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Photo-assisted degradation of anionic and cationic dyes over iron(III)-loaded resin in the presence of hydrogen peroxide

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Abstract

Ferric species were immobilized on different types of ionic exchange resins, and used as a heterogeneous catalyst for degradation of cationic and anionic dye pollutants under visible light ($\lambda > 450$ nm) irradiation in the presence of H_2O_2. It was found that for the photodegradation of cationic dyes, malachite green and methylene blue, the immobilized catalyst on a strongly or weakly cationic resin was much more effective than the catalyst supported on an anionic resin, whereas an opposite order in the catalyst activity was observed for the photodegradation of anionic dyes, phloxine and X3B. This significant difference was mainly attributed to the dye adsorption, which enhanced the electron transfer from the excited dye to the immobilized ferric species. All the catalysts were stable upon visible light irradiation, and less than 3.2 μM of dissolved Fe species was detectable in the filtrate solution. When the ferric species was loaded on an amphoteric exchange resin, the immobilized catalyst was effective to the photodegradation of both cationic and anionic dyes. The anionic dyes were notably adsorbed on such the catalyst, but the catalyst exhibited weak affinity toward cationic dyes. Due to the inner salt effect, some dissolved ferric species was released from the solid, and consequently made some contribution to the overall dye degradation via both the Fenton reaction and the photosensitization pathway. Although the amphoteric resin-supported catalyst was stable to the visible light, it was corroded to a certain level upon UV irradiation. It was found that such photodissolution was greatly depressed in the presence of H_2O_2.

Keywords: Dyes; Hydrogen peroxide; Iron species; Visible light; Degradation; Photo-Fenton

1. Introduction

Photo-Fenton reaction is an attractive environmental remediation technology for degradation of toxic organic pollutants. In this process, hydroxyl radicals ($OH^\bullet$), generated from a conventional Fenton reaction (Eq. (1)), and/or photolysis of ferric species (Eq. (3)), are proposed to be the main reactive species for the oxidation and mineralization of various organic pollutants in water. Although the iron species can be recycled via a thermal pathway (Eq. (2)), the process is relatively slow. The combination with UV irradiation is then found to be an efficient means to recycle Fe species and consequently enhance the overall reaction. The photo-Fenton reaction has been applied in many studies for the destruction of hazardous organic wastes, such as nitrobenzene [1], azo-dyes [2], benzoic acid [3], heroin [4], Orange II [5], and Herbicides [6]. However, the application of this photo-Fenton reaction is limited by its narrow pH (<3), separation and recovery of the iron species, and the cost of expensive UV energy. Therefore, the development of an efficient heterogeneous system that can operate preferably at neutral pH and under visible light irradiation is a working direction in the research field of the photo-Fenton reaction.

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + OH^\bullet + OH^- \qquad k_1 = 76\,\mathrm{M/s} \tag{1}$$

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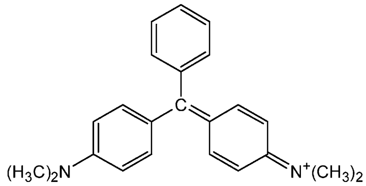
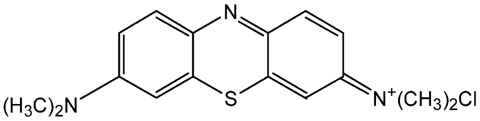
$$Fe^{3+} + H_2O_2 \rightarrow Fe^{2+} + HO_2^{\bullet} + H^{+} \qquad k_2 = 0.002\,\mathrm{M/s} \tag{2}$$

$$Fe(OH)^{2+} + h\nu \rightarrow Fe^{2+} + OH^{\bullet} \tag{3}$$

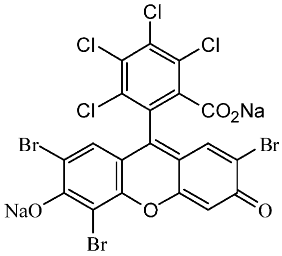
The Fe(III) species have been immobilized on various supports including Nafion membrane [7,8], Alginate gel beads [9], structured silica woven fabrics [10], laponite clay [11], and cationic exchange resin [12]. These catalysts are mostly active under UV light irradiation for organic degradation, following a pathway similar to the photo-Fenton reaction. Recently, Zhao and co-workers [13] have claimed that the ferric species immobilized on a cationic resin is an effective catalyst for the degradation of cationic dyes under visible light irradiation in the presence of H_2O_2. A photosensitization pathway is proposed as the primary step to initiate the dye degradation. Under the same conditions, however, this catalyst is not reactive for the destruction of anionic dyes such as SRB and Orange II, attributed mainly to the poor dye adsorption. Such disadvantage, obviously, would limit its practical application for the treatment of various types of dye pollutants.



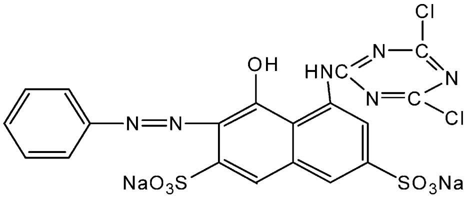
Malachite Green (MG)



Methylene Blue (MB)



Phloxine (PO)



Reactive Brilliant Red X3B (X3B)

In this study, three different types of ionic exchange resin, cationic, anionic, and amphoteric polymer were used as the support of photoactive ferric species so as to improve the catalyst adsorption affinity toward cationic and anionic dyes from aqueous medium. Whereas the anionic exchange resin is known to uptake effectively the anionic dyes, both cationic and anionic dyes may find adsorptive sites on an amphoteric exchange resin, so that they can concomitantly photodegrade on the immobilized Fe(III) catalyst. The photochemical reaction was performed under visible light ($\lambda > 450$ nm) irradiation in aqueous medium of H_2O_2 using malachite green and methylene blue as a model cationic dye and phloxine and X3B as a model anionic dye, respectively. The possible leaching of iron species from the catalyst into aqueous phase was also examined during the photoassisted reactions, especially under UV irradiation.

2. Experimental

2.1. Material and reagents

All the ionic exchange resins were obtained from Zhengguang Resin Co. Ltd., including the strongly cationic resin (CR), weakly cationic resin (WCR), anionic resin (AR), and amphoteric resin (AMR), with an ionic exchange capacity of about 3.0 mmol/g. All the chemical reagents were of laboratory reagent grade and used without further purification, including malachite green (MG), methylene blue (MB), phloxine(PO), reactive azo-dye X3B (X3B), 4-chlorophenol (4-CP), H_2O_2, $(NH_4)\ Fe(SO_4)_2$ and $HClO_4$. Doubly distilled deionized water was used throughout this study.

The amphoteric ion exchange resin, before use, was exchanged with 1 M HCl for three times, followed by a thorough washing with water. The resin after dried at room temperature was ground and sieved by 200 mesh sieve. The resin was then mixed with a fresh solution of ferric ions (0.02 M, pH 3 in $HClO_4$). After the mixture was stirred overnight, the resin was filtrated, dried at 100 °C for 2 h and rinsed thoroughly with water until Fe concentration in the filtrate was constant. The iron content in the final dry resin was determined to be 2.90×10^{-4} mol/g, and the sample was named as AMR–Fe in the text. The immobilized Fe-catalyst on other ionic exchange resin, CR, WCR and AR, was prepared following a similar procedure, and was named in the text as CR–Fe, WCR–Fe, and AR–Fe, respectively. The corresponding Fe-loading was measured to be 8.25×10^{-4} mol/g, 4.16×10^{-4} mol/g, and 8.18×10^{-5} mol/g, respectively.

2.2. Procedure and analysis

The suspension containing 10 mg of the catalyst (AMR–Fe 30 mg) and 50 mL of the aqueous dye solution were first shaken constantly in the dark overnight. After H_2O_2 was added, the equilibrated suspension was irradiated by a Halogen lamp (500 W, Shanghai Yamin). The reactor was thermostated at 25 °C and stirred constantly during the reaction. At certain intervals, small aliquots of the suspension were withdrawn by syringe, centrifuged, and analyzed on an Agilent 8453 UV–Vis spectrometer. The concentration of Fe(II) species was determined via its complex with 1,10-phenanthroline at 511 nm, whereas the total Fe concentration was measured after all the ferric species were reduced by hydroxylamine. When the dye pollutant was present, the Fe content in the filtrate was analyzed by ICP (Inductively Coupled Plasma, IRIS Intrepid II XSP). The pH value was monitored on a PHS-3C pH meter.

3. Results and discussion

3.1. Adsorption

The dye adsorption on each catalyst was measured in the dark after the suspension was sufficiently equilibrated overnight. Table 1 summarizes the data in the initial dye concentration, C_0, the amount of adsorption, n_{ad}, and the suspension pH after dark adsorption (note that this suspension was directly used for the photochemical study). Although the adsorption isotherm was not determined, the data in Table 1 could be served as a qualitative reference for the comparison in adsorption among different immobilized catalysts. As it is expected, the cationic dyes (MG and MB) on the Fe-loaded cationic resin (CR–Fe and WCR–Fe) and the anionic dyes (X3B and PO) on the Fe-loaded anionic resin (AR–Fe) were all strongly adsorbed, due to strongly electrostatic interaction, whereas the adsorption was weak on an oppositely charged resin. Obviously, an ionic exchange mechanism was mainly responsible for such observed adsorption.

On the Fe-loaded amphoteric resin (AMR–Fe), however, the dye adsorption was not the case as expected. The composite catalyst exhibited notable affinity toward anionic dyes, but its interaction with cationic dyes were extremely weak. The amphoteric resin possesses a framework of oppositely charged groups, $RCOO^-$ and $N(R)_4{}^+$, which form an inner salt. During the anionic dye adsorption, the inner salt bond was opened up to uptake the dye anions and sodium cations, respectively. Since the cationic dyes could be notably adsorbed on a carboxylate resin (WCR), their little adsorption on AMR-based catalyst might be attributed to steric hindrance of the large sized dye cations, which limited effective opening of the strong inner salt bond. Similar evidence was also seen during the loading of cationic Fe(III) species. Although about 0.29 mmol Fe/g remained in the prepared catalyst, some amount of ferric species was always detectable in the filtrate when the prepared catalyst was repeatedly dispersed and washing with water. Thus the amphoteric resin employed here was not an expected good support for the uptake of cationic dyes as well for the Fe(III) loading. As it will be shown below, however, this immobilized catalyst was still photoactive to initiate the degradation of both cationic and anionic dyes in the presence of H_2O_2.

3.2. Photodegradation of dye pollutants over Fe-loaded cationic and anionic resins

The photochemical reaction was carried out only after adsorption/desorption equilibrium was reached. The reaction suspension was the same as those indicated in Table 1. Fig. 1 shows the results obtained in the presence of H_2O_2 under visible light irradiation or in the dark. As the cationic dye was first concerned, both MG (Fig. 1A) and MB (Fig. 1B) were efficiently photodegraded on the Fe-loaded cationic resins (CR and WCR), whereas the photoreaction on the anionic resin supported catalyst (AR) was quite slow. The control experiment in the dark, as indicated by an open symbol in the figure, showed a negligible bleaching of the dye, except MB on the CR–Fe catalyst. In fact, a similar dark reaction was observed also for MG degradation on the CR–Fe catalyst. When the initial pH was set at pH 6.3, 5.0 and 4.0, the dark reaction of MG gave the apparent first-order constant of 0.22, 1.68 and $2.77 \times 10^{-3}\ \mathrm{min}^{-1}$, respectively. No substantial free Fe species was detected by ICP in the solution before the photoreaction (1.70 μM) and after 3 h of irradiation (1.81 μM) as well (note that if all the Fe species leached off from the

Table 1
Adsorption data of textile dyes on different Fe(III)-loaded resins in water[a]

Dyes	CR–Fe			WCR–Fe			AR–Fe			AMR–Fe		
	C_0[b]	n_{ad}[c]	pH[d]	C_0[b]	n_{ad}[c]	pH[d]	C_0[b]	n_{ad}[c]	pH[d]	C_0[b]	n_{ad}[c]	pH[d]
MG	4.52	20.8	6.31	2.47	9.2	3.85	0.53	nd[e]	4.73	0.25	nd[e]	4.01
MB	4.08	17.6	4.22	2.54	9.9	3.79	0.59	0.04	6.20	0.29	nd[e]	4.00
X3B	0.80	nd[e]	4.66	0.80	nd[e]	4.76	4.62	13.4	4.68	1.59	0.81	4.03
PO	0.35	nd[e]	5.46	0.35	nd[e]	5.02	1.71	6.74	6.60	0.58	0.57	4.09

[a] The data were determined at room temperature after the suspension was stirred in the dark overnight. AR, CR, WCR and AMR represented anionic, cationic, weakly cationic and amphoteric resins, respectively. The catalyst concentration was 0.200 g/L, except AMR–Fe which was at 0.600 g/L.

[b] C_0 was the initial concentration of the dye solution in a unit of 10^{-4} M.

[c] n_{ad} was the amount of dye adsorption in a unit 10^{-4} mol/g.

[d] The suspension pH after equilibrium.

[e] No detectable.

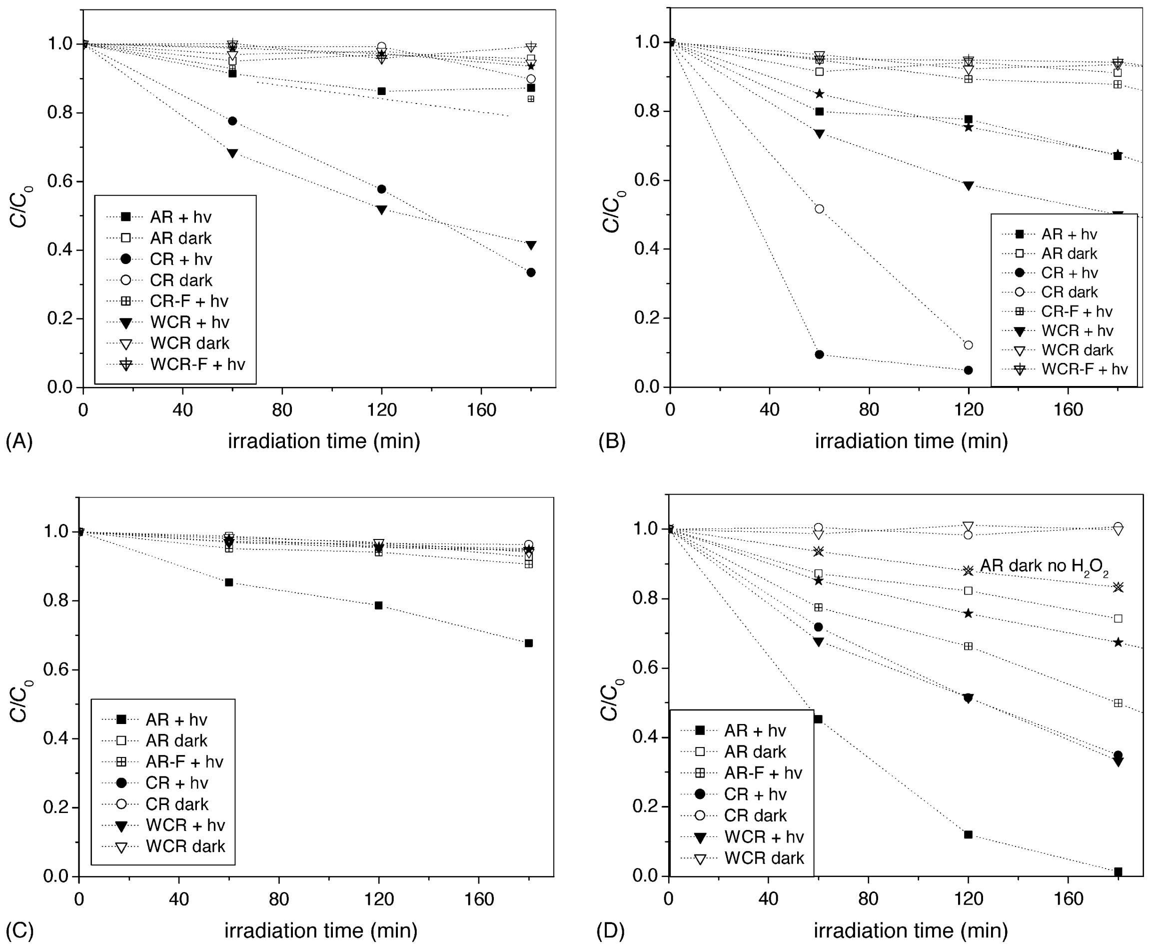


Fig. 1. Degradation of cationic dyes, MG (A) and MB (B), and anionic dyes, X3B (C) and PO (D), in the aqueous suspension of iron(III)-loaded resins (0.20 g/L) and H_2O_2 (2 mM) under visible light irradiation (solid symbol) or in the dark (open symbol). The curve, presented by a five-star, was the corresponding homogenous reaction with H_2O_2 under light irradiation in the absence of catalyst. In the legend, AR, CR and WCR represented the supported iron(III) catalyst on anionic, cationic, and weakly cationic resins, respectively, whereas the symbol F presented the corresponding homogeneous sample in which the solid catalyst was only removed before light irradiation. The initial dye concentration was listed in Table 1.

CR–Fe catalyst the total Fe concentration would be about 165 μM). Such trace amount of ferric species leached from the catalyst was not able to initiate considerable homogenous photoreaction in the presence of H_2O_2, as presented by the crossed symbols in the plot (note that for this study the reaction solution was prepared by simple removal of the solid catalyst from the fully dark-equilibrated suspension, in order to ensure the dissolved ferric species in the exactly same concentration and polymerization state as those dissolved during the heterogeneous reaction). Thus the observed degradation of MB or MG in the dark was attributed to the Fenton reaction mostly on the catalyst surface. It was noted that in a homogenous solution of H_2O_2, the dye MB underwent as well a considerable degradation upon visible light irradiation, whereas MG was relatively stable. The observed difference was attributed to their different chemical reactivities. On the other hand, the anionic dyes, X3B (Fig. 1C) and PO (Fig. 1D), were all photodegraded efficiently on the anionic resin supported catalyst (AR), whereas the photodegradation on the cationic resin-supported Fe catalyst (CR and WCR) was not efficient or relatively slow. The anionic dye PO, similar to MB, was also unstable toward the light irradiation in the presence of H_2O_2. Again, the free Fe species in the filtrate before and after photoreaction was below the ICP detection limit (0.9 μM), and were not able to initiate an obvious Fenton and photo-Fenton reaction (Fig. 1C and D) (note that if all the Fe species leached off from the AR–Fe catalyst the total Fe concentration would be about 16.4 μM). In all the cases (Fig. 1), no significant change in pH (less than 0.20 in unit) was observed during the photochemical processes.

The primary process of the dye photodegradation over the immobilized Fe catalyst occurs basically via a photosensitization pathway, as proposed recently by Zhao and co-workers [13]. The dye in its electronically excited state injects the electron to Fe(III), and thus the dye adsorption is a prerequisite for such efficient surface reaction with the immobilized ferric species. The resulting Fe(II) species is then re-oxidized back to Fe(III) by H_2O_2 via a Fenton pathway, and consequently the produced $OH^\bullet$ radicals led to step-wise degradation of the dye or the dye cation radicals. It is evident that the efficiency of such photosensitization is determined first by the energetics between the excited dye and the Fe(III) acceptor level. Thus among different dye substrates, different rate of the dye photodegradation should be observed. This was seen in Fig. 1, especially for X3B, which was relatively more resistant to degradation. Secondly, the reaction mechanism becomes more complicated when the dye substrate is also photosensitive to degradation, such as in the cases of MB and PO. But the photosensitization pathway is still the dominant one. Thirdly, a weakly cationic resin (WCR) contains carboxylate group, and the catalyst WCR–Fe may undergo the photo-Kolbe reaction generating Fe(II) and other radicals that would contribute into the overall dye degradation on WCR–Fe catalyst upon visible light irradiation. This was seen that although the dyes, MG and MB, were weakly adsorbed on such WCR–Fe catalyst, their photodegradation was still comparable to the one on a strongly cationic resin (CR–Fe). Lastly, no considerable amount of free Fe species was released into the solution before and after 3 h of the photoreaction. Thus, any observed dark degradation was due to the heterogeneous Fenton reaction on the catalyst surface in a weakly acidic medium.

The result in this section supports the early proposal that sufficient adsorption is the necessary condition for the substrate photodegradation over Fe-loaded resin catalyst in the presence of H_2O_2. But it is only true when the energetics is satisfied. The free energy of ferric species would get low when it is immobilized on a support. The cationic dye MG on AR–Fe catalyst, although its adsorption was below detection limit, underwent still some photodegradation (Fig. 1A). The anionic dye X3B was not reactive to ferric ions under visible light irradiation, but it was photodegraded obviously on Fe-loaded AR catalyst, in the aid of adsorption (Fig. 1C). It is evident that the selective adsorption of the dye on an appropriate support is a promising means to improve their photodegradation. In this regard, the versatile support for Fe-loading would be the one that is amphoteric toward both cationic and anionic dyes for their co-adsorption.

3.3. Photodegradation of dye pollutants over Fe-loaded amphoteric resin

Fig. 2 shows the time profiles of the dye degradation on Fe-loaded amphoteric resin under visible light irradiation or in the dark in the presence of H_2O_2. Whereas the anionic dyes, X3B and PO, were photodegraded, because of their strong adsorption, the cationic dyes, MG and MB, which were hardly adsorbed on the catalyst, was also efficiently photodegraded. Relatively, the photodegradation of X3B was again very slow, owing to its high chemical stability. It was found that a considerable amount of ferric species (7.6 μM) was in the aqueous solution of the catalyst, but remained almost the same after 3 h of photoreaction(note that if all the Fe species leached off from the AMR–Fe catalyst the total Fe concentration would be about 174 μM). This amount of dissolved free ferric species could initiate a homogenous Fenton reaction for all of four dyes, as indicated by the open symbols in Fig. 2. The dissolved iron species also permitted the photosensitized dye degradation in a homogeneous solution (the crossed symbol in Fig. 2), and the reaction rate under visible light irradiation was higher than that in the dark. Moreover, the immobilized Fe species did contribute its photoactivity to the overall dye degradation, as demonstrated by the crossed symbols in the figure.

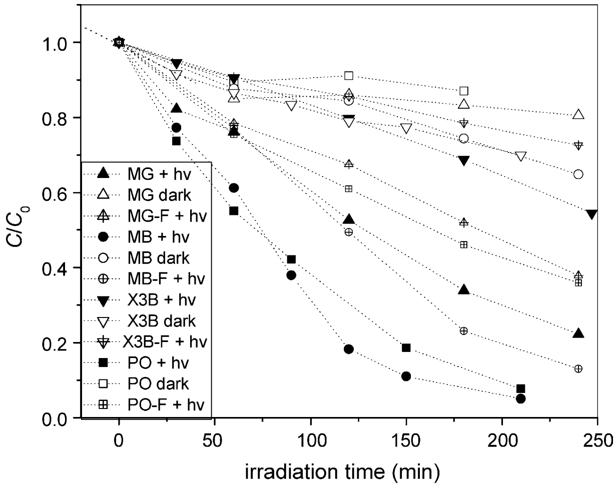


Fig. 2. Degradation of the textile dyes over an amphoteric resin supported Fe-catalyst (0.600 g/L) in the presence of H_2O_2 (2 mM) under visible light radiation (solid symbol) or in the dark (open symbol). The symbol F presented the corresponding homogeneous sample in which the solid catalyst was only removed before light irradiation. The initial concentration of dye solution was listed in Table 1.

The leaching of Fe species from the solid into the aqueous solution was due to the effect of inner salt bond, as discussed early in this paper. Although the dissolved Fe is in the limit of environmental regulation, such trace amount of Fe species was able to initiate both the Fenton and photosensitized reactions under the present conditions. Its continuous dissolution into a fresh aqueous solution would limit its practical application. Thus, this type of amphoteric resin was not as good as we previously expected.

3.4. Stability of amphoteric resin-supported Fe catalyst against UV light

All the catalysts presented above were effective for the dye degradation under UV light irradiation, when the dye was adsorbed on the catalyst (the cationic dyes on AMR–Fe were not adsorbed, but it could undergo UV-assisted degradation, due to some dissolved Fe-species). This offers another support that the preconcentration of dye pollutants on the catalyst enhances not only the photosensitization, but also the dye degradation by $OH^\bullet$ radicals generated from Fe(III) photolysis on the catalyst surface. Since the conclusion was similar to the case under visible light irradiation, the details about the UV-assisted reaction were not included in this paper.

However, it is quite necessary to discuss the photostability of the catalyst, especially upon UV light illumination, which is rarely concerned in the literature. It is known that the Fe(III)-related compounds, such as ferric ions, iron oxide [14] and Fe(III) complex, undergo easily the ligand-to metal charge transfer reaction upon light absorption, with formation of ferrous species and some other reactive radicals. The reoxidation of Fe(II) by molecular oxygen is generally slow, but this oxidation is greatly enhanced in the presence of H_2O_2. Thus, it would be interesting to examine how the Fe species in the oxidation state changes during the photoassisted reaction in the presence of H_2O_2. Since the detection of ferric species was interfered by the colored substrate, this experiment was conducted using 4-chlorophenol as the model organic pollutant. Although the practical situation may vary from one to another, the information obtained from 4-CP reaction would be relevant to the future study on the catalyst photostability. Secondly, the immobilized Fe catalyst, even used only for visible light application, may be exposed occasionally to the UV irradiation, leading to the catalyst deactivation.

Fig. 3 shows the concentration changes of Fe(II), Fe(III) and total Fe with UV irradiation, detected in the aqueous phase of AMR–Fe catalyst, and the corresponding effect of 4-CP or H_2O_2 concentration. In the absence of either 4-CP or H_2O_2, the Fe leaching was notably enhanced by UV light (Fig. 3 (i)). The valence state analysis showed that the leached Fe species was primarily ferrous ions, which was oxidized to ferric ions, followed by back Fe(III) adsorption onto the resin. Due to Fe(III) equilibrium between solution and solid interface, the total Fe concentration remained unchanged after 1 h of irradiation. When 4-CP was present, however, such photodissolution became serious (Fig. 3A). The concentration of total Fe species increased notably with the increase in initial 4-CP concentration (Fig. 3C), but the increase in Fe(II) concentration was followed by the decrease in Fe(III) concentration. Since the organic substrate 4-CP was stable to the UV light at $\lambda > 320$ nm, the enhanced dissolution of Fe species upon 4-CP addition was attributed to the photo-induced redox reaction between 4-CP and Fe(III), and/or the $OH^{\bullet}$ radical consumed by 4-CP shifting the reaction (Eq. (3)) to the right. The reaction possibly occurred either on the catalyst surface or in the solution. When H_2O_2 was added with-

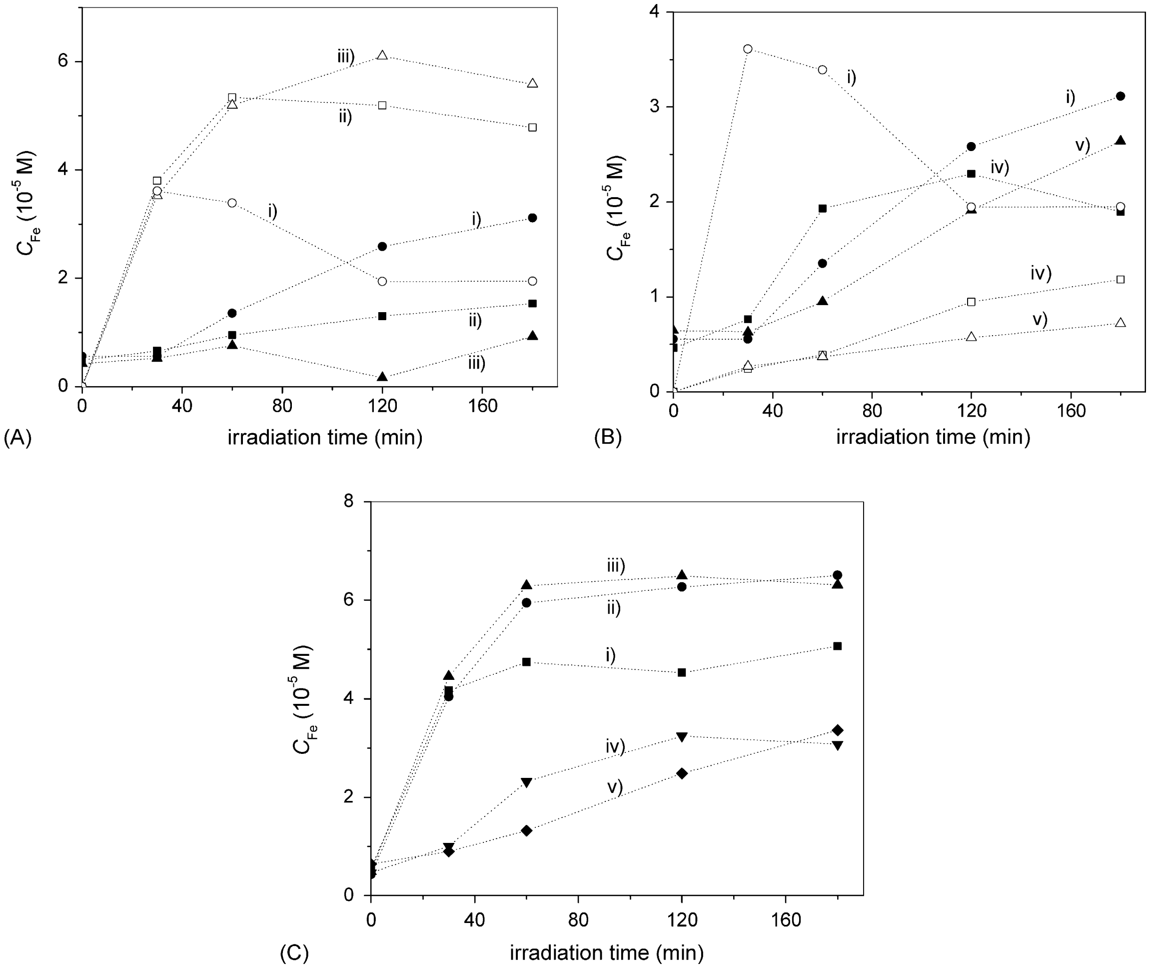


Fig. 3. Effect of 4-chlorophenol (A) or H_2O_2 (B) on the release of iron species from Fe(III)-loaded amphoteric resin into the aqueous solution under UV ($\lambda > 320$ nm) irradiation. The open and solid symbols in (A and B) represented the ferrous and ferric species, respectively. The total amount of iron species, i.e. Fe(II) + Fe(III), was shown correspondingly in (C). The initial concentration of 4-chlorophenol was set at (i) 0, (ii) 0.79, and (iii) 2.3 μM, whereas the initial H_2O_2 was (i) 0, (iv) 2, and (v) 5 mM.

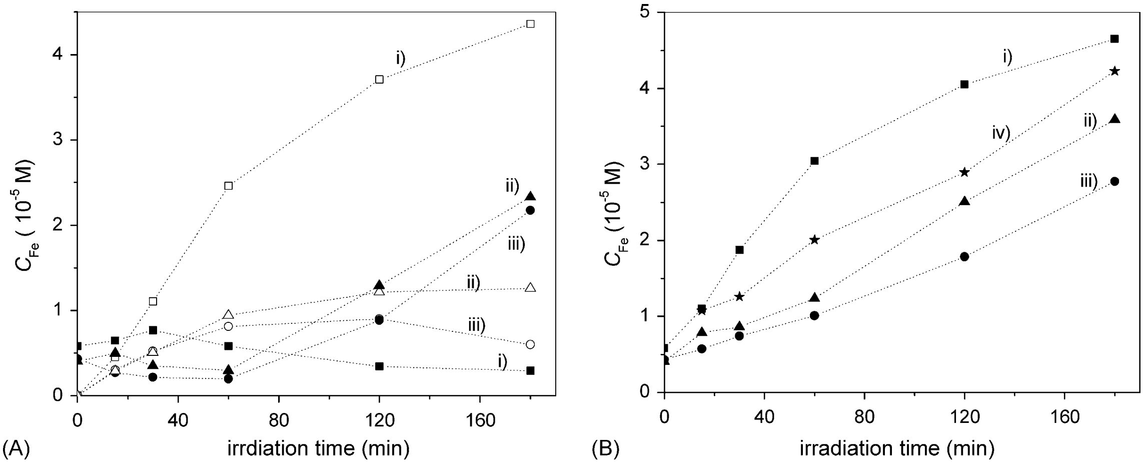


Fig. 4. Effect of H_2O_2 concentration on the release of iron species from Fe(III)-loaded amphoteric resin into the aqueous solution when irradiated by UV ($\lambda > 320$ nm) in the presence of 4-chlorophenol (2.3 μM). The open and solid symbols in (A) represented the ferrous and ferric species, respectively, whereas (B) showed the corresponding change in the amount of total iron species. The initial concentration of H_2O_2 was (i) 0.5, (ii) 2, and (iii) 8 mM. (iv) 4-CP (0.77 μM) + H_2O_2 (2 mM).

out presence of 4-CP, an opposite was observed (Fig. 3B). Increasing H_2O_2 concentration led to a decrease in the total Fe dissolution, in company with the decrease in Fe(II) concentration and the increase in Fe(III) concentration. It was evident that Fe(II) was oxidized by H_2O_2, which was faster than the Fe(III) photolysis, and was faster as well than the photoreaction between 4-CP and Fe(III) species. The photolysis of Fe(III) to generate Fe(II), followed by Fe(II) reoxidation by H_2O_2, occurred mostly on the catalyst surface, so that the iron species was recycled directly on the catalyst without significant diffusion into the solution phase.

In a combined system, the photo-dissolution of Fe-species was also greatly depressed by H_2O_2 (Fig. 4), similar to the case observed in the absence of 4-CP (Fig. 3). In such the system, however, the rate of Fe-dissolution was also decreased with the increase of 4-CP concentration (Fig. 4 B(iv)). It gave another supporting evidence that the oxidation of Fe(II) by H_2O_2 was faster than that of Fe(III) photolysis or its photoreaction with 4-CP. However, the UV-induced dissolution of Fe species still occurred whatever 4-CP and H_2O_2 were both present. This information would be very important to further development of the immobilized photo-Fenton catalyst.

4. Conclusions

The immobilization of iron species onto different types of ionic exchange resin has been studied in order to expand their application for the photodegradation of various dye pollutants with visible light and H_2O_2. Whereas the cationic dyes are efficiently photodegraded on a cationic resin exchanged Fe(III) catalyst, the anionic dye is preferably photodegraded on an anionic resin supported catalyst. Although the amphoteric resin used in this work is not a good support for Fe loading, it offers an idea that both the cationic and anionic dyes are expected to degrade simultaneously upon visible light irradiation in the presence of H_2O_2. It confirms further that adequate adsorption of the target dye pollutants on the catalyst is the prerequisite for its efficient photodecomposition. In addition, a notable dissolution of Fe species into the solution upon UV light irradiation is a serious problem that needs to be faced in the future study about the immobilized Fe(III) catalyst.

Acknowledgements

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