and Metallurgy Co., Ltd., Changsha 410012, China*

Correspondence should be addressed to Hai-feng Xu; xuhaifeng0425@126.com

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Coagulating sedimentation and oxidation treatment of process water in iron ore flotation of Yuanjiacun iron mine had been studied. The process water of this mine carried residual polyacrylamide (PAM), poly(diallyldimethylammonium chloride) (PDADMAC), and Ca^{2+} from the flotation and caused decrease of the iron flotation recovery or grade of the concentrate. The studies on high-intensity magnetic separation (HIMS) tailings for coagulating sedimentation showed that the settling performance of coagulant (named CYH) was better than that of PDADMAC. The analyses of FTIR spectra and zeta potential demonstrated that CYH is adsorbed mainly through electrostatic attraction onto HIMS tailings. Sodium hypochlorite was adopted to oxidize the residual organics in tailings wastewater. When sodium hypochlorite is at the dosage of 1.0 g/L, reaction temperature is of 20°C, and reaction time is of 30 minutes, the removal rates of PAM, COD, and Ca^{2+} were 90.48%, 83.97%, and 85.00%, respectively. Bench-scale flotation studies on the treated tailings wastewater indicated that the iron recovery and grade of concentrate were close to those of freshwater.

1. Introduction

Iron ore resources are extremely rich in China, but most of them belong to complex ultrafine iron ore with high content of impurities [1]. Reverse flotation has been proved to be an efficient process for economic reasons [2–6]. In order to ensure iron concentrate grade and iron recovery, a large number of processing reagents are selected and applied in the iron ore beneficiation. The process water carried plenty of residual processing reagents, and such wastewater with color depth and strong smell could seriously affect the environment and the local people. Prevailing campaign for a cleaner and safer environment with clean surface water and ground water has led to increased recycling of process water within the production cycle of mineral flotation [7]. Since the chemistry property of process water is entirely different from fresh water, there is a concern about the possible effects of the contained components on the efficiency of the flotation process [8]. In iron ore flotation process, a large amount of NaOH is taken to adjust the pH. The wastewater pH of iron ore is more than 9, so physicochemical treatment of iron wastewater from a certain degree is rather difficult [9]. At present, the common methods of treatment of wastewater from flotation are acid-alkali neutralization [10, 11], precipitation, coagulation, and sedimentation [12–15], chemical oxidation degradation [16], constructed wetland [17–19], ion exchange method [20], adsorption method [21, 22], and biological method [23, 24]. However, the single method above cannot completely clear wastewater pollution with harsh operating conditions. In addition, they may also produce secondary pollution.

Yuanjiacun iron mine of Shanxi province in China is the largest iron mine of micrograined hematite combined with magnetite in Asia. The annual processing capacity of iron ore reaches 22 million tons, and the wastewater from mineral processing is about 400 thousand m^3 every day. If the dressing wastewater is directly discharged into the nature, it will not only pollute the surrounding environment but also has a great potential safety risk. Recycling of process water is a good way to solve the problem. In this study, the influence of tailings wastewater components were investigated on flotation of hematite in order to assess the practicability of

Table 1: Wastewater quality monitoring data.

Wastewater index	pH	SS/(mg·L^{-1})	Ca^{2+}/(mg·L^{-1})	Cl^-/(mg·L^{-1})	TFe/(mg·L^{-1})	PAM/(mg·L^{-1})	COD/(mg·L^{-1})
Content	9.12	178	240	210	4.97	3.15	131

Table 2: Particle size distribution of samples.

Size fraction/mm		+0.075	−0.075 +0.045	−0.045 +0.038	−0.038 +0.030	−0.030 +0.015	−0.015 +0.010	−0.010
Yield/%	Individual	3.14	5.37	2.50	5.42	10.57	42.35	30.65
	Aggregate	3.14	8.51	11.01	16.43	27.00	69.35	100

recycling process water in flotation practice first. A novel coagulant CYH was introduced to coagulate HIMS tailings, and the coagulating sedimentation mechanism of CYH was investigated by FTIR spectra and zeta potential. Moreover, sodium hypochlorite was adopted to oxidize the organic pharmaceutical residues in wastewater. Besides, the effect of wastewater treatment was evaluated by recycling of tailings wastewater.

2. Experiment

2.1. Materials and Reagents. All test samples including tailings wastewater, high-intensity magnetic separation (HIMS) tailings, and flotation samples were procured from the Yuanjiacun iron mine concentrator of Shanxi province in China. The tailings wastewater was collected from flotation tailings water combined with magnetic separation tailings water, stirred evenly, and stored in plastic bottle. The wastewater quality was shown in Table 1. Test results showed that the tailings wastewater presented yellowish brown, suspended solids (SS) content and the concentration of Ca^{2+}, Cl^-, and COD exceeded the standard.

The concentration of the HIMS tailings pulp was 11.93% by weight, and the main minerals were quartz (62.54%), amphibole (17.44%), chlorite (4.16%), hematite (4.75%), montmorillonite (3.82%), calcite (2.93%), and feldspar (4.36%). The XRD analysis of the sample was shown as Figure 1. The sample was classified into different size fractions as shown in Table 2. As shown in Table 2, most particle size of the sample was fine and more, even if the sample was on quiescent standing in a month, it also would not naturally subside.

The samples for flotation were taken from on-site samples of flotation, which were ground to 95% passing 0.045 mm, and contained 34.90% specularite and hematite, 8.70% magnetite, 0.20% limonite, 39.40% quartz, 11.40% chlorite and hornblende, and 4.90% calcite and dolomite.

Coagulant (CYH) of the molecular weight of 80 thousands was synthesized in our lab and it was of technical grade. Amphoteric polyacrylamide (PAM) of the molecular weight of 12 million was of technical grade. Collector (named RA-715) and coagulant poly(diallyldimethylammonium chloride) (PDADMAC) were from the Yuanjiacun iron mine concentrator of Shanxi province in China and they were of all technical grade. $CaCl_2$ and NaCl acted as sources of Ca^{2+} and Cl^- ions, respectively. Other chemical materials were bought from commercial companies and their purity was above chemical purity grade.

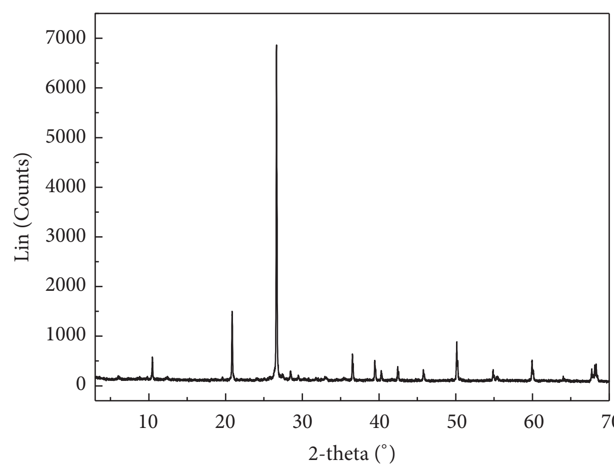

Figure 1: XRD analysis of the HIMS tailings sample.

2.2. Coagulating Sedimentation Experiments. Use 300 mL HIMS tailings pulp mentioned above at every turn to conduct coagulation contrast test. First, add the required amount of CYH into the testing pulp, and mix them up evenly; then add PAM into the testing pulp, and after a period of stirring, leave the samples standing and record the height of supernatant as a function of standing time.

2.3. Oxidation-Sedimentation Experiments. Use 1.0 L iron tailings wastewater mentioned above at every turn to conduct oxidation-sedimentation experiment. When the wastewater was heated to the desired temperature, sodium hypochlorite (10% of available chlorine) was added. After stirring the mixture for the desired time, $FeCl_2$ were added to the mixture, and the KI-starch paper was used to detect the reaction progress until it did not change blue. Afterwards, the pH was adjusted to 9~10 with NaOH solution. After several hours of standing, the supernatant was separated, and then the PAM concentration, COD value, and Ca^{2+} content were measured.

2.4. Batch Flotation Tests. The bench-scale flotation tests were conducted in a XFD-63 flotation cell (self-aeration) whose volume for rougher flotation and cleaning flotation was 0.5 L, using 200 g ore at every turn to obtain a Fe concentrate. Fatty acids (RA-715) were used as collector,

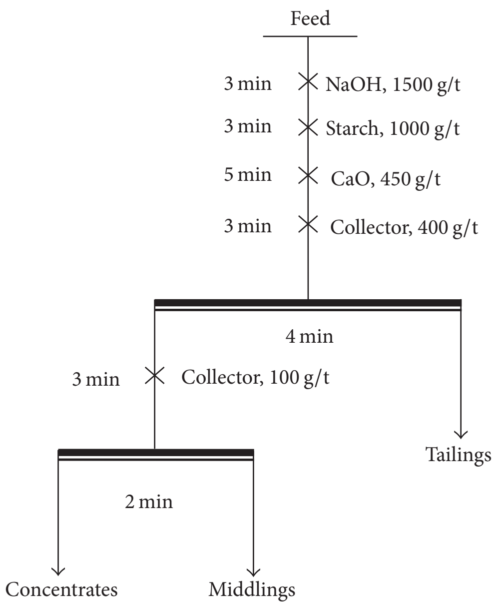

Figure 2: Flow sheet of flotation test.

NaOH was used as pH regulators, starch was used as depressant, and CaO was used as activator. The flotation flow sheet was illustrated in Figure 2.

2.5. FTIR Spectrum. The infrared spectra of samples were recorded by Nicolet AVATAR370 FTIR spectrometer (USA) using the KBr disk technique. The quartz samples used for this purpose were ground in an agate mortar and pestle to pass 5 μm. 50 mg of samples was mixed with 30 mL distilled water in the absence or presence of 100 mg/L coagulant at pH 9 and 25°C. After stirring for 3 min, still standing for 4 h, the solid product in the mixture was filtrated and rinsed three times and then dried in a vacuum oven and recorded infrared adsorption spectra from 400 cm^{-1} to 4000 cm^{-1}.

2.6. Zeta Potential Measurements. Zeta potentials of HIMS tailings samples were measured by using a Brookhaven ZetaPlus zeta potential analyzer (USA). The samples used for this purpose were ground to less than 5 μm in an agate mortar and pestle. 50 mg of the samples was added to 30 mL aqueous solution with or without 100 mg/L coagulant. After stirring for 10 min, then the pH values were adjusted with HNO_3 or NaOH solutions and measured. All measurements were conducted in a 0.1 mol/L KNO_3 background electrolyte solution. The agitated suspension was sampled to record the zeta potential. The results presented were the average of five independent measurements with a typical variation of ±5 mV.

3. Results and Discussion

3.1. Effects of Components Contained in Tailings Wastewater on Flotation Process. Bench-scale flotation studies on the process water showed that tailings wastewater reduced the flotation iron concentrate grade and iron recovery [25]. Large doses of coagulants such as PAM and PDADMAC were used in wastewater treatment in Yuanjiacun concentrator previously. Only in the part of HIMS tailings concentration, doses of PAM and PDADMAC were several times of other similar mines [25]. In order to assess the practicability of recycling of process water in flotation practice, the influence of tailings wastewater components of PAM, PDADMAC, Ca^{2+}, and Cl^- ions was investigated independently. The results were shown in Figure 3.

Figures 3(a) and 3(b) showed that an increasing PAM or PDADMAC concentration reduced the iron flotation recovery. However there was very little reduction in iron grade. When the concentration increased from 0 mg/L to 3 mg/L for PAM and 0 mg/L to 30 mg/L for PDADMAC, the recoveries of iron decreased from 74.66% to 66.74% and 74.66% to 64.63%, respectively. Figure 3(c) demonstrated that an increasing Ca^{2+} ions concentration reduced the iron grade and increased the iron flotation recovery obviously. When the concentration of Ca^{2+} ions increased from 0 mg/L to 180 mg/L, the iron grade of concentration decreased from 66.27% to 64.42%; the iron grade decreased sharply on addition of 360 mg/L. However there was a slight increase in recovery in process water in presence of a number of Ca^{2+} ions. Figure 3(d) showed that, with increasing the dose of Cl^-, there was no change in iron grade and recovery decreased only slightly.

3.2. Coagulating Sedimentation Experiments. Single factor tests of components contained in tailings wastewater showed that PAM, PDADMAC, and Ca^{2+} ions reduced the flotation iron recovery or grade of the concentrate. The origins of PAM, PDADMAC, and other organic species in tailings wastewater were the coagulants, flocculants, and flotation reagents such as PAM and PDADMAC for coagulating sedimentation of concentration and wastewater treatment, starch for depressing hematite flotation, and RA-715 for flotation of activated silicate minerals. The origin of calcium species in tailings wastewater was the ore and flotation reagents such as lime for activating SiO_2 and silicate minerals. The method of coagulating sedimentation was used in the process of concentration of HIMS tailings, flotation concentrate and flotation tailings, and treatment of wastewater of tailings pond in Yuanjiacun concentrator. In order to lower the content of suspended solids, PAM, and PDADMAC, coagulating sedimentation experiments were conducted with a novel coagulant CYH. Given the maximum amount of coagulants and flocculants used in HIMS tailings concentration in Yuanjiacun concentrator at present, we chose coagulating sedimentation testes of HIMS tailings concentration as a representative for detailed investigations to study the performance of CHY.

Figure 4 exhibited the effect of PDADMAC or CYH dosage on coagulation efficiency by using PAM as a flocculant at 26.88 g/(t undressed ore, the same below) initial concentration. The results in Figure 4 showed that, at the coagulant dosage of 8.96 g/t, the settle rate of CYH was faster than that of PDADMAC, even faster than that of PDADMAC at the dosage of 17.92 g/t. The turbidity of liquid supernatant by using CYH as a coagulant at 8.96 g/t initial concentration was lower than that of PDADMAC same as that of initial concentration and was rough equal to that of PDADMAC as

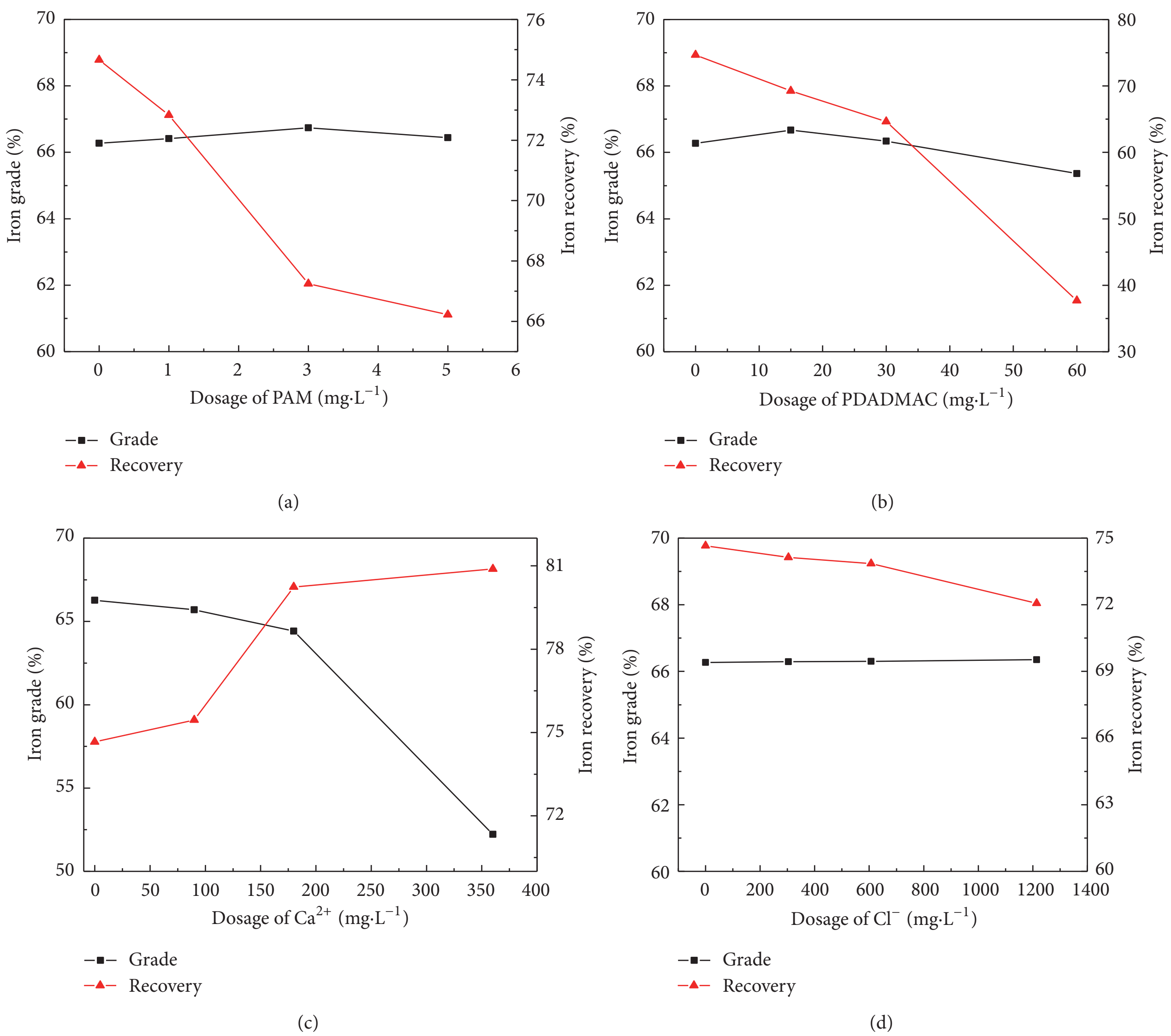

Figure 3: Effects of PAM (a), PDADMAC (b), Ca^{2+} (c), and Cl^{-} (d) on flotation process.

a coagulant at 17.92 g/t initial concentration. When the initial concentration of CYH increased from 8.96 g/t to 17.92 g/t, the settle rate and turbidity changed little. Compared with PDADMAC, CYH exhibited superior coagulating ability.

The flocculation response of PAM as a function of initial concentration by using CYH as a coagulant at 8.96 g/t initial concentration was presented in Figure 5. As it could be observed from Figure 5, the settle rate rapidly increased with increasing flocculant concentration. However, increasing PAM concentration had minor influence on turbidity of liquid supernatant.

3.3. FTIR Spectrum Analysis. As shown in Figure 1, quartz was the highest content of mineral in HIMS tailings. So, we chose quartz as a representative for FTIR spectrum investigation to study the adsorption mechanism of quartz before and after interaction with CYH. The FTIR spectra were presented in Figure 6.

Figure 6 showed that, after interaction with CYH, the stretching and bending vibrations of saturated C-H bonds in CYH molecules appeared at around 2962.17, 2933.24, 2871.53, and 1432.15 cm^{-1} on quartz surfaces, respectively. The results of FTIR spectra exhibited that, after CYH treatment, new adsorption peaks on quartz surfaces did not appear except for CYH's adsorption bands, which inferred that CYH might adsorb onto quartz surface without the formation of new complexes.

3.4. Zeta Potential Measurement. Zeta potentials of HIMS tailings particles as a function of pH values in the absence and presence of CYH were shown in Figure 7. It indicated that the potential of HIMS tailings was high. So coagulating sedimentation treatment of HIMS tailings was rather difficult from a certain degree because of the electrostatic repulse force among particles. As it could be observed from Figure 7, CYH could lower ζ-potential values within the scope of pH

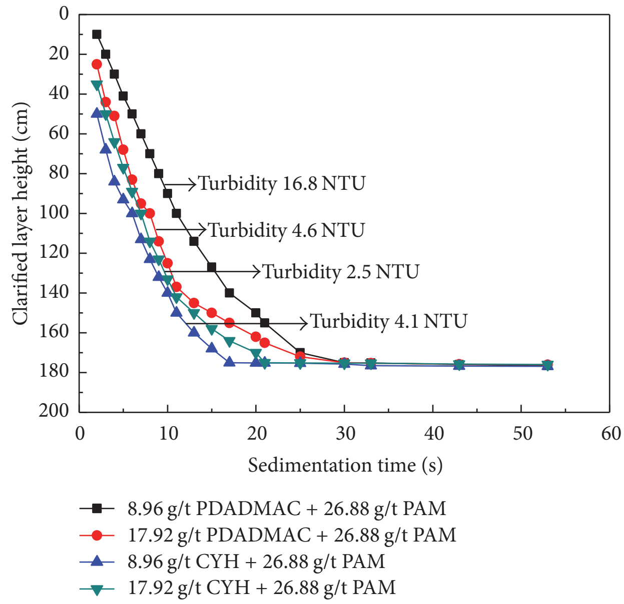

FIGURE 4: Effect of coagulation at different PDADMAC and CYH dosage.

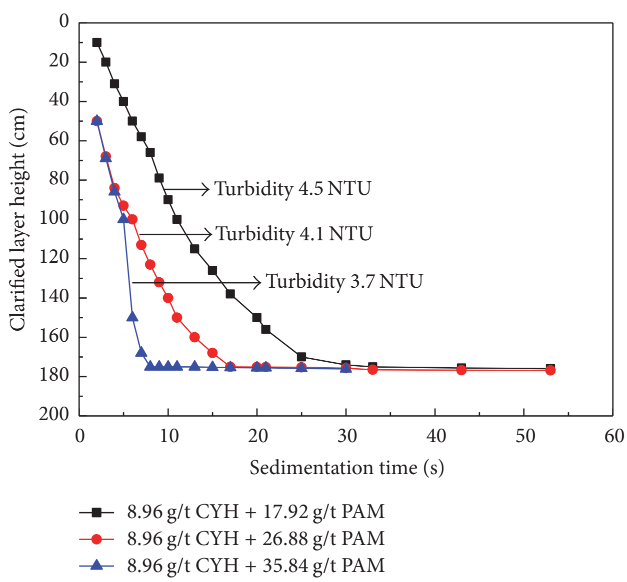

FIGURE 5: Effect of coagulation at different PAM dosage.

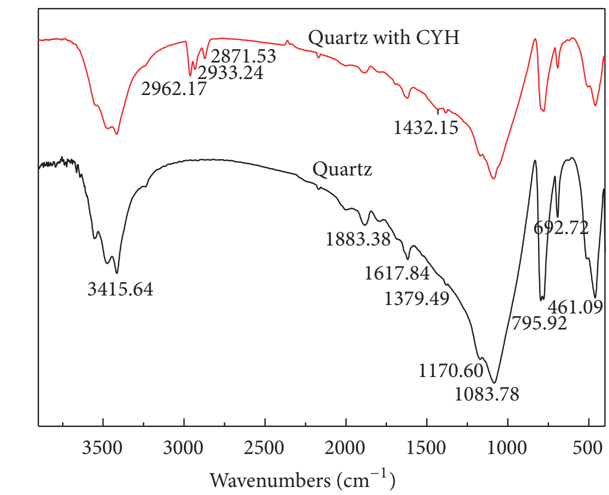

FIGURE 6: FTIR spectra of quartz before and after interaction with CYH.

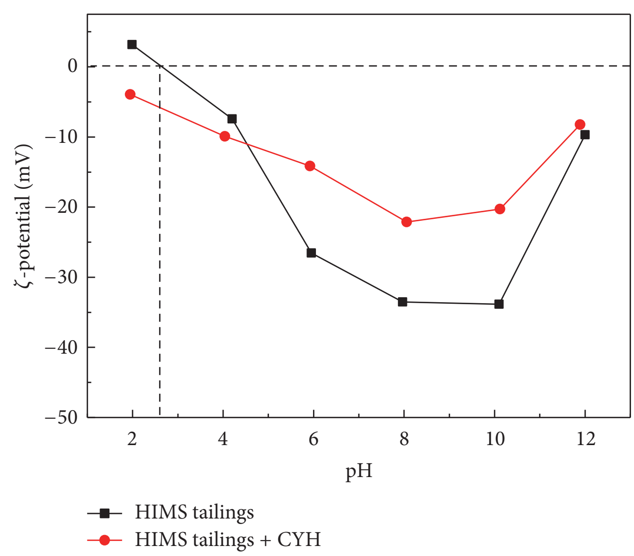

FIGURE 7: The zeta potential of HIMS tailings as a function of pH in the absence and presence of CYH.

5~12, inferring that cationic CYH might adsorb onto particles surfaces. The results of zeta potential indicated that CYH adsorbed onto HIMS tailings mainly through electrostatic attraction, which agreed with the FTIR spectra results.

CYH that is a good coagulant has strong binding force by using hydroxy. After the mixture of HIMS tailings and CYH, the stable silicate mineral groups in the wastewater were exposed; meanwhile, CYH entered into the wastewater and hydrolyzed strongly to be a polyhydroxy polymer compound. CYH adsorbed characteristically onto particles surfaces through electrostatic force, hydrogen bond, hydrophobic association, and van der Waals force, bridging the various silicate minerals in the long CYH chain, forming large flocs to reach rapid subsidence through trapping and rolling.

3.5. Oxidation-Sedimentation Treatment of Clarified Tailings Wastewater. Table 3 showed that, by using CYH as a coagulant, the content of SS of tailings wastewater decreased obviously, and the wastewater became clear. However, the COD value and concentration of PAM in tailings water were still much higher than freshwater. The tailings water still could not meet the requirements of reuse for flotation. Thus, oxidation-sedimentation treatment was still needed in the process.

We chose removal rates of PAM and COD as representatives for detail investigations to study the effect of reaction parameters. Figure 8(a) showed the effect of dosage of sodium hypochlorite (NaClO) on the removal rates of PAM and COD.

Table 3: Rendering of CYH treating tailings wastewater.

Water index	pH	SS/($mg \cdot L^{-1}$)	Ca^{2+}/($mg \cdot L^{-1}$)	TFe/($mg \cdot L^{-1}$)	PAM/($mg \cdot L^{-1}$)	COD/($mg \cdot L^{-1}$)
Tailings wastewater	9.12	178	240	4.97	3.15	131
Tailings wastewater after coagulating sedimentation treatment	9.15	75	196	3.86	2.81	103
Freshwater	7.94	45	142	0.19	0.26	4

Table 4: Water quality monitoring data table.

Water index	pH	SS/($mg \cdot L^{-1}$)	Ca^{2+}/($mg \cdot L^{-1}$)	TFe/($mg \cdot L^{-1}$)	PAM/($mg \cdot L^{-1}$)	COD/($mg \cdot L^{-1}$)
Tailings wastewater	9.12	178	240	4.97	3.15	131
Tailings wastewater after treatment	9.35	48	36	0.36	0.30	21
Freshwater	7.94	45	142	0.19	0.26	4

Table 5: The results of oxidation-sedimentation treatment on the reverse flotation of hematite.

Flotation water	Product	Yield/%	Grade/%	Recovery/%
Freshwater	Concentrate	48.80	65.14	73.97
	Tailing	49.10	20.91	23.89
	Feed	100.00	42.97	100.00
Tailings wastewater	Concentrate	47.90	64.20	70.74
	Tailing	49.45	23.46	26.69
	Feed	100.00	43.47	100.00
Tailings wastewater after treatment	Concentrate	48.85	65.45	73.49
	Tailing	49.80	21.89	25.06
	Feed	100.00	43.51	100.00

As shown in Figure 8(a), the removal rates of PAM and COD rapidly increased with increasing NaClO concentration when it was less than 1.0 g/L and then slowly increased and reached 90.48% and 83.97% at 1.0 g/L dosage, respectively. As it could be observed from Figure 8(b), the removal rates of PAM and COD increased with increasing reaction temperature. When the reaction temperature increased from 15°C to 30°C, the removal rates increased from 87.62% to 94.61% and 77.86% to 87.64% for PAM and COD, respectively. The reaction temperature is higher, the reaction rate is faster, but the decomposition of NaClO is faster. The choice of 20°C as the reaction temperature can ensure NaClO has good oxidation ability, but also conform to the most of natural temperature of tailings wastewater in Yuanjiacun concentrator. Figure 8(c) demonstrated that the removal rates of PAM and COD rapidly increased with prolonging reaction time when it was less than 30 min and then maintained roughly constant.

3.6. Recycling of Treated Tailings Wastewater. The quality of tailings wastewater which had been gone through oxidation-sedimentation treatment was shown in Table 4.

As shown in Table 4, after treatment by using oxidation-sedimentation method, pH of tailings wastewater increased from 9.12 to 9.35, suspended solids content decreased from 178 mg/L to 48 mg/L, and concentration of Ca^{2+} ions, TFe, PAM, and COD decreased from 240 mg/L to 36 mg/L, 4.97 mg/L to 0.36 mg/L, 3.15 mg/L to 0.30 mg/L, and 131 mg/L to 21 mg/L, respectively. And the removal rates of PAM, COD, and Ca^{2+} was 90.48%, 83.97%, and 85.00%, respectively. The water quality of treated tailings wastewater was close to the quality of freshwater.

Bench-scale flotation studies were conducted according to the procedure as shown in Figure 2. These studies would show whether oxidation-sedimentation treatment of the tailings wastewater occurring in the pilot plant had any effect on selectivity and/or recovery of minerals during flotation. Results were given in Table 5. The results showed that the concentrate yield, grade, and recovery increased by 0.95%, 1.25%, and 2.75%, respectively, by using treated tailings wastewater as flotation water compared to that of tailings wastewater without treatment. Compared to freshwater, in the case of the equivalent yield, treated tailings wastewater achieved an excellent concentration containing 65.45% Fe with 73.49% Fe recovery, and the Fe grade increased by 0.31%.

4. Conclusions

(1) Single factor tests of components contained in tailings wastewater in Yuanjiacun concentrator showed that PAM, PDADMAC, and Ca^{2+} ions reduced the flotation iron recovery or grade of the concentrate.

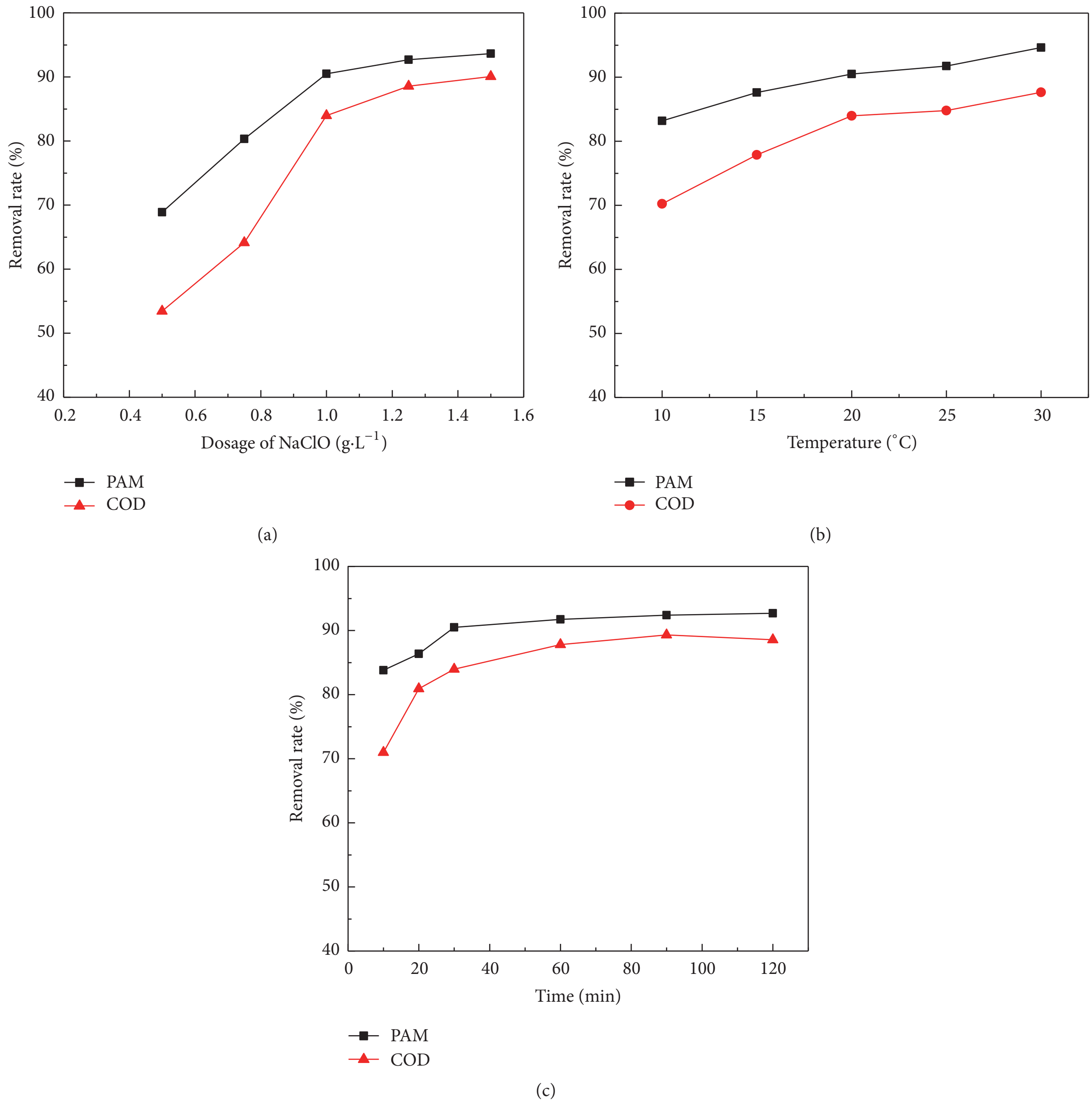

FIGURE 8: Effects of dosage of NaClO (a), reaction temperature (b), and reaction time (c) on the oxidation of tailings wastewater by using NaClO as oxidizing agent. Oximation conditions: (a) natural pH; temperature, 20°C; time, 30 min; (b) natural pH; dosage of NaClO, 1.0 g/L; time, 30 min; (C) natural pH; dosage of NaClO, 1.0 g/L; temperature, 20°C.

(2) When CYH was used to coagulate HIMS tailings, ζ-potential decreased, and silicate minerals formed flocculent mass by bridging; thus suspended matters decreased effectively. This was subsequently confirmed by FTIR spectrum and zeta potential analysis. But the tailings wastewater could not reach recycling standards.

(3) Oxidation experiment showed that a 90.48% reduction in PAM, 83.97% reduction in COD, and 85.00% reduction in Ca^{2+} were achieved at the sodium hypochlorite dosage of 1.0 g/L, reaction temperature of 20°C, and reaction time of 30 minutes. Bench-scale flotation tests on the treated tailings wastewater indicated that the Fe recovery and grade of concentrate were close to those of freshwater.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

Acknowledgments

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