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Enhancement of Rhodamine B removal by low-cost fly ash sorption with Fenton pre-oxidation

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ABSTRACT

The removal of a basic dye, Rhodamine B (RhB), by fly ash adsorption, Fenton oxidation, and combined Fenton oxidation-fly ash adsorption were evaluated. Even though fly ash is a low cost absorbent, a high dose of fly ash was needed to remove RhB. Only 54% of RhB was removed by 80 g L^{-1} fly ash. Solution pH did not significantly affect the RhB sorption by fly ash after 8 h. Fenton reagents at H_2O_2 dose of 6×10^{-3} M and pH 3 rapidly decolorized 97% of RhB within 2 min, and 72% of COD removal was obtained at 30 min reaction time. Spectrum analysis result showed that a large area of UV spectrum at 200–400 nm remained after Fenton reaction. The addition of 1 g L^{-1} fly ash effectively removed COD from Fenton-treated solution, and the UV absorption spectrum at 220–400 nm totally vanished within 2 h. COD removal of RhB by the combined Fenton oxidation and fly ash sorption process was 98%. The COD removal capacity of fly ash for Fenton-treated RhB solution was 41.6 times higher than that for untreated RhB solution. The results indicated that the combined process is a potential technique for RhB removal.

1. Introduction

Textile wastewater is a major water pollutant source in developing countries. It often contains high concentrations of un-fixed dyes (about 20%). Dyes are of great concern because of their widespread use, bio-recalcitrance, and toxic aromatic intermediates [1]. Several techniques have been employed to remove dyes from wastewater, including adsorption, chemical oxidation, electrochemical degradation, and AOP [2]. However, their low removal abilities or high costs often limit their application. For example, granular activated carbon can effectively adsorb various classes of dyes; however, relatively high operating costs of GAC and difficulty to regenerate often limit its large scale application.

Many absorbents have been used to remove dyes from wastewater [3]. Among them, fly ash has the advantages of low-cost and ample availability from coal thermal plants. The sorption of dyes onto fly ash has been evaluated in several studies [3–5]. The factors influencing dye sorption onto fly ash include nature of dyes, origin of fly ash, solution pH, and temperature [3]. On the other hand, fly ash has some drawbacks, including its low sorption capacity for some types of dyes. Therefore, a high dose of fly ash is needed for dye sorption, leading to a sludge disposal problem. It is important to increase the sorption capacity of fly ash when it is applied for dye removal. Wang et al. [6] have employed different methods, including conventional chemical, sonochemical, and microwave, to improve the sorption ability of fly ash. However, studies on changing the properties of dyes through AOP-oxidation to increase fly ash sorption capacity of dye are limited.

Fenton oxidation is a practical and relatively inexpensive technique in comparison with other AOPs. Fenton oxidation uses a mixture of H_2O_2 and Fe^{2+} at acidic condition to generate hydroxyl radicals for dye degradation. Several studies have reported that the optimal molar ratio of H_2O_2:Fe^{2+} for dye oxidation by Fenton reaction was 10–20 [7–9]. To achieve the high dye removal efficiency of dyes, a high dose of Fenton reagents is often required and dye intermediates are often present in the treated solution [10]. Combined AOP pre-oxidation with biological treatment for dye removal have been evaluated to reduce the applied doses of Fenton oxidation [11,12]. There are only a few studies on dye removal by the combined Fenton oxidation-fly ash sorption process.

Rhodamine B (RhB, a basic reactive zanthene dye) was selected as the model dye. RhB is widely used in textile industries for dyeing silk, wool, jute, leather, and cotton. Human exposure to RhB may cause harmful health effects [13]. The aims of this study were (1) to compare the removal of RhB by Fenton oxidation, fly ash sorption , and combined Fenton oxidation-fly ash sorption and (2) to investigate influences of Fenton reagent doses on COD removal from Fenton-treated solution by fly ash.

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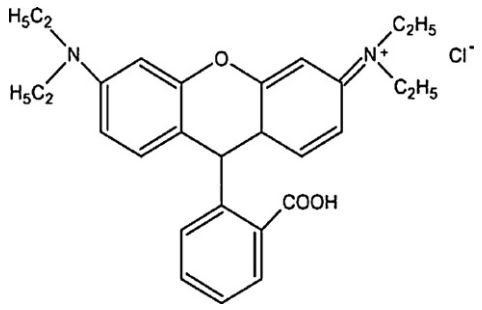

Fig. 1. Molecular structure of Rhodamine B (RhB, Basic violet 10), λ_{max} = 554 nm.

2. Materials and methods

2.1. Dye and fly ash

Rhodamine B (RhB, Basic violet 10, C.I. No. 45170, $C_{28}H_{31}N_2O_3Cl$, MW = 359.9 g mol^{-1}, 95% purity) was obtained from Sigma–Aldrich (US) and was used as received. The molecular structure of the dye is shown in Fig. 1. The fly ash was obtained from a thermal power plant in Kaohsiung, Taiwan. Fly ash was dried at ambient temperature for a week, passed through a 0.25 mm sieve, and stored in a plastic bag for further tests. The pH of the fly ash was measured as follows: 1 g of fly ash was mixed with 10 ml of distilled-deionized water for 24 h, and the mixture was measured by pH meter (Cyberscan 510, Taiwan). The pH of fly ash was found to be 12.7. Major oxides of fly ash were determined by microwave digestion and analysed by ICP-MS. The chemical compositions of the fly ash were SiO_2 (44.9%), Al_2O_3 (15.4%), Fe_2O_3 (8.4%), CaO (16.6%), and MgO (1.8%). The surface area of fly ash was measured by BET (Brunauer–Emmett–Teller-method). BET surface area was measured at 77 K on a PMI's BET Sorptometer (BET-201-AEL). N_2 adsorption–desorption on the surface was used to determine textural properties. The specific surface area of fly ash was 8.9 m^2 g^{-1}. The loss on ignition was 0.19%.

2.2. Sorption experiments

For batch sorption experiment, a known amount of fly ash was added into a 300 ml flask containing 200 ml of RhB solution (100 mg L^{-1}). The solution pH was not adjusted. The flask was sealed with parafilm and shaken in a water bath (20 °C, 50 rpm). At pre-set time intervals, 3 ml samples were withdrawn and centrifuged at 12,000 rpm for 10 min. The pH value and RhB concentration was measured. The RhB concentration was determined based on the constructed calibration curves at absorption wavelength of 554 nm. The UV–vis spectrum during the dye degradation was measured at 200–800 nm using a UV–vis spectrophotometer (Shimadzu, UV-mini 1240, Japan). The sample was diluted with distilled water when the absorbance exceeded the range of calibration curve. COD was determined according to standard method for examination of water and wastewater [14].

2.3. Fenton oxidation

H_2O_2 (30% purity) was obtained from Hayashi Pure Chemical Ind. Ltd. (Japan) and $FeSO_4$ ($FeSO_4{\cdot}7H_2O$, >99% purity) was purchased from Showa (Japan). Appropriate amounts of stock dye solution and ferrous ion were added to a 300 ml beaker and diluted with distilled-deionized water to 100 ml. To obtain desired pH, the solution was adjusted using diluted H_2SO_4 and NaOH solutions and measured by pH meter (Cyberscan 510, Taiwan). H_2O_2 was then added to initiate the Fenton reaction. The H_2O_2:Fe^{2+} molar concentration ratio was kept at 10:1. The initial solution pH was 3. All experiments were conducted at room temperature (20 ± 2 °C). Samples were withdrawn at specific time intervals during the Fenton oxidation. Each sample was supplemented with concentrated NaOH solution to quench the Fenton reaction. The samples were centrifuged at 12,000 rpm for 10 min and then analyzed.

3. Results and discussion

3.1. RhB sorption by fly ash

Effects of fly ash doses on RhB removal were evaluated. RhB of 100 mg L^{-1} and solution pH of 12 were used. Fig. 2a indicates that when 5 g L^{-1} of fly ash was added, 6.7% of RhB was removed after 8 h. RhB removal increased with fly ash doses. When 80 g L^{-1} of fly ash was used, a rapid sorption was observed within the first 1 h and 46% of RhB was removed. Prolongation of the sorption time to 8 h increased the RhB removal to 55%. The results indicate that a high dose of fly ash was needed to remove RhB.

The influence of solution pH on fly ash sorption was investigated. The fly ash dose was 20 g L^{-1} and 1N H_2SO_4 was used to adjust the solution pH. Fig. 2b shows that increasing solution pH from 6 to 12 slightly decreased the RhB removal by fly ash at the initial sorption period (0–4 h). Nevertheless, the RhB removal (18%) was similar for all the applied fly ash doses after 8 h.

From Fig. 2a, the sorption capacity of the fly ash was about 0.0019–0.0023 mmol g^{-1} (8 h) which was lower than the 0.004–0.0115 mmol g^{-1} (72 h) reported by Janos et al. [5]. It is possible that the properties of this fly ash were different from that used

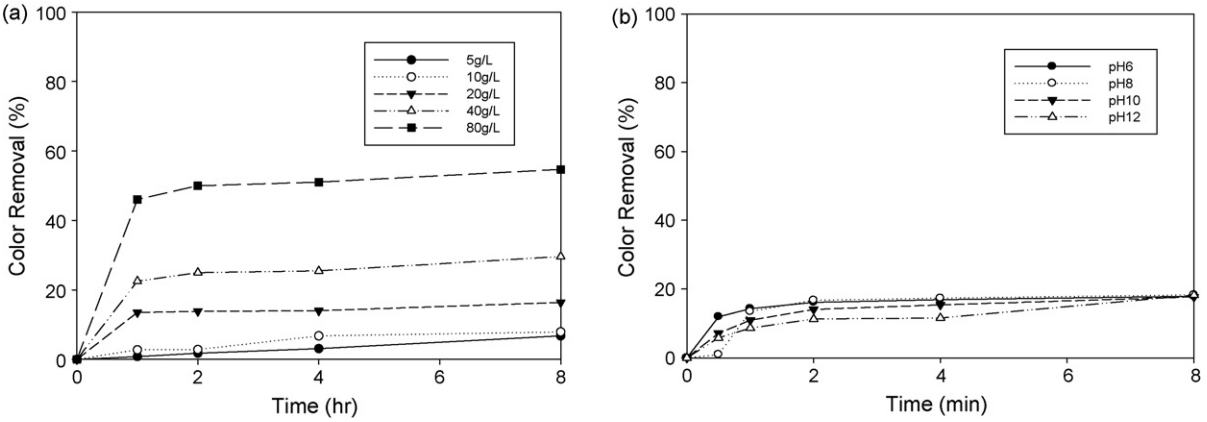

Fig. 2. Effects of fly ash sorption on RhB color removal. RhB concentration was 100 mg L^{-1}. (a) Effect of fly ash doses. Solution pH was not adjusted, (b) Effect of solution pH. Fly ash dose was 20 g L^{-1}.

by Janos et al. [5]; in addition, the sorption period was only 8 h in this study.

Two common isotherms, the Langmuir and Freundlich models, were employed to evaluate the sorption behavior.

(1) Langmuir isotherm

The Langmuir model assumes that the uptake of dye occurs on a homogenous surface by monolayer adsorption without any interaction between adsorbate molecules:

$$q_e = \frac{x}{m} = \frac{q_{max} b C_e}{1 + bC_e},$$

where C_e is the equilibrium concentrations ($mg\,L^{-1}$), q_e the amount of dye sorbed at equilibrium ($mg\,g^{-1}$), q_{max} the maximum monolayer sorption capacity ($mg\,g^{-1}$), and b the equilibrium parameter ($L\,mg^{-1}$) of Langmuir model. A linear form of the Langmuir equation will generate the constants q_{max} and b: $\frac{C_e}{x/m} = \frac{1}{bq_{max}} + \left(\frac{1}{q_{max}}\right) C_e$

(2) Freundlich isotherm

The Freundlich isotherm is empirical for heterogeneous surface energy. It assumes that the adsorption energy of an adsorbate binding to a site on an adsorbent depends on whether or not the adjacent sites are already occupied:

$$q_e = \frac{x}{m} = K_F C_e^{1/n},$$

where K_F ($mg\,g^{-1}$) and $1/n$ are Freundlich constant and exponent, respectively. A linear form of the Freundlich equation will generate the constants $1/n$ and K_F:

$$\ln q_e = \frac{1}{n} \ln C_e + \ln K_F$$

Langmuir isotherm constants of q_{max} and b were $10\,mg\,g^{-1}$ and $0.05\,L\,mg^{-1}$, respectively. The R^2 value for the Langmuir isotherm fitting result was 0.980. The characteristics of Freundlich isotherm of K_F and $1/n$ were $2.87\,L\,g^{-1}$ and 0.233, respectively. The R^2 value for the Freundlich isotherm fitting result was 0.866. From the correlation coefficient (R^2 values), the Langmuir isotherm described the sorption equilibrium data better than the Freundlich isotherms. This suggests that the homogenous surface was responsible for RhB sorption onto fly ash.

Changes in solution pH may affect dye sorption onto fly ash in two ways: (1) the dissolution of dye, (2) the change of surface charges by sorption of H^+ and OH^- onto absorbents [3,5,6,15]. Basic (cationic) dyes, like methylene blue, have positive charge function groups. The major components of the fly ash were Al_2O_3, CaO, Fe_2O_3, and MgO (Table 1), which were positively charged [16]. An increase in solution pH enhances the sorption of hydroxyl ion onto the fly ash and thus enhances the sorption of basic dyes [3,5,6,15]. However, a slight decrease in the RhB sorption by fly ash was observed in the study. This is because even though RhB is a basic dye, it also has an acidic function group (negative charge). The phenomenon that the sorption abilities of fly ash for RhB decrease with an increase of solution pH was also observed by Wang et al. [6]. Because high doses of fly ash were required for RhB removal, methods to increase the fly ash ability for RhB solution was investigated in sequential study.

Table 1
The composition of coal fly ash used in this study.

Composition	Percentage by weight (%)
Al_2O_3	15.44
CaO	16.59
Fe_2O_3	8.4
MgO	1.79
SiO_2	44.88
Loss on ignition	0.19
Surface area (m^2/g)	8.9

3.2. RhB degradation by Fenton oxidation

Effect of Fenton oxidation on color removal of RhB was assessed. The operating conditions were RhB of $100\,mg\,L^{-1}$ and H_2O_2:Fe^{2+} molar ratio of 10:1. Fig. 3a shows the decolorization of RhB by different H_2O_2 doses at initial solution pH of 3. When H_2O_2 of 1.5×10^{-3} M was used, the color removal was rapid and 33% color removal was obtained at 2 min. Increasing the reaction time to 30 min further raised color removal to 42%. When the H_2O_2 dose increased to 3×10^{-3} M and 6×10^{-3} M, 95% and 97% of color removal, respectively, was obtained at 2 min.

The effect of initial solution pH on RhB decolorization by Fenton oxidation was also evaluated. Fig. 3b indicates that when H_2O_2 of 3×10^{-3} M was used, higher RhB color removal (>90%) was obtained at pH 2–3 than that (66%) at pH 4–5 after 30 min reaction time. However, when H_2O_2 dose of 6×10^{-3} M was used, there was no obvious effect of initial solution pH on color removal.

The main reaction pathway for the degradation of RhB is the oxidation of $OH^\bullet$ attack [17]:

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + OH^\bullet + OH^- \quad (1)$$

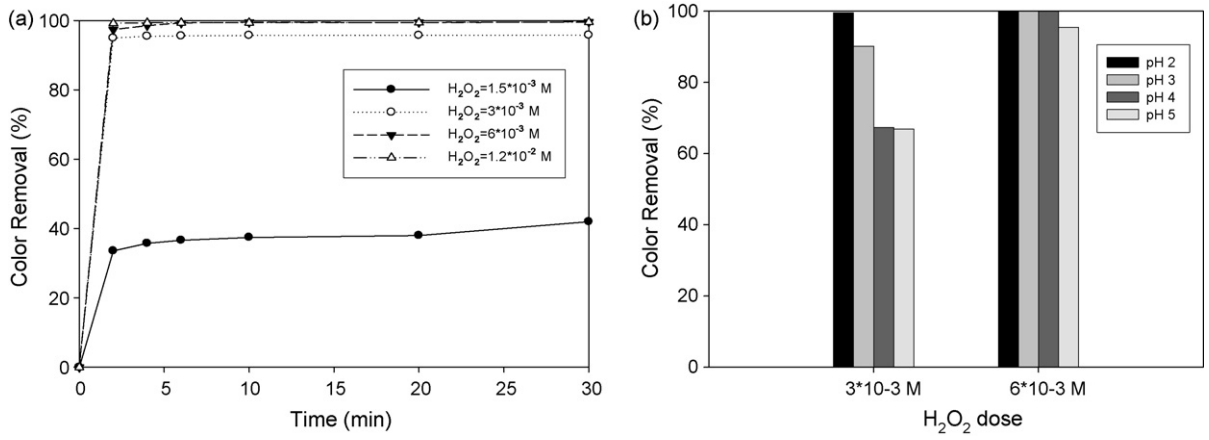

Fig. 3. Effects of Fenton oxidation on RhB color removal. The operating conditions were RhB of $100\,mg\,L^{-1}$ and H_2O_2:Fe^{2+} molar ratio of 10:1. (a) Effect of H_2O_2 dose, initial solution pH of 3. (b) Effect of initial solution pH.

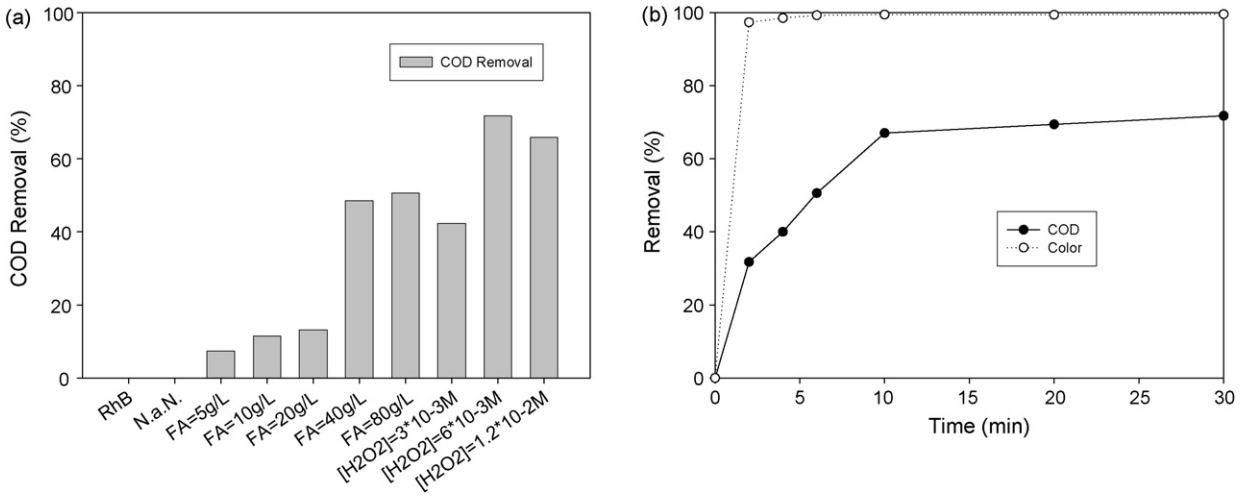

Fig. 4. Color and COD removal by fly ash sorption and Fenton removal. RhB concentration was 100 mg L^{-1}. H_2O_2:Fe^{2+} molar ratio of 10:1 and initial solution pH of 3 for Fenton oxidation. (a) COD removal. Reaction time was 30 min and 8 h for Fenton oxidation and fly ash sorption, respectively. (b) Color and COD removals during Fenton oxidation. H_2O_2 dose of 6×10^{-3} M.

$$Fe^{3+} + H_2O_2 \rightarrow Fe^{2+} + OOH^{\bullet} + H^{+} \quad (2)$$

$$Dye + OH^{\bullet} \rightarrow oxidizeddye + CO_2 + H_2O$$

The hydroxyl radical produced by Fenton reaction can rapidly react with RhB. Dutta et al. [18] utilized Fenton oxidation to degrade a basic dye, methylene blue. They indicated the optimal initial solution pH was acidic (pH 2.4–3.8). A similar result was observed in this study. Fig. 3b indicates there were no obvious effects of solution pH on RhB color removal by Fenton oxidation at H_2O_2 of 6×10^{-3} M. This is because H_2O_2 of 6×10^{-3} M is an overdose for the decolorization of 100 mg L^{-1}. Solution pH of 3 was selected for the sequential experiments.

3.3. COD removal and UV–vis spectrum change

COD removal of RhB by fly ash adsorption and Fenton oxidation was investigated. Fig. 4a illustrates that COD removal increased with an increase in fly ash doses, and 50% of COD removal was obtained at fly ash dose of 80 g L^{-1} at 8 h. As expected, the trends for color and COD removals were similar. Fig. 4a indicates that when H_2O_2 of 3×10^{-3} M was applied in Fenton oxidation, 42% of COD was removed at 30 min. The increase of H_2O_2 dose to 6×10^{-3} M enhanced the COD removal to 71%. However, when H_2O_2 dose was further increased to 6×10^{-3} M, COD removal decreased to 65%.

Fig. 4b indicates the relationships between color and COD removal by Fenton oxidation at H_2O_2 dose of 6×10^{-3} M. As described above, most RhB color (97%, Fig. 3a) was removed within the first 2 min, but most of COD (67%) was removed at the first 10 min. This suggests that COD removal still continued after decolorization ceased, and 71% of COD removal was obtained at 30 min of Fenton oxidation. As mentioned above, hydroxyl radical is a strong oxidant that can react rapidly with RhB. Most oxidation reactions occurred in the initial period. Additionally, the COD removal at H_2O_2 dose of 1.2×10^{-2} M was lower than that at H_2O_2 dose of 6×10^{-3} M in this study. It is possible that when a high concentration of H_2O_2 is used, H_2O_2 acts as free radical scavenger and reduces hydroxyl radical concentration [19].

Changes in the UV–vis spectra of RhB by fly ash sorption and Fenton oxidation were investigated. For RhB solution, the characteristic absorption peak at wavelength 554 nm is attributed to the rhodamine group (chromophore structure). Fig. 5a illustrates the spectrum changes at different fly ash doses of 5–80 g L^{-1} after 8 h sorption. As expected, the spectrum area reduced with the increase of fly ash doses.

Fig. 5b indicates the effect of Fenton oxidation on the changes of RhB UV–vis spectrum at operating conditions of solution pH of

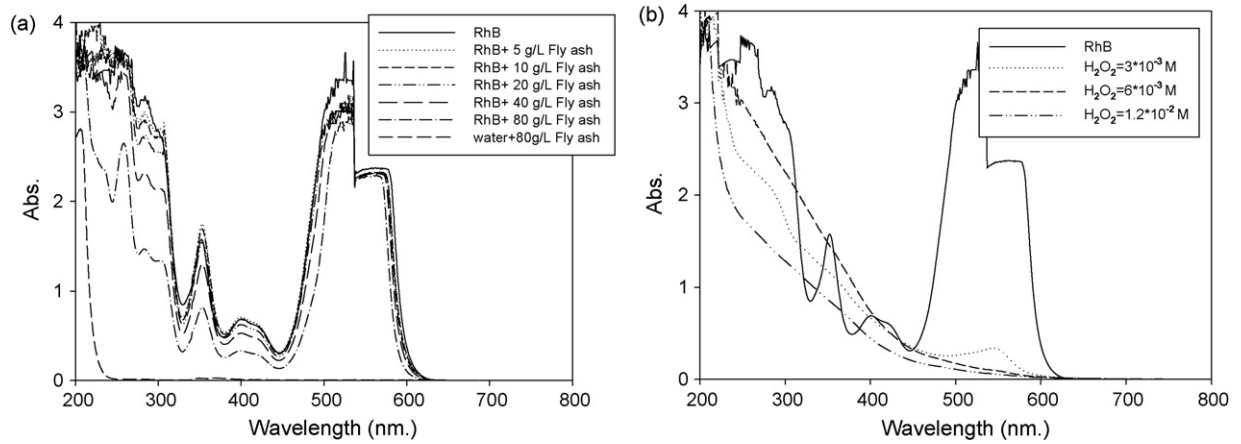

Fig. 5. UV–vis spectrum changes of RhB solution during fly ash sorption and Fenton oxidation. RhB concentration of 100 mg L^{-1}. (a) Fly ash sorption. Sorption time of 8 h. (b) Fenton oxidation. H_2O_2:Fe^{2+} molar ratio of 10:1, initial solution pH of 3, and reaction time of 30 min.

3 and H_2O_2:Fe^{2+} = 10:1 after 30 min reaction time. Fenton oxidation at H_2O_2 of 3×10^{-3} M (assigned as FT1) effectively reduced the visible bands at 450–600 nm. However, a large UV band at 200–400 nm remained. Even though increasing the H_2O_2 dose to 6×10^{-3} M (assigned as FT2) and 1.2×10^{-2} M (assigned as FT3) could further decrease the visible bands spectra at 450–600 nm, the change of UV spectra at 200–400 nm was not obvious. This implies that some aromatic intermediates might have accumulated in the treated solution that could not be degraded by hydroxyl radicals, even at high Fenton reagent dose ($H_2O_2 = 6 \times 10^{-3}$ M to 1.2×10^{-2} M). Behnajady et al. [20] also observed that UV absorption bands at 200–400 nm remained in ultrasound-treated RhB solution. AlHamedi et al. [21] utilized UV/H_2O_2 to destroy RhB and analyzed RhB intermediates by GC/MS. They indicated that the major intermediates in the UV/H_2O_2-treated solution were low molecular weight aliphatic, alcohols, and acids.

Since neither Fenton nor fly ash alone could effectively remove RhB COD in this study and intermediates were present in the Fenton-treated solution, combined Fenton oxidation-fly ash sorption was used to evaluate its ability to remove RhB in the following study.

3.4. RhB removal by combined Fento oxidation and fly ash sorption process

First, the COD removal of Fenton-treated solutions (FT1, FT2, and FT3) by fly ash was evaluated. Fig. 6 illustrates that 1 g L^{-1} of fly ash further increased the COD removal from 42% to 48% for FT1, from 72% to 98% for FT2, and from 65% to 88% for FT3. The increase rates of COD removal by addition of 1 g L^{-1} of fly ash were 6%, 26%, and 23% for FT1, FT2, and FT3, respectively. It is possible that (1) high Fenton reagents ($H_2O_2 = 6 \times 10^{-3}$ M to 1.2×10^{-2} M) converted more RhB to low-molecular-weight (LMW) intermediates which easily enter and sorb onto the fly ash in small-size pores

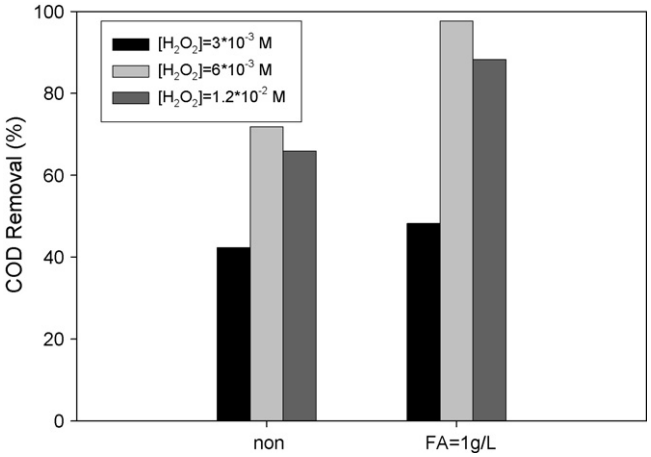

Fig. 6. COD removal of Fenton-treated solution by 1 g L^{-1} of fly ash. Fenton-treated solutions were prepared by addition of H_2O_2 concentration of 3×10^{-3} M, 6×10^{-3} M, and 1.2×10^{-2} M. H_2O_2:Fe^{2+} molar ratio was 10:1 for Fenton oxidation.

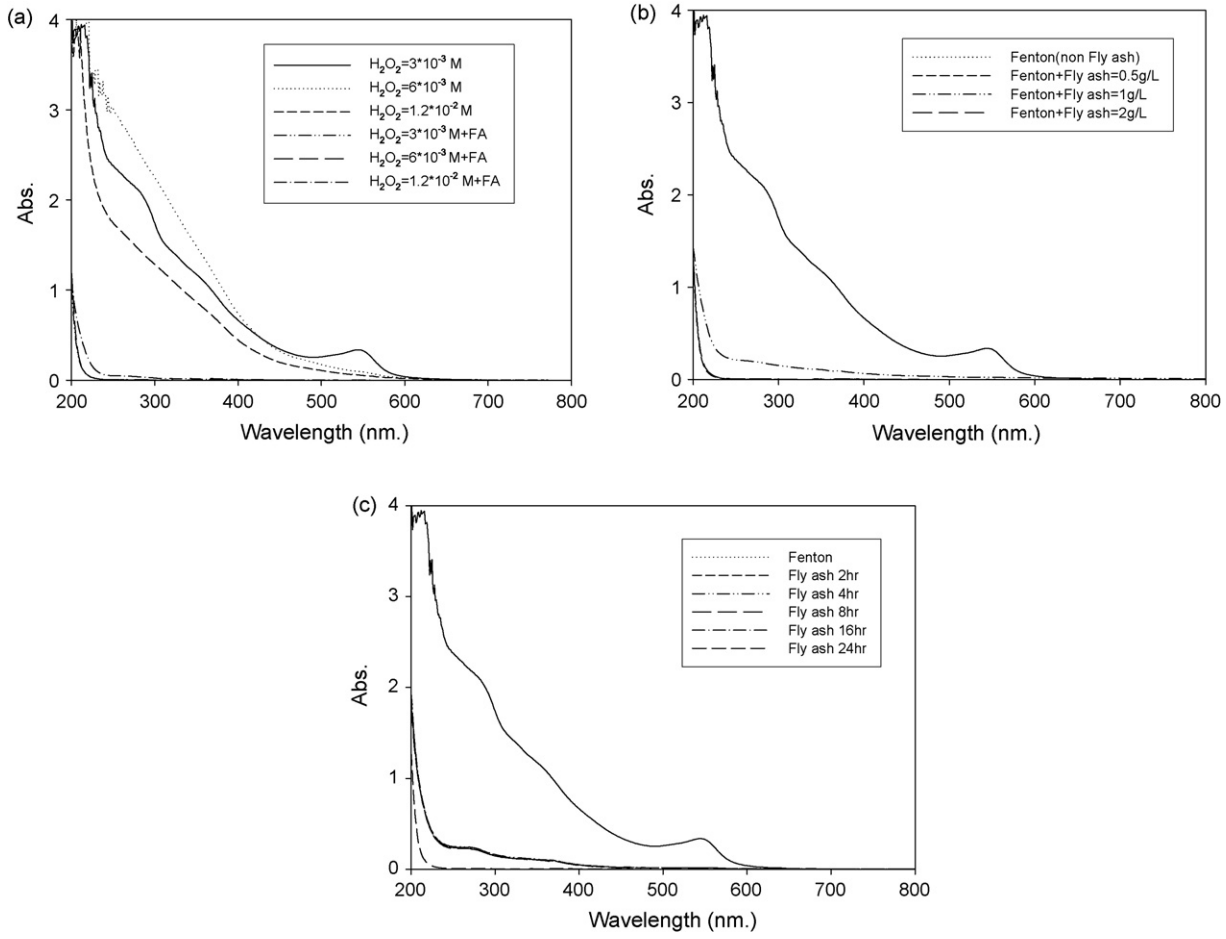

Fig. 7. Effects of fly ash on spectrum changes of Fenton-treated solution. (a) Effect of 1 g L^{-1} of fly ash on different Fenton-treated solutions (oxidized by different H_2O_2 dose. H_2O_2:Fe^{2+} molar ratio of 10:1). (b) Effect of fly ash doses on Fenton-treated solution (oxidized by H_2O_2 of 6×10^{-3} M). (c) Spectrum change of Fenton-treated solution after addition of 1 g L^{-1} fly ash. Fenton-treated solution was oxidized by H_2O_2 of 6×10^{-3} M.

[22], or (2) higher Fenton reagents changed more RhB to negative charges of intermediates than the lower reagents. The negative charges of intermediates sorbed on the positively charged surface of fly ash more easily [21]. The final solution pH of the Fenton-treated solution after fly ash addition was 6. After adding 1 g L^{-1} of fly ash, the pH of Fenton-treated solution increased to 12. At this high pH, the leaching from fly ash should be slight.

The fly ash sorption abilities for COD removal from RhB solution and Fenton-treated RhB solution were also compared. Figs. 4a and 6 indicate that fly ash sorbed much more COD from the Fenton-treated solution than from the untreated RhB solution. For example, the COD removal ability of fly ash was 0.625 mg COD/g fly ash at fly ash dose of 80 g L^{-1}. In contrast, 26 mg COD/g L^{-1} fly ash was obtained from the addition of fly ash to the FT2 solution. The COD removal ability of fly ash in the FT2 solution was 41.6 times higher than in the raw RhB solution.

The effect of fly ash sorption on UV-spectrum changes of FTs was investigated. Fig. 7a shows that the addition of 1 g L^{-1} of fly ash effectively reduced the spectrum band sorption area at 200–600 nm for these FT solutions. This suggests that fly ash could sorb the UV absorption-related intermediates like aromatic compounds. However, Fig. 6 also shows that 52% of COD remained in the FT1 solution after the addition of 1 g L^{-1} fly ash. This implies that some compounds (such as intermediates and undecolorized RhB), which were not responsible for UV absorption, existed in the FT1 solution.

Different doses of fly ash on spectrum changes of FT2 were evaluated (Fig. 7b). When 0.5 g L^{-1} of fly ash was applied, there was still small UV band absorption at the 200–400 nm area in the FT2. Increasing the fly ash dose to above 1 g L^{-1} completely diminished the UV spectrum wavelength to >220 nm. Fly ash of 1 g L^{-1} was suggested to remove COD and intermediates from TF2. The spectrum changes of FT2 with sorption time after the addition of 1 g L^{-1} fly ash was also evaluated. Fig. 7c indicates a rapid decrease in UV-absorption spectrum by fly ash sorption. The UV spectrum wavelength above 220 nm was totally removed after 2 h sorption time. Therefore, 2 h sorption time was suggested for fly ash to remove intermediates from the TF2 solution.

Because intermediate identification analysis, like GC/MS, was not conducted in this study, the mechanism responsible for the intermediate removal of FT by fly ash is unclear. Further study on RhB intermediate removal by fly ash is suggested. To conclude, the combined process is a potential technique for RhB removal.

4. Conclusions

The effects of fly ash sorption, Fenton oxidation, and combined Fenton oxidation-fly ash sorption were evaluated for RhB removal. A solution pH from 6 to 12 did not obviously influence the fly ash sorption of RhB after 8 h sorption. Even though fly ash was low-cost, high doses of fly ash were required for RhB removal. When 80 g L^{-1} of fly ash was applied, the COD removal ability of fly ash was 0.625 mg COD/g fly ash. Even though Fenton oxidation at H_2O_2 dose of 3×10^{-3} M to 1.2×10^{-2} M completely decolorized RhB, 29–58% COD remained in the Fenton-treated solution. When H_2O_2 dose of 6×10^{-3} M was used, color and COD removal were 99% and 71%, respectively. The addition of 1 g L^{-1} of fly ash further increased the COD removal to 98%, and the UV adsorption band >220 nm vanished after 2 h sorption time. The COD removal capacity of fly ash for Fenton-treated RhB solution was 41.6 times higher than that for untreated RhB solution. The results indicated that the combined process is a potential technique for RhB removal.

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