

Treatment of jean-wash wastewater by combined coagulation, hydrolysis/acidification and Fenton oxidation

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Abstract

Performance of a full-scale combined treatment plant for jean-wash wastewater (JWW) was investigated. The combined process consisted of chemical coagulation, hydrolysis/acidification and Fenton oxidation. Chemical coagulation treatment with polymeric ferric sulfate (PFS)/lime alone proved to be effective in removing the COD (>70%) and part of the color (>50%) from the JWW. Fenton oxidation combined with hydrolysis/acidification as pretreatment offered a noticeable BOD removal efficiency. The average removal efficiencies for COD, BOD, SS, color and aromatic compounds of the combined process were about 95%, 94%, 97%, 95% and 90%, respectively, with the average effluent quality of COD 58 mg/L, BOD 19 mg/L, SS 4 mg/L and color 15(multiple), consistent with the national discharge limits for textile wastewater. The result indicated that the combined procedure could offer an attractive solution for JWW treatment with considerable synergistic advantages.

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Keywords: Jean-wash wastewater; Chemical coagulation; Hydrolysis/acidification; Fenton oxidation

1. Introduction

Jean-wash wastewater (JWW) is derived from jean clothes wash processes with various types of wash techniques such as Antique, Black, Wash, Canyon, Crinkle, Denim, Destroyed, Rinse, Sandblasted, Stone, etc. The materials employed in jean-wash processes cover glacial acetic acid, ferment powder, soda, phosphate, film, pumice, potassium permanganate, coloring stabilizer, whitening agent and various types of dyes, etc. JWW can be classified as a kind of textile wastewater, and exhibits strong color, a large amount of suspended solids, dissolved salts, highly fluctuating pH and high COD concentration with poor biodegradability, although the types and concentration of pollutants in JWW differ acutely as wash techniques changes.

Textile industry produces large volumes of effluents that contain appreciable quantities of organic compounds that are not easily amenable to chemical or biological treatment [1–14].

The non-biodegradability of textile wastewater is due to a high content of dyestuffs, surfactants and additives which generally are organic compounds of complex structures. Important pollutants in textile effluent are mainly recalcitrant organics, color, toxicants and inhibitory compounds, surfactants, chlorinated compounds, pH and salts [12]. Many attempts have been made to treat textile wastewater using conventional wastewater treatment methods such as chemical coagulation, electrochemical oxidation, filtration and biological treatment [1–14], most of which are incapable of adequately treating the wastewater when applied alone [1]. Although effective for color and COD removal, chemical coagulation process exhausts a large amount of flocculent reagent and can generate a large amount of sludge [7]. Biological treatment processes are generally efficient for biochemical oxygen demand (BOD) and suspended solids (SS) removal, but they are largely ineffective for removing color from the wastewater. The recalcitrant nature of various dyes, together with their toxicity to microorganisms, makes aerobic treatment difficult [12]. Chemical oxidations by ozone or Fenton oxidation are of great interest but their costs are very high to treat raw textile wastewater [1]. Thus, the use of combined processes has been suggested to overcome the disadvantages of individual unit processes [9–10]. Due to the complexity of the JWW, any single

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Table 1
Characteristics of JWW

	Maximum	Minimum	Mean
COD (mg/L)	2500	430	1200
BOD ₅ (mg/L)	575	112	330
SS (mg/L)	287	37	138
pH	7.9	5.9	6.9
Color (multiple)	1000	50	300
Cl (mg/L)	750	102	133
Nitrate (mg/L)	62	0	57
Sulfate (mg/L)	210	86	107
Conductivity (μS/cm)	3050	810	880

treatment method would be inadequate, and to date literature regarding JWW treatment has been scarce.

It is well known that hydrolysis/acidification process can improve the biodegradability of the wastewater and reduce the treatment cost without aeration. Chemical oxidation using Fenton reagent, a mixture of Fe²⁺ salts with hydrogen peroxide H₂O₂ [15], has been found quite effective in dealing with various types of industrial wastewaters [15–21], and appeared to be the most promising method, in terms of cost-effectiveness and ease of operation [16]. Fenton oxidation was intended to destroy the recalcitrant organic chemicals in the wastewater with an additional aim to enhance their biodegradability [19]. In the present work, a combined process consisting of chemical coagulation, hydrolysis/acidification and Fenton oxidation is proposed for JWW treatment.

2. Materials and methods

2.1. Wastewater

The wastewater was obtained from a jean-wash industrial district (Zhuzhou, Hunan, southern China). There were 11 small mills in the jean-wash industrial district, and all the effluent from the mills is collected to a wastewater treatment plant. The mills had intermittent and fluctuating wastewater flow with variable wastewater composition depending on various final products

and production regimes. Acute variability of effluent amount, extraordinary complexity of effluent quality, high concentration of refractory pollutants and poor biodegradability characterized the wastewater. The sample raw wastewater was drawn from the equalization tank, firstly filtered by screen filters to remove large suspended solids before the wastewater was used for the subsequent experiment. The characteristics of the wastewater are depicted in Table 1. The national discharge limits require that COD and BOD of the wastewater should be reduced down to 100 mg/L and 30 mg/L, respectively in order to discharge to a receiving water body.

2.2. System description

Fig. 1 shows the scheme of the full-scale JWW treatment system with capacity of 2700 t/d. After being pretreated by screen filters, the wastewater was homogenized in an equalization tank. Injected with chemical coagulant into the pipe ahead of the feed pump, the wastewater was lifted to an inclined-board sedimentation tank, then the sludge was pumped into sludge thickener, and the supernatant flowed into hydrolysis/acidification tank. The effluent then was oxygenated by Fenton oxidation and filtered by sand filter. The main functions and design parameters for each unit were described as following:

- *Screen filter*: There exists in JWW a large amount of suspended solids such as pumice and textile residues, which should be removed by screen filter. Two screens, with aperture 2.0 mm and 0.5 mm, respectively, were used.
- *Equalization tank*: The equalization tank has dimensions of 16 m × 14 m × 4.2 m, holding approximately 710 m³ of working volume and 6.2 h of average hydraulic retention time (HRT).
- *Sedimentation tank*: Together with injection of coagulant into the feed pipe ahead of a wastewater-feed pump (where the coagulant were well mixed), the wastewater from equalization tank was lifted by the pump to an inclined-board sedimentation tank, where the solid precipitates were separated from the aqueous phase. The sedimentation tank has dimensions of

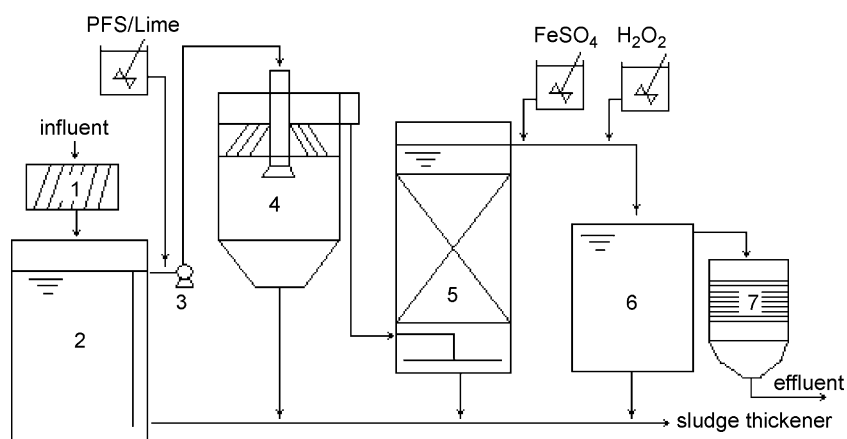


Fig. 1. Flow diagram of the JWW treatment plant. (1) Screen filter, (2) equalization tank, (3) wastewater-feed pump, (4) sedimentation tank, (5) hydrolyzation tank, (6) oxidation tank, (7) sand filter.

8.5 m × 3.5 m × 5.5 m, with surface burden of 1.61 m³/(m² h) in sedimentation area and average HRT of 1.4 h. The coagulant used was polymeric ferric sulfate (PFS), and the pH value of the wastewater was regulated in the range 9.0 ± 0.5 by using lime in the form of slurry (20 g/L) [5].

- **Hydrolysis/acidification tank:** There were two hydrolysis/acidification tanks, each having dimensions of 8 m × 8 m × 6.5 m, with average HRT of about 7.2 h. The wastewater was distributed by branch-conduits installed at the bottom of the tank. Hanging elastic stuffing materials, with strand silk diameter 0.35 mm and specific surface area 200 m²/m³, were filled as microorganism carriers in the tank.
- **Oxidation tank:** The oxidation tank has dimensions of 16 m × 7 m × 5.5 m, with average HRT of about 4.1 h. The effluent from hydrolysis/acidification tank flowed into the oxidation tank with ferrous sulfate and hydrogen peroxide introduced through metering pump individually. Concurrently the oxidation tank has functions of sedimentation and sludge exclusion.
- **Sand filter:** The effluent from oxidation tank was polished by sand filter tank stuffed with quartz sand and/or sintered aggregate. The sand filter has dimensions of 16 m × 6 m × 3.5 m.

2.3. Materials

Polymeric ferrous sulfate (PFS), polymeric aluminum sulfate (PAS), hydrated ferrous sulfate (FeSO₄·7H₂O), hydrogen peroxide (H₂O₂) and lime (Ca(OH)₂) of commercial grade were utilized during the experiments. H₂O₂ solution of 30% was used. Lime was prepared in the form of slurry (20 g/L). PFS, PAS and ferrous sulfate were also prepared in solutions.

2.4. Analytical methods

The performance of the combined system was monitored by analyzing the parameters of COD, BOD₅, SS, color and pH. All the samples were collected intermittently and analyzed during experiments for almost 1 year.

COD was measured using COD571 meter (Shanghai Huayan, China). BOD₅ was determined with BOD870 (Jianshu Jiangyan, China). SS was measured using gravimetric method. pH was measured with digital acidimeter PHS-3C (Shanghai REX, China). The color of wastewater was determined with standard dilution multiple method [22]. The UV–vis spectrum was taken by TU-1901 spectrophotometer (Beijing Purkinje, China). Conductivity was determined by DDS-307 digital conductivity meter (Shanghai Tianda, China). Chloride, nitrate and sulfate were measured by PIC-10 ionic chromatogram meter (Qingdao Puren, China).

3. Results and discussions

3.1. Coagulation

PFS and PAS of industrial grade coagulants were applied to test the capability for JWW coagulation on a laboratory scale.

The results indicated that PFS appeared to be the appropriate choice with the highest COD/color removal efficiency and the least coagulants consumption (data not shown). Jar-test indicated that PFS has the highest coagulation efficiency when used to treat JWW at pH 9.0; therefore, experiments were run during which PFS was applied at an increasing concentration while the pH was maintained constant (9.0 ± 0.5) by using lime slurry [5]. As Fig. 2 presents, a PFS concentration of 150 mg/L was necessary to attain removal efficiency above 70% and 50% for COD and color, respectively. Thus PFS concentration of 150 mg/L was applied in the full-scale JWW treatment plant for economic reasons. After coagulation and sedimentation, the COD, BOD, SS and color of the effluent achieve 351 mg/L, 149 mg/L, 55 mg/L and 150 (multiple), respectively, with corresponding removal efficiency of 71%, 56%, 62% and 50%, respectively.

3.2. Hydrolysis/acidification

Because the quality of effluent from coagulation stage was still far from the required standard for discharge, and the pollutants in the effluent are generally refractory compounds which are very difficult to deal with by ordinary activated sludge method [1], it is necessary to apply advanced oxidation for further treatment. Of the advanced oxidation processes, Fenton oxidation appeared to be the most potential and economical way for textile treatment. However, despite the high efficiency, the process is limited by the acidic pH required and the high amount of sludge in the coagulation step [2]. It is thus expected that anaerobic hydrolysis/acidification process would be an appropriate pretreatment for Fenton oxidation.

Controlling the anaerobic treatment in first stage of the anaerobic treatment, which needs short reaction time, anaerobic hydrolysis/acidification process is known as an effective pretreatment for aerobic treatment of refractory wastewater [23–25]. It was reported that the process has following advantages: (1) the refractory organic substance and SS are greatly reduced, hydrolyzed and acidified, and the sludge produced in subsequent process is largely decreased; (2) the biodegradability of the wastewater and the degrading rate of refractory pollutants

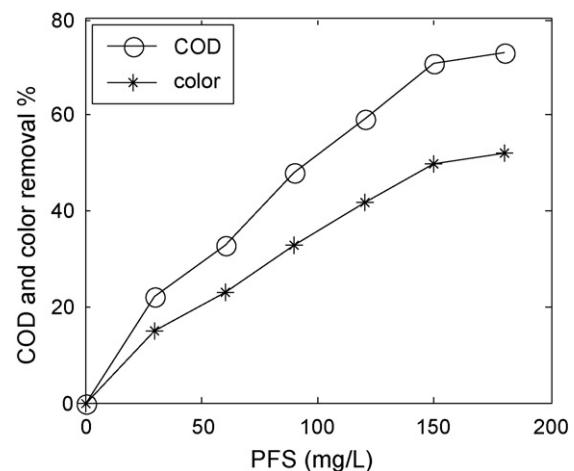


Fig. 2. COD and color removal efficiency as a function of PFS concentration.

can be improved; (3) concurrently acts as a buffer for the influent load fluctuation, thus resulting in a relative stable inflow quality for subsequent process; (4) greatly reduce the operating cost, construction cost and facility area; (5) could destroy the chromophoric groups of the dyestuff molecule, and remove its chroma effectively for dyeing wastewater with high chroma [24]. The hydrolysis/acidification process in the present work was expected to depress pH value of the effluent from coagulation, to reduce partially the refractory substance in the wastewater, and to destroy the chromophoric groups of the dyestuff molecule in JWW, thus to offer beneficial conditions for the subsequent Fenton oxidation and to reduce Fenton reagent dosage and sludge generation.

At the initial stage of the process the JWW sludge in nearby ditches was introduced into the hydrolysis/acidification pond for microorganism inoculation and acclimation, and the necessary N, P nutrients were introduced into the wastewater for microorganism propagation. After continuous operation for about a month, biofilm was formed on the stuffing materials and the treatment efficiency tended to “steady-state”, and pH value of the effluent depressed from 6–8 down to 5–6, together with about 30% COD and 50% color removal efficiency, respectively.

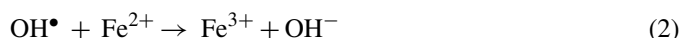
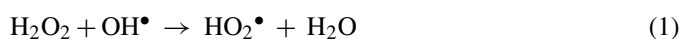
3.3. Fenton oxidation

Fenton oxidation is one of the best-known metal catalyzed oxidation reactions of water-miscible organic compounds. The mixture of FeSO_4 or any other ferrous complex and H_2O_2 (Fenton's reagent) at low enough pH, results in Fe^{2+} catalytic decomposition of H_2O_2 and proceeds via a free radical chain process that produces hydroxyl radicals which have extremely high oxidizing ability and can oxidize hard-to-decompose organic compounds in a short time. The Fenton's reagent has not only oxidation function but also coagulation by the formation of ferric-hydroxo complexes. At pH values higher than 4.0, ferrous ions easily form ferric ions, which have a tendency to produce ferric hydroxo complexes working as coagulants. The coagulation step acts as a polishing step, and removes the remaining after Fenton oxidation [11].

The treatment efficiency of Fenton oxidation is highly pH dependent [10,26]. It is believed that the pH value has to be in the acidic range to generate the maximum amount of hydroxyl radicals to oxidize organic compounds. At high pH ($\text{pH} > 4$), the generation of hydroxyl radicals gets slower because of the formation of the ferric-hydroxo complexes [11]. Therefore, the initial pH value has to be between 2 and 4 to generate the maximum amount of hydroxyl radicals to oxidize organic compounds [2–4,6–14]. However, Tekin et al. reported that with the initial pH range 3.0–4.5 no significant differences in treatment efficiency were observed [20]. Kang and Chang [6] reported that for dyeing wastewaters, the removal efficiency of COD decreases with increasing pH values and the pH range for maximum removal of both COD and color is 3–5. Lin and Jiang [19] reported that the results of Fenton oxidation on semiconductor wastewater were very good between pH 2 and 5. Thus, Fenton oxidation appears still appropriate at pH 5.

The efficiency of the Fenton oxidation process depends also on the organic matter content, temperature, Fe^{2+} : H_2O_2 ratio and H_2O_2 concentration are also crucial [17]. Depending on the effluent being treated, different operating conditions should therefore be employed to achieve high degradation efficiencies. The general approach in the treatability studies was to optimize the operational conditions in the Fenton oxidation unit to achieve maximum treatment efficiency while minimizing the use of chemicals (acid and base for pH adjustment, and hydrogen peroxide and ferric sulphate as the Fenton reagents) and hence minimizing operation costs of the treatment plant [20]. To investigate the treatability and the optimal operational conditions for JWW effluent from the hydrolysis/acidification process using Fenton reagent, two sets of experiments were performed on laboratory scale to independently study the effect of initial Fe^{2+} and H_2O_2 concentrations at pH 5.0 and room temperature for practical and economic reasons.

The effect of FeSO_4 concentration on COD removal efficiency is given in Fig. 3. According to Fig. 3, maximum COD removal (60%) was obtained in the case of using 100 mg/L of Fe^{2+} (1.786 mM), 100 mg/L of H_2O_2 (2.941 mM), and pH at 5.0. However, color removal was mediocre, usually about 40%. Although at Fe^{2+} concentration of 80 mg/L, the highest color removal efficiency was reached; the COD removal efficiency achieved its summit at 100 mg/L concentration of Fe^{2+} , then 100 mg/L concentration of Fe^{2+} was chosen as optimum concentration. However, as seen from Fig. 3, the COD and color removal efficiency got hardly significant improvement and even started to decrease for higher doses of FeSO_4 . It may be explained by redox reactions since OH radicals may be scavenged by the reaction with the hydrogen peroxide present or with another Fe^{2+} molecule as below [27]:



The Fe^{3+} formed can react with H_2O_2 as well as with hydroperoxyl radicals with regeneration of Fe^{2+} in the solution resulting in decrease in COD/color removal. Hence the plateau

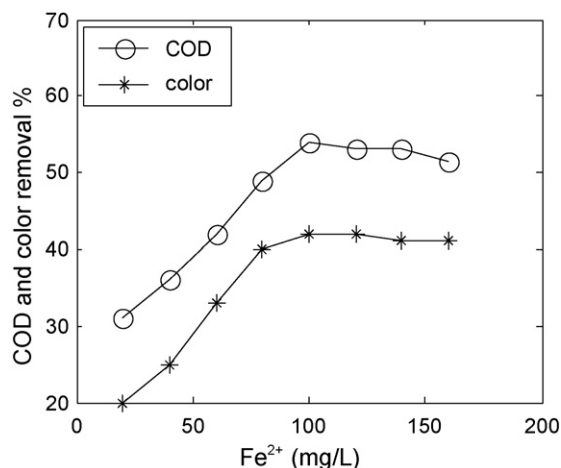


Fig. 3. Effect of Fe^{2+} concentration on COD and Color removal using 100 mg/L H_2O_2 at pH 5.0 and ambient temperature.

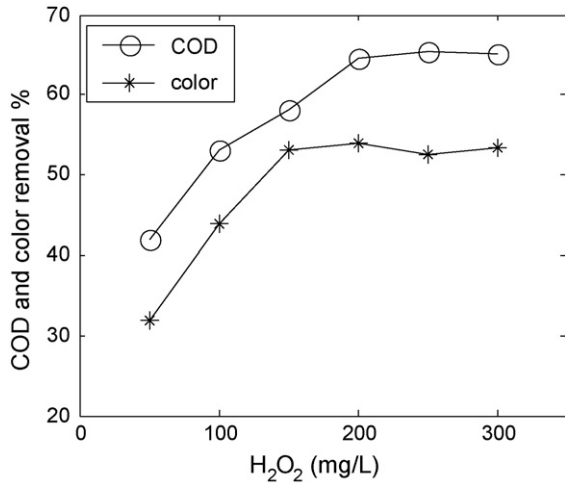


Fig. 4. Effect of H₂O₂ concentration on COD and color removal using 100 mg/L Fe²⁺ at pH 5.0 and ambient temperature.

or decrease in COD/color removal after 100 mg/L of Fe²⁺ might be explained due to the scavenging effect of over doses of Fe²⁺ on OH radicals. Meric et al. [11] had also found the phenomena while Fenton oxidation process was used for dyeing wastewater treatment [11,27].

The results of the color removal at 100 mg/L concentration of Fe²⁺ for determination of optimum H₂O₂ concentration are illustrated in Fig. 4. As shown in Fig. 4, COD and color removal efficiency increased by increasing H₂O₂ concentration from 50 mg/L to 300 mg/L. However, at concentrations of 150 mg/L and 200 mg/L or higher a plateau was reached for color and COD removal, respectively. Thus, 200 mg/L (5.882 mM) of H₂O₂ was defined as optimum concentration providing 52% color and 65% COD removals as seen in Fig. 4. Increasing the concentration of H₂O₂ did not improve COD and color removal, more in turn it seems to decrease because of scavenging effect of H₂O₂ on •OH radicals [27]. Although the pollutant reduction efficiency appears a little lower than that of some literatures reported [3,4,7,11] from the efficiency point of view, the COD and color of the effluent from Fenton oxidation achieved 78 mg/L and 24 (multiple), respectively, which

already satisfy the national discharge limits for textile wastewater.

The Fe²⁺:H₂O₂ ratio in Fenton oxidation differs for various wastewater treated. Fongsatitkul et al. [3] suggested that the FeSO₄·7H₂O and H₂O₂ molecular ratio for textile wastewater be 1:1, i.e., the Fe²⁺: H₂O₂ ratio by weight be about 1:1.65. Guedes et al. [17] reported that the optimum Fe²⁺: H₂O₂ ratio by weight is 1:2. For the optimum concentration 100 mg/L of Fe²⁺ and 200 mg/L (5.882 mM) of H₂O₂ for JWW treatment, the Fe²⁺: H₂O₂ ratio (by weight) is 1:2, which is approximately consistent with these results.

Based on the results above, it therefore seems that, although the relative high pH value (pH 5.0) of effluent from hydrolysis/acidification process might have a little negative impact on the treatment efficiency of the subsequent Fenton oxidation, the combination of hydrolysis/acidification and Fenton oxidation remains appropriate for JWW treatment, because the cost-free hydrolysis/acidification process reduced largely the refractory pollutants in the wastewater and the dosage of Fenton reagent, and avoided the trouble and cost for pH regulation.

3.4. Combined system

The combined system (Fig. 1) had been run continuously for 1 year under following conditions (1) coagulation: PFS 150 mg/L, together with HRT 1.4 h; (2) hydrolysis/acidification: HRT 7.2 h; (3) Fenton oxidation: Fe²⁺ 100 mg/L, H₂O₂ 200 mg/L, with HRT 4.1 h.

Fig. 5 shows the average results of the pollutants reductions from each process unit and the whole combined system. It can be seen that after the combined procedure of chemical coagulation, hydrolysis/acidification and Fenton oxidation, the COD, BOD₅, SS and color in JWW was reduced to 78 mg/L, 28 mg/L, 11 mg/L and 24 (multiple), respectively, satisfying the national discharge limits for textile wastewater.

The use of sand filter as the final polishing process was based on the consideration that the jean-wash mills hoped to reuse the treated wastewater in their jean-wash productions, because the water quality demand is not very rigid in some production stages such as rinsing process which exhausts a great deal of

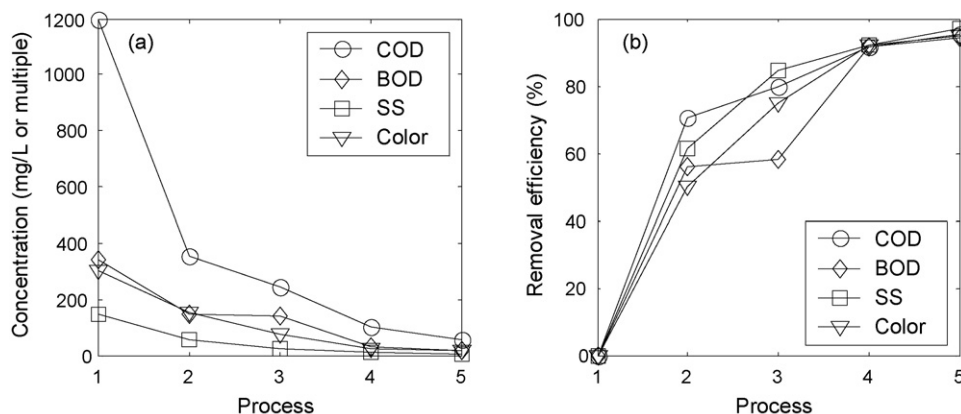


Fig. 5. Pollutant reduction of raw wastewater (1) upon chemical coagulation (2), hydrolysis/acidification (3), Fenton oxidation (4) and sand filter (5). (a) Pollutant concentration, (b) accumulative removal efficiency.

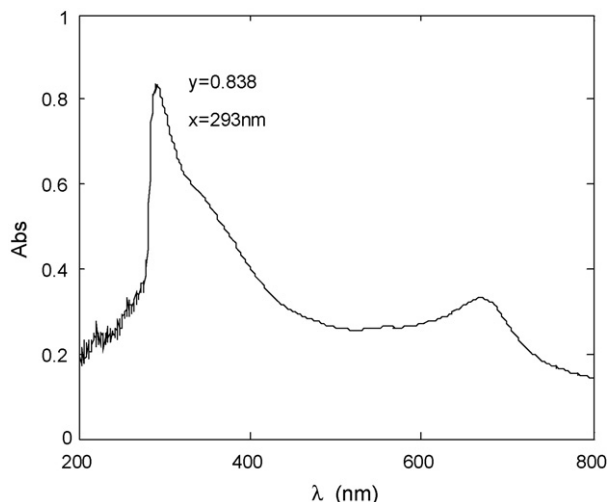


Fig. 6. The UV-vis spectrum of the original JWW.

water. With the combination of sand filter, COD, BOD₅, SS and color of the whole system effluent reached 58 mg/L, 19 mg/L, 4 mg/L and 15 (multiple), respectively, with removal efficiency of 95%, 94%, 97% and 95%, respectively.

It is noticeable that although the hydrolysis/acidification process attained COD, color and SS removal efficiency of 30%–60%, BOD concentration of the effluent (149 mg/L) remained almost the same as that of the influent (142 mg/L). However, the subsequent Fenton oxidation reduced BOD acutely (from 142 mg/L to 28 mg/L), further demonstrating the feasibility of combination of hydrolysis/acidification and Fenton oxidation processes.

Figs. 6 and 7 show the UV-vis spectrums of the original and treated JWW, respectively. It can be seen that there is an absorbing peak at about 290 nm, indicating π - π^* electron transition on benzene ring [28]. The absorbance of the original and treated JWW is 0.838 and 0.0843, respectively, indicating the aromatic compound removal efficiency of the combined process is about 90%.

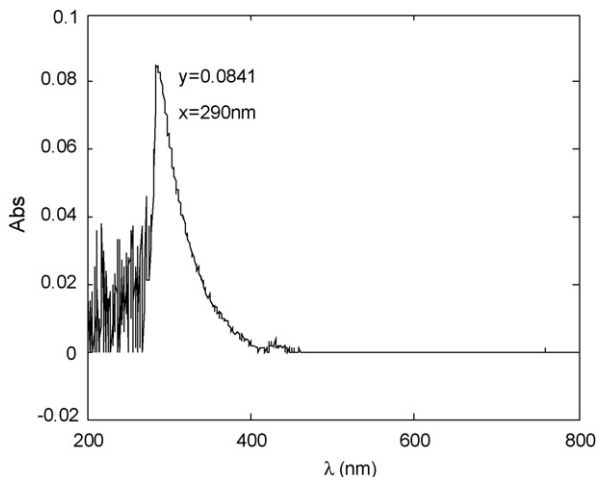


Fig. 7. The UV-vis spectrum of the treated JWW.

4. Conclusion

A combined JWW treatment system consisting of chemical coagulation, hydrolysis/acidification and Fenton oxidation on a full-scale of 2700 t/d was studied and the performance of the system was measured for more than 1 year. The results showed that the average removal efficiencies of COD, BOD, SS and color were about 95%, 94%, 97% and 95%, respectively, with the average effluent quality of COD 58 mg/L, BOD 19 mg/L, SS 4 mg/L and color 15 (multiple). The effluent quality was better than the requirement of the standards for textile wastewater discharge in China. Thus the combined process can serve as an attractive alternative solution for JWW treatment.

It is believed that anaerobic hydrolysis/acidification process is generally used as pretreatment of subsequent aerobic biological treatment to improve the biodegradability of refractory wastewater. The results of the present work suggest that the hydrolysis/acidification process could also be employed as pretreatment for Fenton oxidation. However, although the performance appears satisfactory, further researches are in great demand on the mechanisms of the considerable synergistic effect on pollutants removal.

Acknowledgments

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