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Operating conditions on the optimization and water quality analysis on the advanced treatment of papermaking wastewater by coagulation/Fenton process

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ABSTRACT

A coagulation/Fenton process was used for the advanced treatment of papermaking wastewater. Optimal reaction conditions were proposed based on the removal efficiency and the operating costs. The overall removal efficiency of chemical oxygen demand (COD) reached 86.5% for polyaluminium chloride and polyacrylamide concentrations of 120 and 1 mg/L, H_2O_2 dosage of 4 mmol/L, and $[H_2O_2]/[Fe^{2+}]$ molar ratio of 1. The distribution and transformation of the polarity and molecular weight of the organic matter in raw wastewater, in the coagulation process effluent, and in the Fenton process effluent were also studied. The coagulation process efficiently removed the hydrophobic fraction, which comprised 60% of the fractions in the raw wastewater. The decrease in COD during the coagulation process reduced the required H_2O_2 dosage in the subsequent Fenton process. The cost of the coagulation/Fenton process amounted to 0.78 RMB/m^3, which was lower than that of the classic Fenton process alone (1.39 RMB/m^3). Thus, this study offered an efficient and cost-effective method for the advanced treatment of papermaking wastewater.

Keywords: Papermaking wastewater; Coagulation/Fenton process; Water quality analysis; Economic analysis

1. Introduction

Papermaking wastewater is an industrial substance that is difficult to treat because of its high suspended solid (SS) content, high color, and poor biodegradability. Primary clarification is the main wastewater treatment process that is currently being used in the papermaking industry. A biological treatment unit is the secondary treatment applied to wastewater. However, the effluent still contains considerable levels of chemical oxygen demand (COD), toxicity, and color [1,2] even after the above-mentioned biological process. COD, which originates from persistent substances in the wastewater, cannot be further reduced by biological process alone [3]. The colored components of wastewater are lignin and lignin derivatives, and the previous studies proved that the maximum color treatment efficiency is only 30% when the biological process is used [4]. With the improvement of environmental protection standards and the need of energy-saving and reducing emission applications, an advanced treatment for papermaking wastewater is

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necessary to satisfy the wastewater discharge quality and to reuse wastewater. Some methods such as the nanofiltration process [5], electrocoagulation [6], coagulation–flocculation process [7], and advanced oxidation processes (AOPs) [8] have been employed for advanced treatment.

Fenton oxidation, which is the most common method of AOPs, has been widely used to treat papermaking wastewater because of its high oxidation efficiency, easy handing, and environmental friendliness [9,10]. The classic Fenton process is characterized by the reaction of ferrous ions with hydrogen peroxide (H_2O_2) to generate hydroxyl radical ($HO^{\cdot}$) which has great potential to degrade recalcitrant organic pollutants [11]. The selection of the better treatment process, besides considering the removal efficiency of pollutants, should also take into account the treatment costs. Though H_2O_2 plays a key role as an oxidant in Fenton reaction for generating the $HO^{\cdot}$ [12,13], the running cost of this method is higher than that of the conventional chemical process because of the high cost of H_2O_2. Some rational measures should be put forward to minimize the operating costs of Fenton process including the optimization of H_2O_2 concentration. On the other hand, the influence of wastewater characteristics (polarity and molecular weight (MW)) on Fenton process should be taken in consideration, but there is a lack of sufficient information on these for guidance.

Meanwhile, coagulation was used for treating pulp and papermaking wastewater to remove lignin and other colloidal solids [14–17]. Previous studies have tested polyaluminium chloride (PAC), polyferric, and polyacrylamides (PAMs) as a flocculants in the coagulation process. These coagulants efficiently remove lignin and SS. Therefore, the combination of coagulation and Fenton oxidation can be used as an attractive method for treating wastewater [18]. Ma and Xia [19] showed that combining the Fenton process with the coagulation process was effective in the treatment of water-based printing ink wastewater. The overall color, COD, and SS removal efficiencies reached 100, 93.4, and 87.2%, respectively. Moreover, Fenton/coagulation was successfully used for detoxifying dye effluent [20,21] and for treating olive mill wastewater [22]. However, the removal efficiency and operating costs of the coagulation/Fenton process on papermaking treatment have not been studied in detail yet.

In this study, the effectiveness of the coagulation/Fenton process in treating papermaking wastewater was explored. Experiments were conducted to examine the effects of various operating conditions on the performance of the treatment system; and the distribution and transformation of the polarity and MW of organic matters in wastewater were investigated to elucidate the performance of the coagulation/Fenton process .

2. Materials and methods

2.1. Wastewater samples

Papermaking wastewater was obtained from the secondary sedimentation tank of Tranlin Company in Shandong Province, China. The wastewater was slightly alkaline (pH 7.6). The concentration of COD ranged between 130 and 250 mg/L, and mean value of biological oxygen demand after 5 d (BOD_5) was 20 mg/L. The SS concentration was 100–130 mg/L and the color ranged 20–50 times based on dilution method.

2.2. Coagulation/Fenton process

2.2.1. Coagulation process

The coagulation process was carried out with 1 L of water samples in a jar test apparatus. Based on the preliminary experiments, PAC was chosen as the efficient coagulant. The optimal coagulant was studied further under different operating conditions involving various pH values and coagulant dosages. The optimal coagulant was added to 1 L wastewater samples and the solution was stirred for 5 min at 200 rpm. Then 1 mg/L PAM was added to the solution for further mixing for 2 min. The treated solution was left to standstill for 30 min to settle out the flocs. The pH value was adjusted to the desired level by using HCl or NaOH solutions.

2.2.2. Fenton process

The Fenton oxidation experiments were conducted by using the jar test method at room temperature and atmospheric pressure. The operating parameters included pH value, $[H_2O_2]/[Fe^{2+}]$ molar ratio, and H_2O_2 dosage. The initial pH value was adjusted by using HCl or NaOH solutions. $FeSO_4{\cdot}7H_2O$ and H_2O_2 (30%, w/w) were added to the solution under vigorous magnetic stirring. NaOH solution was added after the reaction duration to increase the pH value to terminate the Fenton reaction . The supernatant was withdrawn for COD analysis after allowing the treated wastewater to stand for 30 min.

The flow diagram of the coagulation/Fenton process is showed in Fig. 1. The experimental wastewater was initially subjected to the coagulation

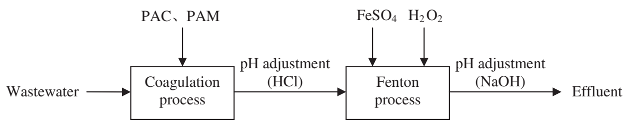

Fig. 1. The flow diagram of the coagulation/Fenton process.

process with the presence of the PAC and PAM. Then the supernatant water was withdrawn and subjected to the Fenton process.

2.3. Water quality analysis

2.3.1. Polarity distribution

The polarity (hydrophobic (HPO)/hydrophilic (HPI)) distribution of wastewater was analyzed based on the method of Quanrud [23]. The organics were extracted and fractionated using XAD-8/XAD-4 resin chromatography. These resins were pretreated before the experiments [24]. The water samples were acidified (pH = 2) and passed through two columns in a series filled with XAD-8 and XAD-4 resins. Three-way distributions of dissolved organic carbon (DOC) were obtained based on fractions retained on the XAD-8 and XAD-4 resins. The retained fractions on XAD-8 were HPO, whereas the retained fractions on XAD-4 were transphilic, and the non-retained fractions on both resins were HPI [23].

2.3.2. MW distribution

The MW distributions were determined by ultrafiltration method [25] using a 300 mL stirred ultrafiltration cup (SINAP, SCM-300, China). The samples were filtered through 0.45 μm and passed through the ultrafiltration membranes with nominal MW cutoffs of 30, 10, 3, and 1 kDa successively. The MW distribution was categorized as follows: 0–1 kDa, 1–3 kDa, 3–10 kDa, 10–30 kDa, and 30 kDa–0.45 μm. The driving force for filtration was 0.15 atm with nitrogen gas (99.5%). The used membranes were cleaned with 1 mol/L NaOH and 1 mol/L HCl.

2.4. Chemical analysis

COD was determined using the spectrophotometry method (Hach DR2800, American). The pH value was measured using a portable pH analyzer (pHB-2, China). Total organic carbon (TOC) was determined using a TOC analyzer (Shimadzu, TOC_V_{CPH}, Japan).

3. Results and discussion

3.1. Coagulation process studies

Using the coagulation process, to remove lignin and other colloidal solids in papermaking wastewater was cost-effective [8]. Furthermore, this process could considerably reduce the COD of the wastewater, thereby resulting in lower H_2O_2 requirement in the subsequent Fenton process [12]. Experiments on pH and PAC dosage were performed to optimize the parameters that affect the coagulation efficiency.

3.1.1. Effect of pH

The pH value is an important parameter that affects the coagulation efficiency [17]. Fig. 2 shows the efficiency of coagulation with PAC at different pH values. The maximum COD reduction (66%) was observed at pH 5. COD removal efficiency decreased after increasing or decreasing the pH value (from pH 5). The same COD reduction phenomenon was observed when paper and pulp mill effluent was subjected to the coagulation process [26]. Therefore, the pH value of 5 was selected for the following experiments. PAC hydrolysis was performed, and complex ions with high positive charge and low polymerization degree were formed at low pH; charge neutralization was selected as the main mechanism of the coagulant [17]. PAC reduces coagulation efficiency by forming the free aluminum ions at extremely low pH levels. The coagulation function will decrease because of the

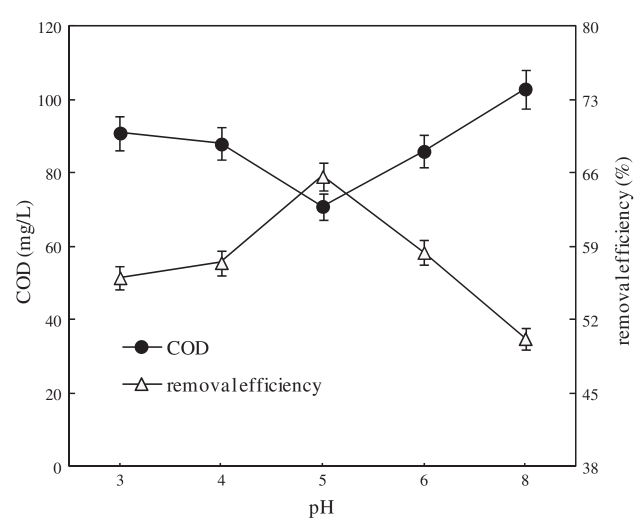

Fig. 2. Effect of pH on COD removal of treated water in the coagulation process (conditions: PAC, 120 mg/L; PAM, 1 mg/L; COD of raw wastewater, 207 mg/L).

reduction of the degree of PAC depolymerization when the pH level is too high [27].

3.1.2. Effect of PAC dosage

Fig. 3 shows the effect of PAC dosage on COD removal performance. COD reduction increased with increasing PAC dosage. According to the adsorption and charge neutralization theory, the negatively charged colloids in wastewater are adsorbed by the positively charged colloids of dissociation of PAC. On the other hand, a slow decrease of the COD removal efficiency was observed when PAC dosage exceeded 144 mg/L. It might be contributed to the re-stabilization of colloidal particulates when PAC dosage was used in excess of optimum dosage value. The COD removal efficiency exceeded 60% when the PAC loading was 120 mg/L.

3.2. Fenton process studies

3.2.1. Effect of pH

The pH value is an important factor in the Fenton process. The effects of pH on COD removal efficiency are shown in Fig. 4. COD removal efficiency increased with increasing pH level from 2 to 3. The removal efficiency was maintained between 55 and 63% with increasing pH levels from 3 to 6. The highest oxidation removal efficiency of COD was achieved at pH 3. The oxidation removal efficiency decreased at low pH (<3) because of the formation of complex species $[Fe(H_2O)_6]^{2+}$, which reacted slowly with peroxide [28]. At high H^+ concentrations, the peroxide was solvated to form a stable peroxone ion $[H_3O_2]^+$, thereby reducing the reaction between peroxide and ferrous ion [28]. COD removal efficiency began to decrease slightly at pH 4 owing to the autodecomposition of H_2O_2 and the deactivation of iron ion to iron oxyhydroxides [19]. Therefore, a pH value of 3 was chosen for all the Fenton process experiments in this study.

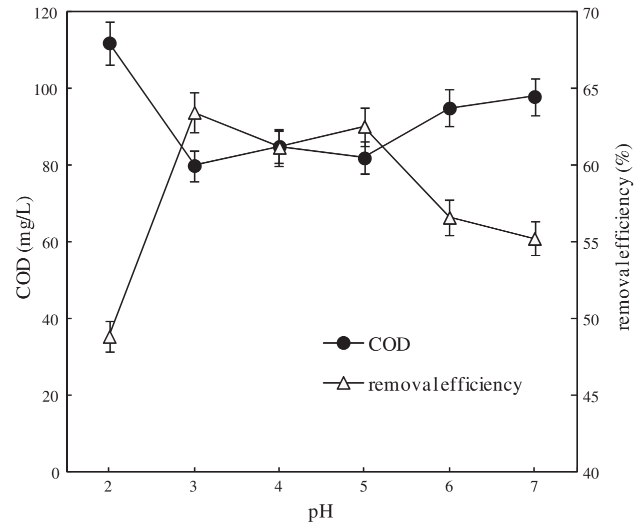

Fig. 4. Effect of pH on COD removal of treated water in the Fenton process (conditions: $[H_2O_2]/[Fe^{2+}]$ = 1; $[H_2O_2]$, 2 mmol/L; COD of raw wastewater, 219 mg/L).

3.2.2. Effect of $[H_2O_2]/[Fe^{2+}]$ molar ratio

A favorable $[H_2O_2]/[Fe^{2+}]$ molar ratio is worthy to study in Fenton process. In this study, the molar ratios of $[H_2O_2]/[Fe^{2+}]$ ranged from 8:1 to 1:4 were used to

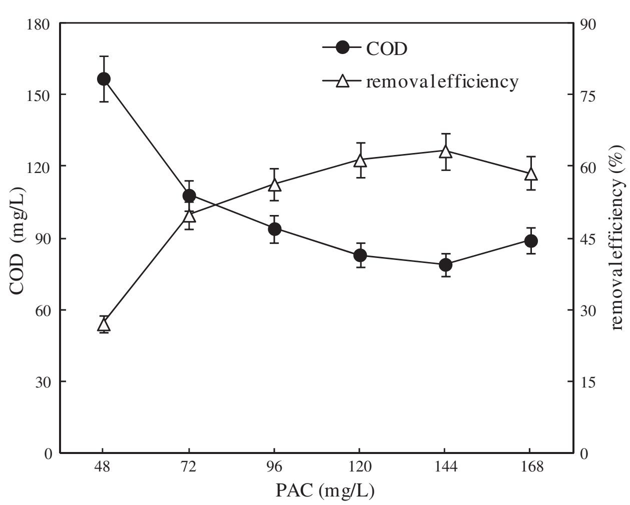

Fig. 3. Effect of PAC dosage on COD removal of treated water in the coagulation process (conditions: PAM, 1 mg/L; pH 5; COD of raw wastewater, 215 mg/L).

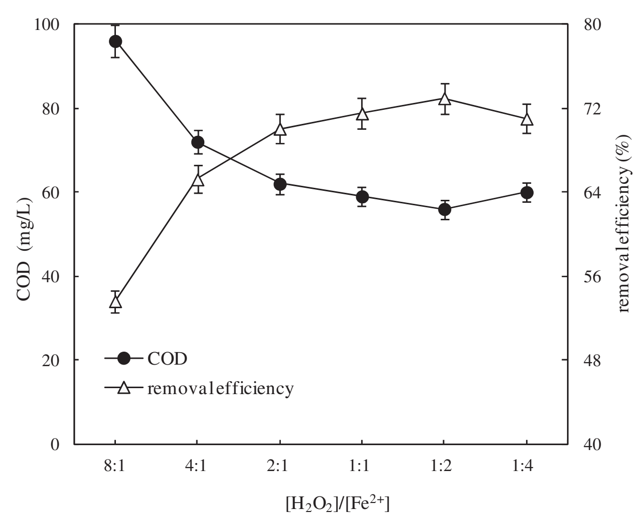

Fig. 5. Effect of $[H_2O_2]/[Fe^{2+}]$ molar ratio on COD removal of treated water in the Fenton process (conditions: pH 3; $[H_2O_2]$, 4 mmol/L; COD of raw wastewater, 207 mg/L).

evaluate the COD removal efficiency in the Fenton process. As shown in Fig. 5, the removal efficiency of COD significantly increased with increasing $[H_2O_2]/[Fe^{2+}]$ molar ratio from 8:1 to 1:1.Then COD removal efficiency slightly increased when the $[H_2O_2]/[Fe^{2+}]$ molar ratio was 1:2 and decreased when it was 1:4. The radical chain reactions in Fenton process might be terminated when more Fe^{2+} existed in the reaction system. This is because the $HO^{\cdot}$ was produced as a result of reaction (Eq. (1)) that mainly reacts with Fe^{2+} (Eq. (2)) [29,30]. Taking the operating costs and removal efficiency into account, the $[H_2O_2]/[Fe^{2+}]$ molar ratio of 1 was selected for the Fenton process.

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + HO^{\cdot} + OH^{-} \quad k_1 = 76\,M^{-1}\,s^{-1} \tag{1}$$

$$Fe^{2+} + HO^{\cdot} \rightarrow Fe^{3+} + OH^{-} \quad k_2 = 3.2 \times 10^8\,M^{-1}\,s^{-1} \tag{2}$$

3.2.3. Effect of H_2O_2 dosage

H_2O_2 plays a pivotal role as an oxidant in the Fenton process for generating the $^{\cdot}OH$. Fig. 6 presents the effect of H_2O_2 dosage on COD removal with a fixed $[H_2O_2]/[Fe^{2+}]$ molar ratio of 1 at pH 3. COD removal efficiency increased from 57 to 75% when the H_2O_2 dosage increased from 1 to 5 mmol/L. The maximum COD reduction was achieved at a H_2O_2 dosage of 5 mmol/L. A continuous increase in H_2O_2 dosage reduced the COD removal efficiency, which could be attributed to the scavenger effect of excess H_2O_2. The oxidation removal efficiency increased with increasing H_2O_2 dosage because of the increment of $^{\cdot}OH$ (Eq. (1)). The degradation rate decreased as the H_2O_2 dosage continued to increase beyond the suitable level. The scavenging effect of $HO^{\cdot}$ by $HO^{\cdot}$ and the consumption of H_2O_2 by $HO^{\cdot}$ were the important elements (Eqs. (3) and (4)) [19,30,31]. The COD removal efficiency reached 70% when the H_2O_2 dosage was 4 mmol/L. In this study, 4 mmol/L was selected as the suitable H_2O_2 dosage for the Fenton process.

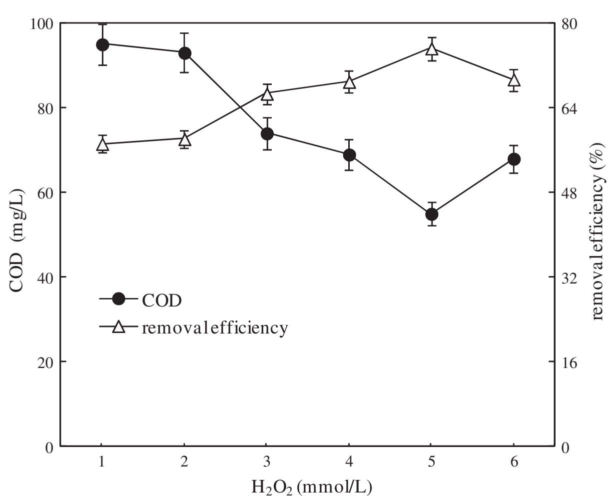

Fig. 6. Effect of H_2O_2 dosage on COD removal of treated water in the Fenton process (conditions: pH 3; $[H_2O_2]/[Fe^{2+}]$ = 1; COD of raw wastewater, 222 mg/L).

$$HO^{\cdot} + HO^{\cdot} \rightarrow H_2O_2 \quad k_3 = 5.2 \times 10^9\,M^{-1}\,s^{-1} \tag{3}$$

$$HO^{\cdot} + H_2O_2 \rightarrow HO_2^{\cdot} + H_2O \quad k_4 = 2.7 \times 10^7\,M^{-1}\,s^{-1} \tag{4}$$

3.3. Coagulation/Fenton process

Based on the optimization of the coagulation and Fenton processes, the initial papermaking wastewater was initially subjected to a coagulation process. Subsequently, the supernatant water was withdrawn and subjected to the Fenton process. Experimental parameters were controlled under individual selected conditions. The characteristics of the coagulation/Fenton process treated effluent and the overall treatment efficiency are summarized in Table 1. A total of 49% COD could be removed in coagulation process, whereas 73% COD was removed in the Fenton process at pH 3, at H_2O_2 dosage of 4 mmol/L and $[H_2O_2]/[Fe^{2+}]$ molar ratio of 1. Given the overall removal efficiency of COD (86%), the coagulation/Fenton process proved to be an effective advanced treatment for papermaking wastewater.

3.4. Water quality analysis

3.4.1. Polarity distribution

The polarity (HPO/HPI) distribution analysis is beneficial to make a suggestion for treatment method. Nguyen et al. [32] confirmed the biological treatment could be effective for the removal of HPO and readily biodegradable trace organics but was less effective for the removal of HPI and persistent compounds. Moreover, coagulation and adsorption contributed to the removal of HPO [33]. Fig. 7 shows the polarity distribution of dissolved organic compounds (DOC) in raw wastewater, coagulation-treated effluent, and Fenton-treated effluent. The HPO fraction was the largest (60%) DOC fraction in raw papermaking wastewater. Although the characteristics of

Table 1
Overall treatment efficiency of the coagulation/Fenton process

Parameter	Raw wastewater	Coagulation treated effluent	Removal efficiency (%)	Fenton treated effluent	Removal efficiency (%)	Overall efficiency (%)
COD (mg/L)	244	124	49.2	33	73.4	86.5

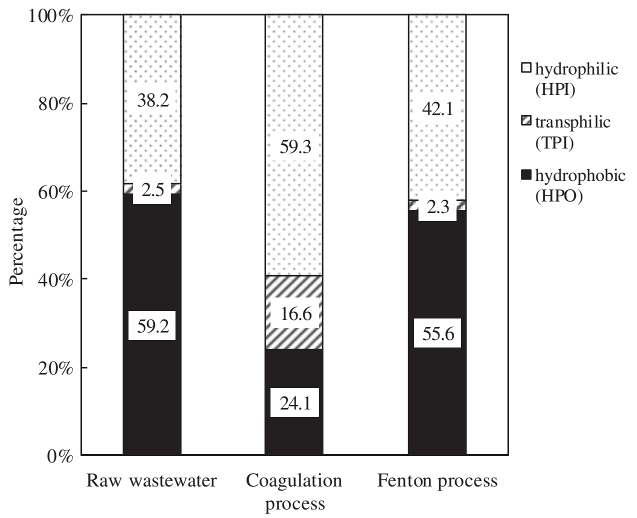

Fig. 7. Polarity distribution of DOC in raw wastewater, the coagulation process effluent, and the Fenton process effluent.

papermaking wastewater vary in a wide range, lignin which exists in the form of colloidal particles is the main non-degradable organic pollutant in wastewater. Lignin is relatively HPO and aromatic with a complex chemical structure [34].

Fig. 7 shows that the HPO fraction of DOC was effectively reduced from 59.2 to 24.1% in the coagulation process. The coagulation process efficiently removed the HPO fraction in the papermaking wastewater, thereby achieving a removal efficiency of 48.3%. Based on the charge neutralization and sweep flocculation mechanism, PAC provides a positively charged ion and lignin provides the opposite charge. At low pH level, the wastewater contained lignin coordinates with PAC and from insoluble metal complexes. At high pH level, the organic compounds adsorbed onto the pre-formed flocs of metal hydroxides and get precipitated. These results demonstrate that it is efficient to remove lignin from wastewater in the coagulation process [18]. By contrast, Fenton process was a non-selective reaction on degrading organic compounds with generating $HO^{\cdot}$ from Fig. 7.

3.4.2. MW distribution

The distribution and transformation of the MW of organic matter was further studied to evaluate the efficiency of the coagulation/Fenton process in papermaking wastewater treatment. As shown in Fig. 8, the majority of the organic matter (70%) in raw wastewater was high MW organic compounds (MW > 10 kDa). These organic compounds could not be degraded in the biological treatment process. Coagulation process remarkably reduced the amount of high MW organic matter that contributes to wastewater COD. The molecules with MWs ranging from 0 to 1 kDa comprised 61.9% of the total organic compounds in the coagulation process effluent, but only 2.7% of the organic compounds in raw wastewater.

Likewise, the Fenton effluent showed a similar transformation. The majority of the molecules with MWs of 10–30 kDa, and over 30 kDa were mainly transformed into 0–1 kDa molecules (comprising of 88.0% of the total molecules) in the Fenton process effluent. On the other hand, the low MW molecules (0–1 kDa) in the Fenton process effluent were more abundant than those in the coagulation process effluent from Fig. 8. Therefore, the coagulation/Fenton

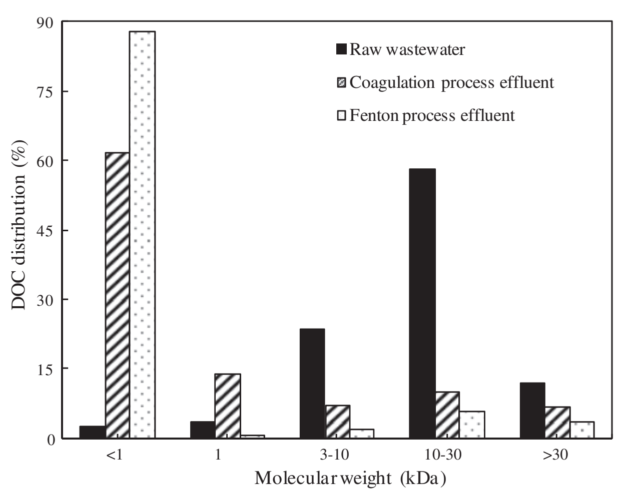

Fig. 8. Molecular weight distribution of DOC in raw wastewater, the coagulation process effluent, and the Fenton process effluent.

Table 2
Consumption of the reagents and their costs

Treatment technology	Dosage (kg/m^3)						Operating cost (RMB/m^3)
	Spent acid	PAC	PAM	NaOH	$FeSO_4 \cdot 7H_2O$	H_2O_2	
Fenton process	0.01	0	0.001	0.04	1.236	0.989	1.39
Coagulation/Fenton process	0.12	0.12	0.002	0.04	0.618	0.247	0.78

Note: Unit price of reagent (RMB/t): PAC, 440; PAM, 12540; NaOH, 2000; $FeSO_4 \cdot 7H_2O$ (99.0 wt.% of purity), 240; H_2O_2 (30%, w/w), 1000; spent acid, 100.

process could increase the biodegradability of wastewater [12]. As indicated by the differences in the organic matter MW transformation between the two processes, the retention and degradation performance of the coagulation and Fenton processes improved the effluent water quality of the papermaking wastewater.

3.5. Economic analysis

An efficient and cost-effective treatment process must strike a balance between treatment efficiency and operating costs. The consumption of the chemical reagents in the treatment technology of coagulation/Fenton and classic Fenton alone are shown in Table 2. The cost of the treatment of classic Fenton process alone was about 1.39 RMB/m^3, while in the case of the coagulation/Fenton process, the cost was only 0.78 RMB/m^3 when met with the same COD discharge requirement (<60 mg/L). In general, the treatment costs of the advanced treatment by classic Fenton process for papermaking wastewater were 1.19–1.8 RMB/m^3 yielding an 80% of COD removal in engineering practice [35]. From the perspective of economy, the coagulation process reduced the cost of H_2O_2 in the subsequent Fenton process, thereby resulting in lower treatment cost. It was indicated that the coagulation/Fenton process was more economical for the degradation of papermaking wastewater. Then the coagulation/Fenton process can be potentially used in industrial applications.

4. Conclusions

The coagulation/Fenton process proved to be an efficient and economical advanced treatment method for papermaking wastewater. By the coagulation process, 49% of COD could be reduced under optimal conditions, as follows: pH of 5, the PAC loading of 120 mg/L, and PAM loading of 1 mg/L. When the dosage of H_2O_2 was 4 mmol/L and the $[H_2O_2]/[Fe^{2+}]$ molar ratio was 1, the maximum COD reduction (73%) was obtained via the Fenton process at pH 3. The polarity distribution analysis results indicated that the coagulation process could efficiently remove the HPO fraction, which comprised the largest fraction of DOC (60%) in raw wastewater. Experiments on MW distribution showed that the high MW molecules in DOC were transformed into low MW molecules via the coagulation/Fenton process. The costs of the coagulation/Fenton process amounted to 0.78 RMB/m^3. Therefore, the coagulation/Fenton process may be a suitable advanced treatment method for papermaking wastewater.

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