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Degradation of salicylic acid by photo-assisted Fenton reaction using Fe ions on strongly acidic ion exchange resin as catalyst

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Abstract

The photo-assisted Fenton degradation of salicylic acid (SA) was studied by using strongly acidic ion exchange resin (SAIER) exchanged with Fe ions as catalyst in the presence of UV light (254 nm) and H_2O_2. The X-ray photoelectron spectroscopy (XPS) results revealed that the Fe ions are indeed anchored by the sulfonate groups on the surface of the SAIER and are Fe^{3+}. Our results also indicate that the Fe/SAIER catalyst can significantly enhance the degradation rate of SA without remarkable Fe leaching to the solution, implying that the catalyst has photo-catalytic activity. In addition, the effects of $[H_2O_2]/[SA]$ molar ratio, UVC light power, solution pH, catalyst loading, and initial SA concentration on the degradation of SA were investigated in detail.

Keywords: Photo-assisted Fenton reaction; Hydrogen peroxide; Salicylic acid; Degradation

1. Introduction

Since 1990s, photo-assisted Fenton reaction has been studied extensively and regarded as a very effective approach to degrade many organic compounds including non-biodegradable azo dyes in wastewater treatment [1–13]. Traditionally, the photo-assisted Fenton reaction is conducted in a homogeneous phase by irradiating a solution containing Fe^{3+} or Fe^{2+} ions and H_2O_2 in the presence of UV light. The decomposition of H_2O_2 in acidic media is catalyzed by oxidizing Fe^{2+} to Fe^{3+} and subsequently photo-reduced back to Fe^{2+} by photons. Accordingly, the $^{\bullet}OH$ radicals generated from the reaction initiate the degradation of organic compounds.

However, it should be pointed out that the Fe sludge with a large volume after the photo-assisted Fenton reaction is a big disadvantage because the removal of Fe ions from the treated water is a very costly process, which needs a large amount of chemicals and manpower. To overcome this drawback, several attempts have been made in the development of heterogeneous photo-assisted Fenton reaction [14–17]. For example, Fernandez et al. [14–16] investigated photo-assisted Fenton degradation of non-biodegradable azo dye (Orange II) and found that the degradation could be effectively catalyzed by Nafion cation-transfer membrane exchanged with Fe ions in the presence of H_2O_2. The sulfonate groups on the surface of the Nafion film could tightly anchor the Fe ions so that no Fe ions were detected in the solution. In addition, Puma and Yue [17] studied the heterogeneous photo-assisted Fenton oxidation of indigo carmine dye on iron-Nafion pellet and found that the iron-Nafion based catalyst is effective in reducing the concentration of indigo carmine dye in aqueous solution. However, it should be stressed that the photo-catalyst immobilized on Nafion films is too expensive to be used for a practical industrial application. Thus, it is necessary to explore a suitable catalyst support that has similar functions as Nafion film but is much cheaper. According to previous studies [18], there are many sulfonate groups on the surface of strongly acidic ion exchange resin (SAIER). Due to this feature, the SAIER could be a promising candidate for catalyst support for photo-assisted Fenton reaction.

In the present work, Fe/SAIER catalyst was prepared by using SAIER as support through a simple ion exchange reaction with $FeCl_3$ in solution. Salicylic acid (SA) was chosen as the model pollutant because it is a common chemical in dye industry. The photo-assisted Fenton degradation of salicylic acid in aqueous solution under different conditions was investigated. The effects of $[H_2O_2]/[SA]$ molar ratio, UVC light power, solution pH, catalyst loading, and initial SA concentration on the degradation of salicylic acid were discussed in detail.

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2. Experimental

In this investigation, the SAIER supplied by Jiangyin Chemical Plant, Jiangsu, PRC, was utilized as support to prepare Fe/SAIER catalyst for the photo-assisted Fenton reaction. The exchange equivalent of the SAIER was about 5 mM/g dry resin. The SAIER was exchanged with 0.2 N $FeCl_3 \cdot 6H_2O$ solution at room temperature for 30 min under stirring, then washed with deionized water until the pH value of the washed water reached 6–7. The obtained Fe/SAIER catalyst was dried at 60 °C overnight. To determine the Fe mass concentration of the Fe/SAIER catalyst, the Fe/SAIER was extracted by concentrated HCl solution (37 wt.%). Then, the resulting solution was subsequently diluted and the Fe mass concentration in the solution was determined using an Induced Coupled Plasma (ICP) (Model: Perkin Elmer Optima 3000 XL). The surface chemical composition analyses were performed on a PHI 5600 spectrometer. The take-off angle used was 45°. The surface chemical compositions of the Fe/SAIER catalyst were determined by measuring the peak areas of the detected elements. After the peak areas were determined, the atomic concentration of various elements on the surface of the catalyst could be calculated using the known factors. When the binding energy was studied, the binding energy of C1s was shifted to 284.8 eV as the reference.

The photo-catalytic activity of the Fe/SAIER catalyst was investigated in the degradation of salicylic acid in water in the presence of UVC light and H_2O_2 in a batch photo-reactor. The reactor was cylindrical with two UV lamps (Philips, 8 W 254 nm) inserted in the center except specified. To suspend the Fe/SAIER catalyst in the reactor, compressed air was bubbled from the bottom to the top with a flow rate of 1.5 L/min. The total volume of the solution was 1.8 L. The photo-assisted Fenton reaction was performed at room temperature. The starting point of the reaction was defined as the time when the UV light was turned on and a certain amount of H_2O_2 was added to the photo-reactor.

The concentration of salicylic acid in water was determined by HPLC (HP Model, series II 1090). In addition, to determine the Fe leaching out from the Fe/SAIER catalyst, the Fe concentration in the solution was measured by ICP.

3. Results and discussion

3.1. Characterization of Fe/SAIER catalyst

The Fe/SAIER catalyst prepared with 0.2 N $FeCl_3 \cdot 6H_2O$ solution was first characterized with XPS and the survey spectra of the SAIER before and after ion exchange are shown in Fig. 1(a) and (b), respectively. From the spectra, several important facts are revealed. Firstly, before ion exchange reaction, only Na, S, O, and C were detected, indicating that the SAIER we used is a Na-type SAIER. Secondly, after ion exchange reaction, in addition to Na, S, O, and C, another element, Fe was also detected, implying that some Na ions were replaced by Fe ions, and this was confirmed by the decrease in the intensity of Na^+ peak in Fig. 1(b). Thirdly, the intensity of Na ions in Fig. 2(b) is not zero, illustrating that Fe ions did not replace all Na^+ ions on the surface of SAIER through ion exchange reaction. Finally, it can be deduced that the Fe ions were anchored by $-SO_3^-$ groups on the surface of the SAIER. These Fe ions could be used to accelerate photo-assisted Fenton reaction according to published results [14–17].

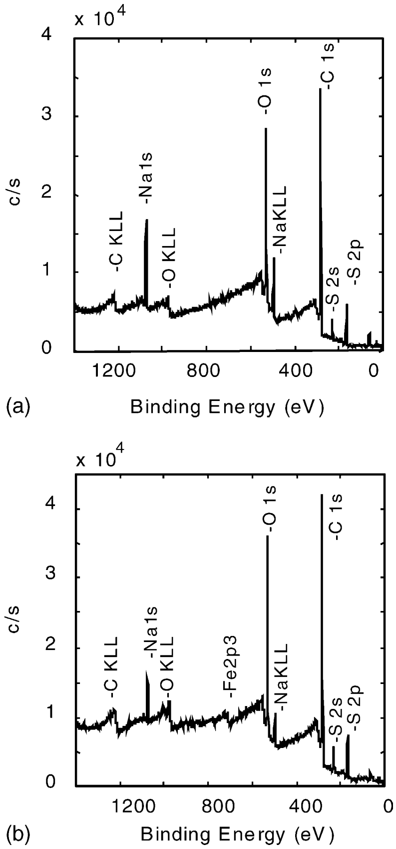

Fig. 1. XPS survey spectra of the SAIER (a) before and (b) after ion exchange.

Apart from determining the presence of Fe ions on the surface of the SAIER, the oxidation state of the exchanged Fe on the surface of the Fe/SAIER catalyst was also determined by XPS and the result is presented in Fig. 2(a). Using the binding energy of C1s at 284.8 eV as a reference, the binding energy of the Fe ions on the surface of the SAIER was determined to be 712.3 eV, strongly suggesting that the Fe ions detected were mainly Fe^{3+}. The binding energy of Fe^{3+} detected is higher than that of Fe^{3+} in $FeCl_3$ [19]. The increase in the binding energy of Fe^{3+} is owing to the fact that the sulfonate group on the surface of the SAIER has stronger electrostatic attractive force than the Cl^-. Fur-

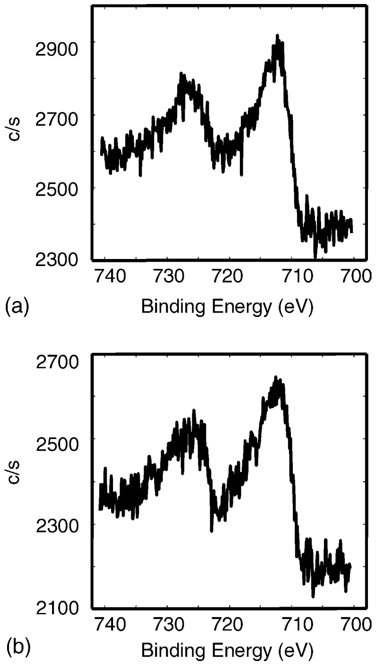

Fig. 2. XPS spectra of the Fe 2p3/2 region for the Fe/SAIER catalyst (a) before and (b) 120 min photo-assisted Fenton reaction.

thermore, neither Fe with zero oxidation state nor Fe^{2+} was detected.

The Fe mass concentration with respect to the total weight of the Fe/SAIER catalyst was determined to be 6.75 wt.% by ICP.

3.2. Mechanism of Fe/SAIER catalyst for photo-Fenton reaction

After characterization of the Fe/SAIER catalyst, the evaluation of the catalyst was performed in the degradation of salicylic acid in the presence of 2 × 8 W UVC. To reveal the mechanism of the Fe/SAIER catalyst under the UVC light, the degradation kinetics of salicylic acid under different control conditions were measured and the results are presented in Fig. 3. As can be seen clearly from the plot, with 2 × 8 W UVC and 10 g Fe/SAIER but without H_2O_2, the degradation of salicylic acid is negligible, indicating that direct photolysis cannot lead to significant degradation of salicylic acid. In addition, the result also implies that the adsorption of salicylic acid on the catalyst is insignificant. With 2 × 8 W UVC and H_2O_2 but without any catalyst, the degradation of salicylic acid is obvious, while with 2 × 8 W UVC and H_2O_2, and 10 g Fe/SAIER catalyst, the degradation of salicylic acid was significantly accelerated, and the concentration of salicylic acid in solution was reduced to zero in 120 min. However, we still cannot determine whether the degradation of the salicylic acid is caused by the Fe/SAIER catalyst or by the presence of Fe ions in the solution due to Fe ions leaching out from the catalyst surface.

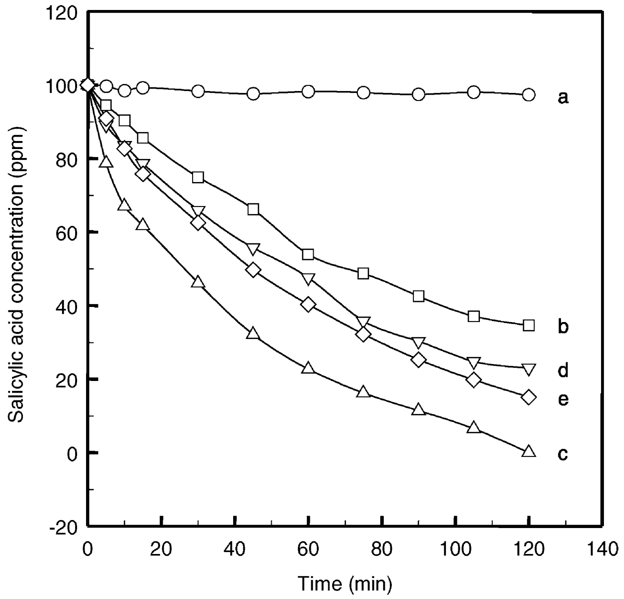

Fig. 3. Degradation of 100 mg/l salicylic acid under different reaction conditions. (a) pH 2.0; 2 × 8 W UVC; 10 g Fe/SAIER; (b) pH 2.0; 2 × 8 W UVC; $[H_2O_2]/[SA]$ molar ratio = 20; without Fe/SAIER; (c) pH 2.0; 2 × 8 W UVC; $[H_2O_2]/[SA]$ molar ratio = 20; 10 g Fe/SAIER; (d) pH 2.0; 2 × 8 W UVC; $[H_2O_2]/[SA]$ molar ratio = 20; 1 mg/l Fe^{3+}; and (e) pH 2.0; 2 × 8 W UVC; $[H_2O_2]/[SA]$ molar ratio = 20; 2 mg/l Fe^{3+}.

To check whether the degradation of salicylic acid was achieved by the presence of the Fe^{3+} leaching out from the catalyst, the Fe concentration in the solution as a function of time was determined by ICP and the result is presented in Fig. 4. It is seen that a very small amount of Fe leaching from the catalyst (less than 1.5 mg/l) occurred, which is not significant with respect to the total Fe content on the catalyst.

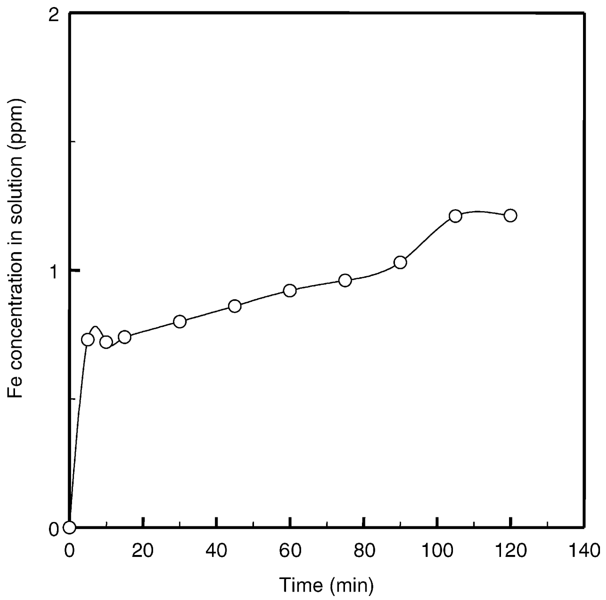

Fig. 4. Fe concentration in the solution as a function of time. Conditions: pH 2.0; 2 × 8 W UVC; $H_2O_2]/[SA]$ molar ratio = 20; 10 g Fe/SAIER catalyst.

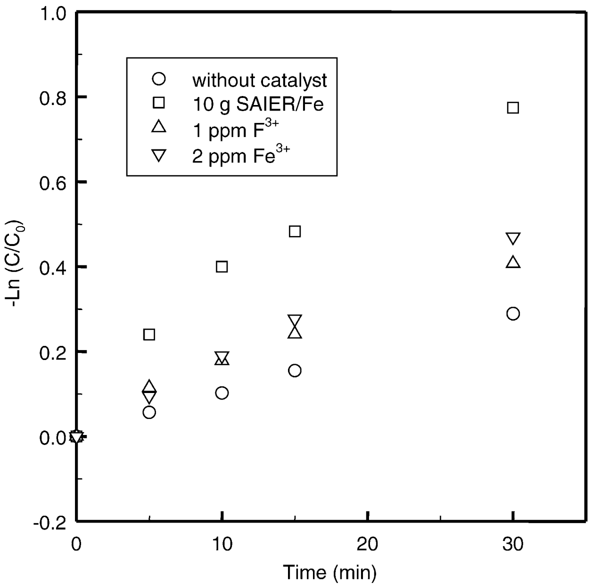

Fig. 5. $-\ln(C/C_0)$ vs. time. Conditions: pH 2.0; 2×8 W UVC; $[H_2O_2]/[SA]$ molar ratio = 20.

To qualitatively evaluate the contribution of the Fe/SAIER catalyst to the degradation of salicylic acid, two additional experiments, the degradation of salicylic acid in the presence of 1 and 2 mg/l Fe^{3+} in the solution, were conducted and the results are also presented in Fig. 3. By comparing the results shown in Fig. 3, it can be clearly deduced that the degradation of salicylic acid comes from three aspects. The first is from the oxidation of salicylic acid by OH radicals from direct photolysis of H_2O_2 ($UVC + H_2O_2$), the second is from the catalysis by the Fe/SAIER catalyst; and the last one is from the catalysis by the Fe^{3+} in the solution leached out from the catalyst.

To quantitatively study the contribution of the Fe/SAIER catalyst to the degradation of salicylic acid, using data shown in Fig. 3 in the first 30 min, we plotted $-\ln(C/C_0)$ versus time in Fig. 5, where C and C_0 are the salicylic acid concentrations at time = t and time = 0, respectively. As can be seen from the plot, in the first 15 min, $-\ln(C/C_0)$ versus t exhibits a reasonable good linear relationship. Assuming that the degradation of salicylic acid in the first 15 min obeys the first order kinetics, we fitted the data and obtained the reaction rate constant k under different conditions, as listed in Table 1. The reaction rate constant k clearly reveals that the combined effect of H_2O_2 and UVC, plus the presence of the Fe ions in solution accounts for about 50% of the one observed with the supported catalysts. The supported Fe/SAIER is responsible for half of the total degradation of salicylic acid.

Table 1
Reaction rate constant k under different conditions

Conditions	Reaction rate constant k
2 × 8 W UVC, $[H_2O_2]/[SA] = 20$, without catalyst	1.04×10^{-2}
2 × 8 W UVC, $[H_2O_2]/[SA] = 20$, 10 g Fe/SAIER	3.54×10^{-2}
2 × 8 W UVC, $[H_2O_2]/[SA] = 20$, 1 ppm Fe^{3+}	1.71×10^{-2}
2 × 8 W UVC, $[H_2O_2]/[SA] = 20$, 2 ppm Fe^{3+}	1.87×10^{-2}

The mechanism of Fe/SAIER as a heterogeneous catalyst under 2 × 8 W UVC can be expressed by the following equations:

$$\frac{Fe^{3+}}{SAIER} + h\nu \rightarrow \frac{Fe^{2+}}{SAIER} \tag{1}$$

$$\frac{Fe^{2+}}{SAIER} + H_2O_2 \rightarrow \frac{Fe^{3+}}{SAIER} + OH^- + {}^\bullet OH \tag{2}$$

$$\begin{aligned} {}^\bullet OH + HOC_4H_4COOH &\rightarrow \text{degraded products} \\ &\rightarrow CO_2 + H_2O \end{aligned} \tag{3}$$

It should be noted that two important experimental phenomena were observed. One is the color change of the solution and the other is the solution pH. After the reaction is started, the color of the solution changed quickly from clear to yellow, indicating that the degradation of salicylic acid occurred. The solution pH decreased about 0.1, implying that the pH does not change significantly during the reaction.

The oxidation state of Fe ions on the surface of the catalyst after 2 h reaction was also determined by XPS and the result is presented in Fig. 2(b). A careful comparison between Fig. 2(a) and (b) indicates that significant change was observed in the oxidation state of Fe before and after the photo-assisted Fenton reaction. The binding energy of the Fe ions was determined to be 711.5 eV, which is smaller than that of the Fe ions on the surface of the catalyst before the reaction. The decrease in the binding energy is due to the fact that some Fe^{3+} ions were reduced to Fe^{2+} that has a smaller binding energy [19].

Quantitative surface chemical compositions of the Fe/SAIER catalyst before and after 2 h photo-assisted Fenton reaction were measured by XPS and the results are summarized in Table 2. As can be seen from the table, after the reaction, the surface concentrations of Na, S, and Fe decrease while the surface concentrations of C and O increase. This phenomenon may be due to the fact that some reaction intermediates containing C, H, and O, remained on the surface of the Fe/SAIER catalyst because the surface of the catalyst becomes dark after the reaction.

Table 2
Surface chemical compositions (at.%) of the Fe/SAIER catalyst determined by XPS

Element	Before reaction	After 2 h reaction
Na (1s)	2.47	1.51
Fe (2p)	1.25	1.16
S (2p)	7.37	6.81
O (1s)	21.70	22.67
C (1s)	67.21	67.85

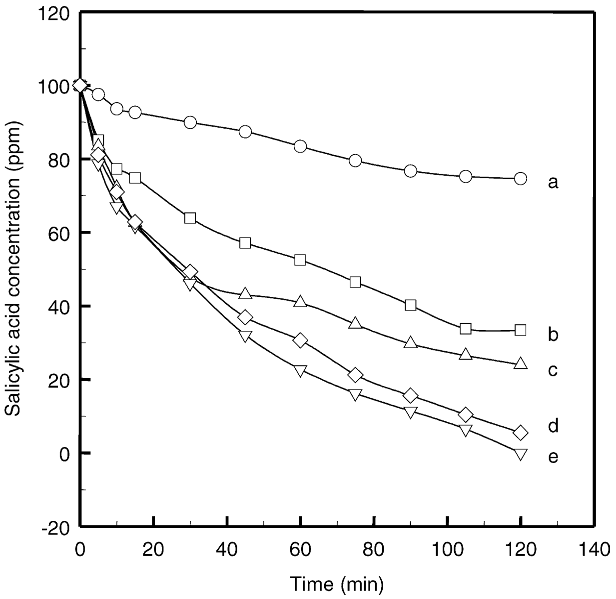

Fig. 6. Effect of $[H_2O_2]/[SA]$ molar ratio on the degradation of salicylic acid. Conditions: pH 2.0; 2 × 8 W UVC; 10 g Fe/SAIER catalyst; $[H_2O_2]/[SA]$ molar ratio = (a) 5; (b) 10; (c) 15; (d) 20; and (e) 40.

After determination of the mechanism of the Fe/SAIER catalyst, it is necessary to investigate the effects of some important variables such as H_2O_2 concentration, solution pH, UV light, on the degradation of salicylic acid since previous studies have revealed that such variables can significantly influence the degradation of organic compounds by photo-assisted Fenton reaction [2–7,13,15]. In the following subsections, the effects of various variables on the degradation of salicylic acid are discussed in detail.

3.3. Effect of $[H_2O_2]/[SA]$ molar ratio

Fig. 6 shows a plot of salicylic acid concentration as a function of time under different $[H_2O_2]/[SA]$ molar ratios. Clearly, with increasing the ratio, the degradation kinetics of salicylic acid was significantly accelerated until the molar ratio attained 20. When the ratio is larger than 20, no further acceleration of salicylic acid degradation but a slight decrease was observed. The results are very similar to those observed in the degradation of Orange II as reported by Fernandez et al [15]. It is believed that this phenomenon is owing to the so-called scavenging effect when utilizing a high H_2O_2 concentration on the further generation of hydroxyl radicals in aqueous solution as indicated by the following reaction [20,21].

$$H_2O_2 + {}^{\bullet}OH \rightarrow {}^{\bullet}HO_2 + H_2O \qquad (4)$$

Based on the results shown in Fig. 6, during the degradation of salicylic acid under 2 × 8 W UVC, the critical $[H_2O_2]/[SA]$ molar ratio, at which the Fe/SAIER catalyst exhibited the highest efficiency, was determined to be about 20.

Suppose salicylic acid can be completely mineralized into CO_2 and H_2O as indicated by the equation as below:

$$C_8H_6O_3 + 14\,H_2O_2 \rightarrow 8CO_2 + 17H_2O \qquad (5)$$

Theoretically, at least 14 mol H_2O_2 are needed to completely mineralize 1 mol salicylic acid into CO_2 and H_2O. In our experiment, the critical $[H_2O_2]/[SA]$ molar ratio is larger than 14, indicating that adding suitable excess amount of H_2O_2 in the reaction system can effectively enhance the degradation of salicylic acid.

3.4. Effect of solution pH

The effect of solution pH on the homogeneous photo-assisted Fenton reaction has been studied extensively. The optimum pH was determined to be about 3.0. This is due to the fact that at this pH, approximately half of Fe(III) is present as Fe^{3+} ions and half as $Fe(OH)^{2+}$ ions, which are the photo-active species. In addition, at a lower pH, the scavenging effect of ${}^{\bullet}OH$ by H^+ is important while at a higher pH, the formation of ${}^{\bullet}OH$ becomes slow owing to the hydrolysis of Fe^{2+} and the precipitation of FeOOH in the solution.

Fig. 7 depicts the salicylic acid concentration as a function of time when the solution pH is varied. It is very clear that similar to the homogeneous photo-assisted Fenton reaction, the solution pH can significantly influence the degradation of salicylic acid. For example, at pH 2.0, it needs about 120 min for the salicylic acid concentration to become zero while at pH 3.1, only 75 min are needed. The highest efficiency of the Fe/SAIER catalyst was observed at about pH 3, which is also the optimal pH for homogeneous photo-assisted Fenton reaction. The results at pH 3.1 and pH 3.7 are almost

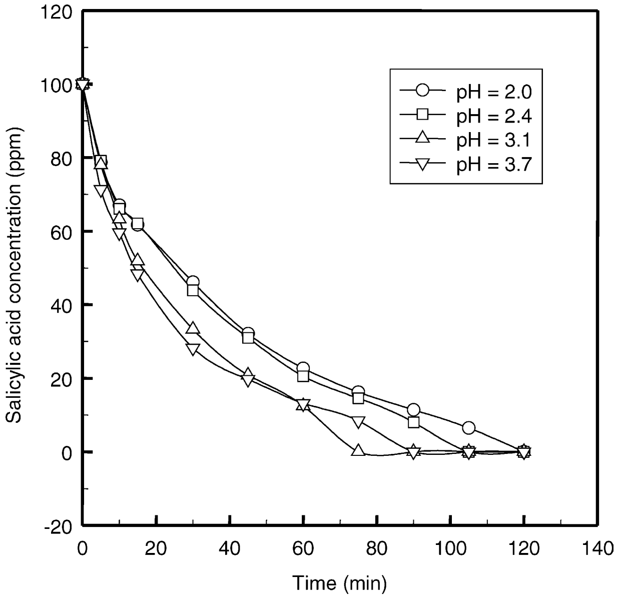

Fig. 7. Effect of solution pH on the degradation of salicylic acid. Conditions: 2 × 8 W UVC; $[H_2O_2]/[SA]$ molar ratio = 20; 10 g Fe/SAIER catalyst.

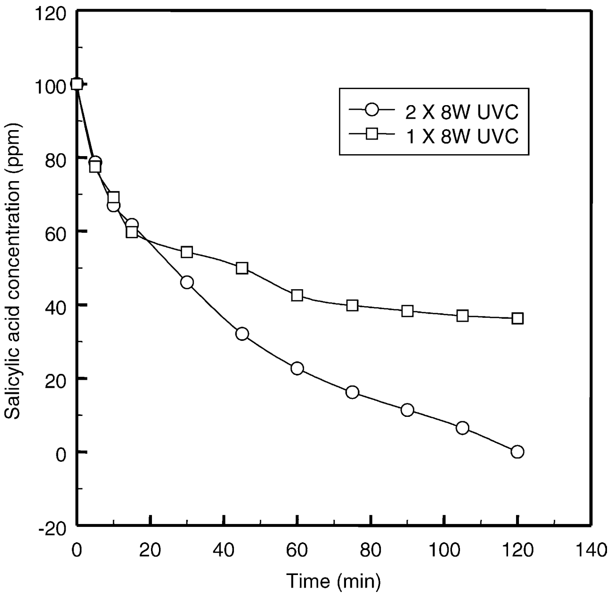

Fig. 8. Effect of UVC power on the degradation of salicylic acid. Conditions: pH 2.0; $[H_2O_2]/[SA]$ molar ratio = 20; 10 g Fe/SAIER catalyst.

the same. This may be due to the acidic product which lowers the solution pH to near 3 quickly from the initial pH of 3.7. These observations agree well with those observed for the photo-assisted Fenton degradation of Orange II when Fe/Nafion membrane was used as the catalyst in the degradation of Orange II [14,15].

3.5. *Effect of UVC light power*

The effect of UV light power on the degradation of organic compounds in both homogeneous and heterogeneous photo-assisted Fenton process has been investigated [13,15]. For example, Kang et al. reported that application of a very high level of UV light power in the process of $H_2O_2/UV/Fe^{2+}$ as a strategy to accelerate the initial removal of COD and color for the studied wastewater is not economically efficient. In addition, to gain benefit from increasing UV light power in the process of $H_2O_2/UV/Fe^{2+}$, a considerable time appears to be prerequisite, allowing enough time for the photo-reduction of ferric ions to occur [13].

The effect of the UVC light power on the degradation of the salicylic acid in the presence of 10 g Fe/SAIER catalyst and H_2O_2 was studied and is presented in Fig. 8. It is clearly seen that when two 8 W UVC lamps were replaced by only one 8 W UVC lamp, the degradation of salicylic acid was remarkably decreased. This is due to the fact that when the UVC power decreases 50%, the number of $^\bullet OH$ radicals generated from the photo-Fenton reaction also decreases significantly. Accordingly, the degradation of salicylic acid was decreased remarkably. These results agree well with published data [13].

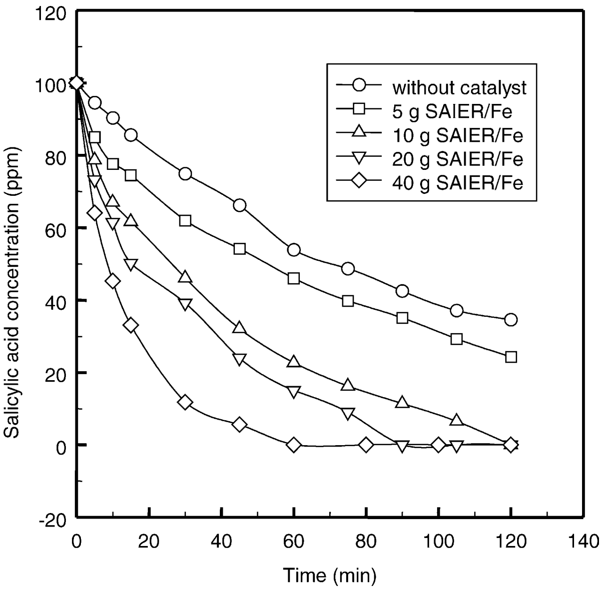

Fig. 9. Effect of Fe/SAIER catalyst loading on the degradation of salicylic acid. Conditions: pH 2.0; 2 × 8 W UVC; $[H_2O_2]/[SA]$ molar ratio = 20.

3.6. *Effect of Fe/SAIER catalyst loading*

Effect of the catalyst loading on the degradation of organic aromatics compounds has been studied extensively. Normally, a high catalyst loading results in a faster degradation of the compounds until a saturated catalyst loading is achieved. Fig. 9 presents a plot of salicylic acid concentration as a function of time when different Fe/SAIER catalyst loadings are used. As can be seen from the plot, the more Fe/SAIER catalyst was used, the faster the salicylic acid was degraded completely. This is because at a higher catalyst loading, more Fe ions are involved in the photo-assisted Fenton reaction, resulting in more $^\bullet OH$ radicals in the solution. Accordingly, the complete degradation of salicylic acid needs less time at a higher catalyst loading.

On the other hand, in our case, only when the catalyst loading is larger than 10 g Fe/SAIER catalyst/1.8 L 100 mg/l SA solution, significant degradation of SA can occur. In addition, the saturated catalyst loading should be equal to or larger than 40 g Fe/SAIER catalyst/1.8 L 100 mg/l SA solution on the basis of the results shown in Fig. 9.

3.7. *Effect of initial salicylic acid concentration*

To investigate the effect of the initial salicylic acid concentration on the salicylic acid degradation kinetics, two initial salicylic acid concentrations were used while the $[H_2O_2]/[SA]$ molar ratio used was kept the same. Fig. 10 depicts a plot of normalized salicylic acid concentration as a function of time for two different initial salicylic acid concentrations. Obviously, at the same experimental conditions, the salicylic acid with a lower initial concentra-

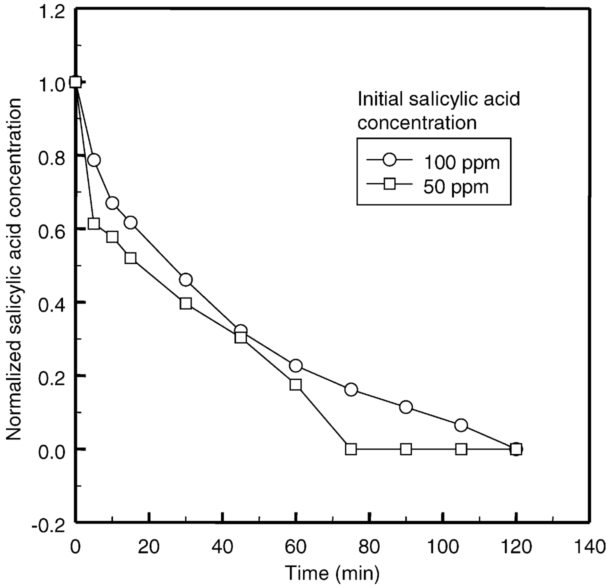

Fig. 10. Effect of initial salicylic acid concentration on the degradation of salicylic acid. Conditions: pH 2.0; 2 × 8 W UVC; $[H_2O_2]/[SA]$ molar ratio = 20; 10 g Fe/SAIER catalyst.

tion is degraded faster and needs less time to be degraded completely as expected.

4. Conclusions

The use of Fe^{3+} ions supported on the strongly acidic ion exchange resin (Fe/SAIER) as a heterogeneous catalyst in the degradation of salicylic acid in the presence of 2 × 8 W UVC (254 nm) and H_2O_2 is studied. The X-ray photoelectron spectroscopy (XPS) results reveal that the Fe ions are indeed anchored by the sulfonate groups on the surface of the SAIER and are identified as Fe^{3+}. Our results also indicate that the Fe/SAIER catalyst can significantly enhance the degradation rate of salicylic acid in the presence of 2 × 8 W UVC without remarkable Fe leaching to the solution, implying that the catalyst has photo-catalytic activity. The main factors that can significantly influence the degradation of salicylic acid were found to be $[H_2O_2]/[SA]$ molar ratio, solution pH, UVC light power, and catalyst loading. The critical $[H_2O_2]/[SA]$ molar ratio was determined to be 20, at which the Fe/SAIER catalyst exhibits the best efficiency. The optimal solution pH was determined to be about 3, which is almost the same as that observed in the homogeneous photo-assisted Fenton reaction. An increase in UVC light power or Fe/SAIER catalyst loading results in a faster degradation of salicylic acid.

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