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Effect of hydrogen peroxide on aniline oxidation by electro-Fenton and fluidized-bed Fenton processes

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ABSTRACT

In this study, the electro-Fenton and fluidized-bed Fenton processes under the given conditions were used to oxidize aniline. Factors such as feeding mode and concentration of the hydrogen peroxide were explored.

Results showed that the feeding mode of H_2O_2 did not significantly affect the aniline oxidation in the electro-Fenton process. However, the aniline oxidation slightly decreased with the two-step addition of H_2O_2 in the fluidized-bed Fenton process. Presumably the decline of remaining Fe^{2+} led to destitute hydrogen radicals from the Fe^{2+}-catalyzed H_2O_2. In addition, the removal efficiency of aniline was maintained at a maximum as H_2O_2 concentration was higher than 0.04 M in the electro-Fenton process. Meanwhile, the almost exhausted H_2O_2 would increase the amount of Fe^{2+} in the solution for the electro-Fenton process. This is because the Fe^{2+} is regenerated through the reduction of Fe^{3+} on the cathode. The electro-Fenton process has a stronger oxidative ability with regard to the production of the oxalic acid than fluidized-bed Fenton process which was attributed to a higher consumption of H_2O_2. Therefore, in the aspect of H_2O_2 depletion, the mineralization efficiency of the fluidized-bed Fenton process was higher than that of the electro-Fenton process.

1. Introduction

Advanced oxidation processes (AOPs) can be used to destroy hazardous and refractory organic pollutants. AOPs are defined as the oxidation processes in which the hydroxyl radicals (•OH) are derived in sufficient quantity to effect wastewater treatment. The Fenton process is an AOP, which involves a catalytic reaction between ferrous ion (Fe^{2+}) and hydrogen peroxide (H_2O_2). The advantages of the Fenton process are a low capital cost, easy operation, and non-toxic by-products. In the last few years, Fenton processing has received considerable attention [1–5]. However, the Fenton process produces a huge amount of ferric hydroxide ($Fe(OH)_3$) sludge, which requires additional separation and disposal. The disadvantages limit the further application of the Fenton process in treating wastewater. Therefore, the electro-Fenton and fluidized-bed Fenton processes have been developed to reduce the production of iron sludge.

During the Fenton process, the hydroxyl radicals were derived from the ferrous catalyzed H_2O_2 [6].

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

The reaction between •OH and ferrous ions would lead to the formation of ferric ions and other radicals, as shown below [7–9]:

$$Fe^{3+} + H_2O_2 \rightarrow Fe^{2+} + H^+ + HO_2\bullet \quad (2)$$

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

$$\bullet OH + H_2O_2 \rightarrow H_2O + HO_2\bullet \quad (4)$$

$$\bullet OH + \bullet OH \rightarrow H_2O_2 \quad (5)$$

According to the above reactions, the hydroxyl radicals from depleted H_2O_2, therefore, were used for oxidation of organic substances. In general, a high concentration of H_2O_2 can improve the organo-oxidation to form low carbon chemicals. Moon et al. [10] mentioned that the oxidation of polyaniline organic substances could be enhanced by increasing the concentration of H_2O_2, as the H_2O_2 was conditioned at low concentrations. The authors suggested that the optimum $[H_2O_2]_{initial}$ for polyaniline-oxidation was 0.05 M in the $FeCl_3$–H_2O_2 system. As the $[H_2O_2]_{initial}$ further increased, the polyaniline-oxidation would then decrease. However, at a higher dosage of H_2O_2, the polyaniline-oxidation was prohibited due to the formation of a large amount of $HO_2\bullet$. Mohanty and Wei [11] and Zhang et al. [12] used the Fenton process for degradation of toxic organics. The results suggested that H_2O_2 feeding mode had significant effects on removal efficiency. Consequently, both the concentration and feeding mode of hydro-

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gen peroxide play an important role in the Fenton reaction. The electrode material plays an important role in electro-Fenton process. The RuO_2 metallic oxide electrodes undoubtedly have a high stability; however, a pure RuO_2 electrode is too expensive for technological applications. Therefore, a binary electrode was used to overcome this problem. RuO_2 and IrO_2 are the best metallic oxide electrode materials. Recently RuO_2/IrO_2 was applied successfully as an anode in the electro-Fenton process [13,14]. Hence, the titanium net coated with RuO_2/IrO_2 was used as an anode in the study.

Aniline ($C_6H_5NH_2$) is an organic compound consisting of a benzene ring and has been categorized as hazardous waste. Aniline is widely used in various industries such as dye manufacturing, pesticides, rubber chemicals, and pharmaceuticals. The toxic ingredients include human carcinogens, based on sufficient evidence of carcinogenicity in experimental animals [15]. The Fenton technologies such as Fenton, photo-Fenton, electro-Fenton and fluidized-bed Fenton processes have been already investigated for degradation of aniline in the previous work [3,16–18]. Electro-Fenton processes could continuously regenerate Fe^{2+} at the cathode, which also minimized sludge production. Besides, the feasibility of overall mineralization has low cost if the operation parameters are optimized [19]. However, the comparison of aniline oxidation between the electro-Fenton and fluidized-bed Fenton processes has never been reported. Therefore, this study researched the effects of the feeding mode and concentration of hydrogen peroxide on the aniline oxidation efficiencies and mineralization.

2. Materials and methods

2.1. Chemicals and analytical method

All chemical substances used in the experiment were of reagent grade. All the solutions were prepared from deionized water and were conducted at room temperature. Quartz (SiO_2) powder which ranged between 0.42 and 0.84 mm in diameter was used as the carriers. The measured point of zero charge (PZC) was 6.3.

The 1.0 ml samples taken at selected intervals were immediately injected into the tubes containing the NaOH solution to stop any further reaction and were then filtered through 0.45 μm syringe micro-filters to separate the precipitated iron from the solution. Subsequently, the residual aniline solution was analyzed by an HP4890 gas chromatograph equipped with a flame ionization detector and an HP-5 column (0.53 mm diameter × 15 m length). The temperature of the chromatographic column was initially set at 85 °C for 3 min, and then increased to 200 °C by 65 °C min^{-1}. The temperature was maintained at 200 °C for the final 2 min. The injector and detector temperatures were 250 °C, and the flow rate of nitrogen carrier gas was 14.6 ml/min. The concentration of ferrous ions was determined by light absorbance measurement at 510 nm after being complexed with 1,10-phenanthroline using a UV–vis spectrophotometer [20]. The concentration of H_2O_2 was determined by standard iodometric method where potassium iodide with $Na_2S_2O_3$ solution was used as a reactant [21]. To avoid concentration of H_2O_2 affect COD measurement. For COD determination, the samples were left overnight to withdraw the residual hydrogen peroxide that might remain in the sample solution. The TOC and COD of the water sample were measured by a Shimadzu TOC-VCPH and by closed-reflux titrimetric method, respectively, according to Standard Methods [20].

2.2. Electro-Fenton and fluidized-bed Fenton reactor

An acrylic reactor measuring 15 × 21 × 20 cm^3 with 5 L of working volume was used for the electro-Fenton experiments as shown in Fig. 1(a). The electro-Fenton anodes were special nets made from RuO_2/IrO_2-coated titanium metal (DSA—dimensionally stable anodes) and the cathodes were stainless-steel nets. The working area on the cathode and anode were 900 and 600 cm^2, respectively. All electrodes were connected to a DC power supply which, when needed, constantly discharged current at 4 A into the mixture. Two mixers were installed to provide complete agitation in the reactor. A 1.35 L fluidized-bed reactor was used in this study as shown in Fig. 1(b). The fluidized-bed Fenton reactor consisted of inlet, outlet, and recirculation compartments. The expanded height of the SiO_2 carrier suspension can be controlled by adjusting the internal circulation flow rate.

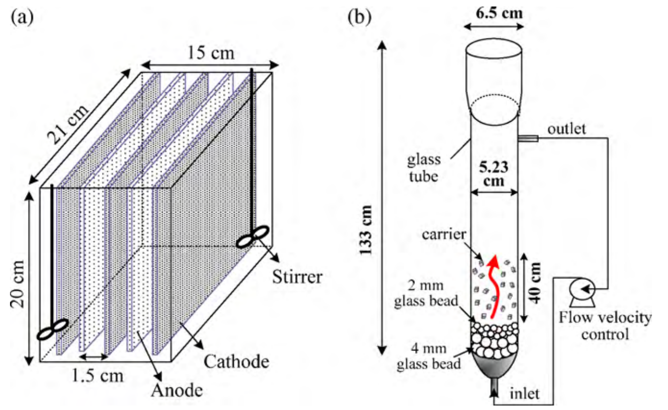

Fig. 1. The schematic diagram of (a) electro-Fenton reactor and (b) fluidized-bed Fenton reactor.

There were two methods, one-step and two-step, for H_2O_2 addition. In the one-step method the H_2O_2 addition was about 58 mM. In the two-step method the H_2O_2 addition was 46.5, 43 mM at 0 min and 11.5, 15 mM at 10 min, respectively, in the electro-Fenton and fluidized-bed Fenton processes.

3. Results and discussion

3.1. Effect of hydrogen peroxide feeding mode

The current of 4 A was chosen from the authors' previous study [22] which found out that 4 A was the most appropriate current to remove this kind of chemical. A previous Fenton study [23] has shown that the optimum pH for Fenton oxidations was near 3. Therefore, about pH 3 was selected as experimental condition in these two Fenton processes. In another Fenton study [4], Fe^{2+} was the limiting component under the studied conditions, hence, 1.07×10^{-3} M Fe^{2+} was chosen in this study.

Figs. 2 and 3 compared the one-step and two-step addition with H_2O_2 employed in this study. The results have shown that aniline was rapidly degraded in the first 10 min and then slowed down until the reaction was over (Fig. 2(a)). The aniline oxidation for the system of one-step addition with H_2O_2 was similar to that for the two-step, which was about 95% after reacting for 60 min in the electro-Fenton process. In the fluidized-bed Fenton process, the aniline oxidation for the one-step addition with H_2O_2 was better than that for the two-step. However, the aniline oxidation of both feeding modes coincided, at about 80%, after reacting for 60 min. Lu et al. [9] reported the Fenton system underwent a two-stage reaction. The first stage was the reaction in the first 30 s in which the decomposition rate of dichlorvos was high, and there was a Fe^{2+}/H_2O_2 reaction. The second stage was a slower Fe^{3+}/H_2O_2 reaction taking place after 30 s. Fig. 2(a) shows the aniline decomposed rapidly in 2 min, and the decomposition gradually weaken between

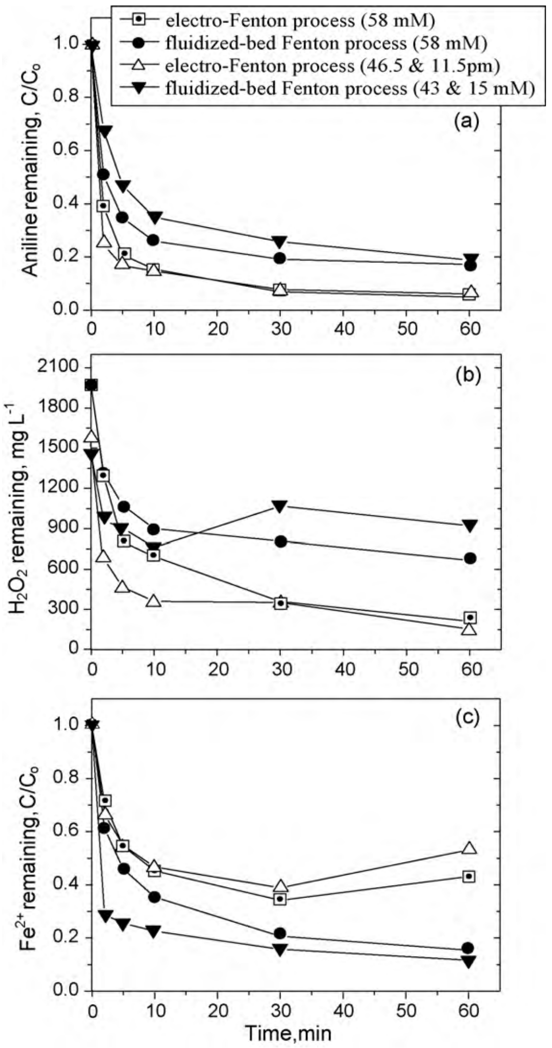

Fig. 2. Effect of H_2O_2 feeding mode on the (a) aniline, (b) H_2O_2 and (c) Fe^{2+} at the electro-Fenton and fluidized-bed Fenton processes. Initial conditions: 0.01 M aniline solution with 1.07×10^{-3} M Fe^{2+} at pH 3.2.

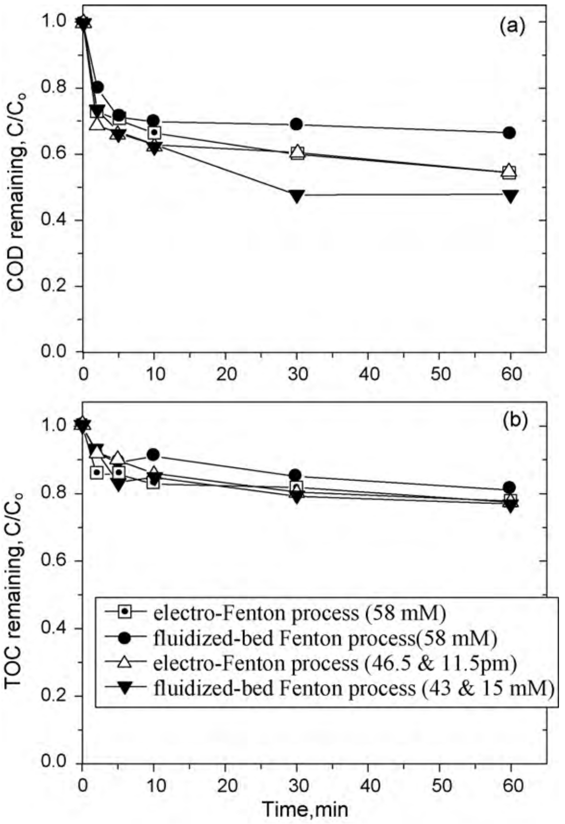

Fig. 3. Effect of H_2O_2 feeding mode on the (a) COD and (b) TOC at the electro-Fenton and fluidized-bed Fenton processes. Initial conditions: 0.01 M aniline solution with 1.07×10^{-3} M Fe^{2+} at pH 3.2.

2 and 30 min. The aniline remaining, however, was not substantially changed in the following 30 min. Therefore, the reaction in the first 2 min was classified as the first stage and from 2 to 30 min was classified as the second stage, as described by the aniline initial average rate (M min^{-1}) and reaction rate constant (K), respectively. As can be seen in Table 1, the initial average rate and rate constant (K) in the electro-Fenton process were higher than that of the fluidized-bed Fenton process, indicating that the aniline oxidation was better in the electro-Fenton process. A higher residual H_2O_2 was detected in the fluidized-bed Fenton process as shown in Fig. 2(b). The hydroxyl radicals poorly derived in the beginning of reaction in the fluidized-bed Fenton process, which was anticipated as being the reason for low aniline oxidation. On the other hand, low remaining H_2O_2 by the two-step addition with H_2O_2 in the fluidized-bed Fenton process, which eliminated the derivation of hydroxyl radicals, led to a decrease of initial average rate aniline oxidation (Table 1).

Table 1
Aniline oxidation rates and reaction rate constants at the electro-Fenton and fluidized-bed Fenton processes with H_2O_2 addition.

Process	H_2O_2	Initial average rate ($\times 10^{-3}$ M min^{-1}) (0–2 min)	K ($\times 10^{-2}$ min^{-1}) (2–30 min)
Electro-Fenton process	One step	3.06	5.3
	Two step	3.78	3.7
Fluidized-bed Fenton process	One step	2.47	2.9
	Two step	1.60	2.9

Experimental condition: 0.01 M aniline, 1.07×10^{-3} M Fe^{2+}, 74 g SiO_2/L, current 4 A, pH 3.2.

As shown in Fig. 2(c), H_2O_2 feeding mode did not significantly affect the remaining Fe^{2+} in the electro-Fenton process. However, the two-step addition with H_2O_2 has the lowest remaining Fe^{2+} in the fluidized-bed Fenton process. This implied that H_2O_2 decomposition cannot be efficiently catalyzed by Fe^{2+}, and, therefore, the residual curve of H_2O_2 lifted gradually after reacting for 10 min in the two-step addition with H_2O_2.

Fig. 3 shows the effect of the H_2O_2 feeding mode on the COD and TOC. Apparently, H_2O_2 feeding mode affects the removal efficiency of COD in the fluidized-bed Fenton process (Fig. 3(a)). The removal efficiency of COD in the two-step addition with H_2O_2 was better than that in the one-step. It can be derived from the occurrence of heterogeneous chemical oxidation (H_2O_2/iron oxide) in the two-step addition with H_2O_2 after a reaction of 10 min. The removal efficiency of COD in the one-step addition with H_2O_2 was similar to that in the two-step in the electro-Fenton process. On the other hand, the removal efficiency of TOC was not affected by the H_2O_2 feeding mode in both processes (Fig. 3(b)). Furthermore, aniline could be only slightly mineralized to about 19–25%.

3.2. Effect of hydrogen peroxide concentration

The effects of H_2O_2 concentrations on the electro-Fenton and fluidized-bed Fenton processes are shown in Figs. 4 and 5. The removal efficiency of aniline kept at 92–95% as H_2O_2 concen-

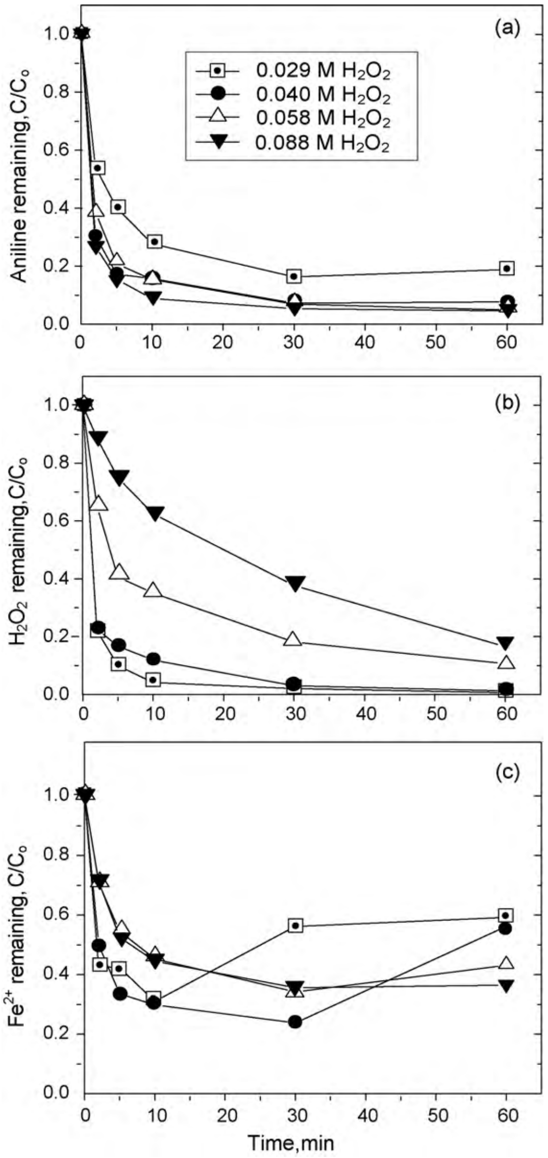

Fig. 4. Effect of H_2O_2 on the electro-Fenton process. Initial conditions: 0.01 M aniline solution with 1.07×10^{-3} M Fe^{2+} at pH 3.2.

tration was increased higher than 0.04 M in the electro-Fenton process (Fig. 4(a)). The removal efficiency of aniline in the fluidized-bed Fenton processes was increased by increasing the H_2O_2 concentration, when the H_2O_2 ranged from 0.029 to 0.088 M (Fig. 5(a)). The removal efficiency of aniline was about 96% at 0.088 M H_2O_2 after reacting for 60 min. When H_2O_2 concentration increased from 0.04 to 0.088 M, the aniline oxidation kept constant after reacting for 30 min in both processes; however, it was found that H_2O_2 continuously decomposed (Figs. 4(b) and 5(b)). This was due to the self-decay of H_2O_2, consumed by intermediates, and/or •OH scavenging effect of H_2O_2 (Eq. (4)). The almost exhausted H_2O_2 induced an increase in remaining Fe^{2+} in the solution, as H_2O_2 was less than 0.058 M after reacting for 10 min in the electro-Fenton process (Fig. 4(c)). Presumably, the result is because the Fe^{2+} is regenerated through the reduction of Fe^{3+} on the cathode. The increments of the Fe^{2+} remaining in the solution with a decrease in residual H_2O_2 did not come up in the fluidized-bed Fenton process, presumably due to iron from the aqueous phase via crystallization onto the surface of SiO_2 carriers (Fig. 5(c)).

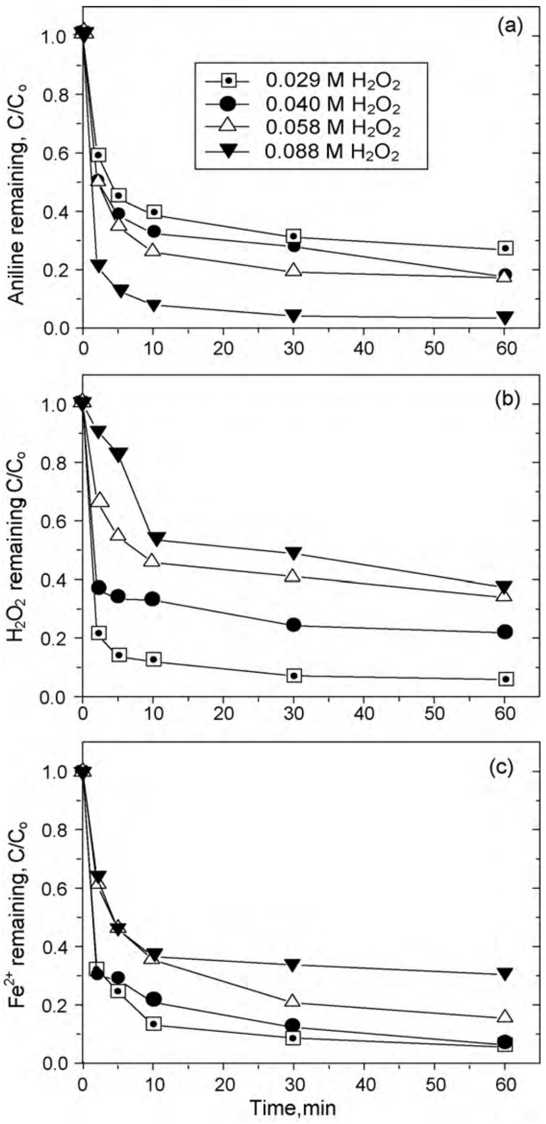

Fig. 5. Effect of H_2O_2 on the fluidized-bed Fenton process. Initial conditions: 0.01 M aniline solution with 1.07×10^{-3} M Fe^{2+} at pH 3.2.

3.3. Mineralization of aniline in different processes

The tendency of TOC in 0.01 M aniline solution with 1.07×10^{-3} M Fe^{2+} by the electro-Fenton and fluidized-bed Fenton processes for various H_2O_2 concentrations is depicted in Fig. 6. A gradual decay in pollutant degradation rate with increasing treatment time can be observed in both processes (Fig. 6(a) and (b)). The removal ratio of the soluble TOC was faster at 0.08 M H_2O_2 in the electro-Fenton process. However, the degradation of remaining contaminants continued slowly. The H_2O_2 concentrations show no distinct effect on the removal ratio of TOC in the fluidized-bed Fenton process. The removal ratio of TOC in the electro-Fenton and fluidized-bed Fenton processes after reacting for 60 min was about 20–30% and 18–35%, respectively.

The mineralization efficiency for each process after a given time *t* was defined as follows:

$$\text{Mineralization efficiency (\%)}: \quad \frac{\Delta[\text{TOC}]}{\Delta[\text{H}_2\text{O}_2]} = \left(\frac{\text{TOC}_0 - \text{TOC}_\text{f}}{H_0 - H_\text{f}}\right) \times 100 \qquad (6)$$

TOC_0 and TOC_f are the initial and final (after 60 min of reaction) TOC concentration (M), respectively. H_0 is the initial H_2O_2 concentration and H_f is the final H_2O_2 concentration (M) (after 60 min of reaction).

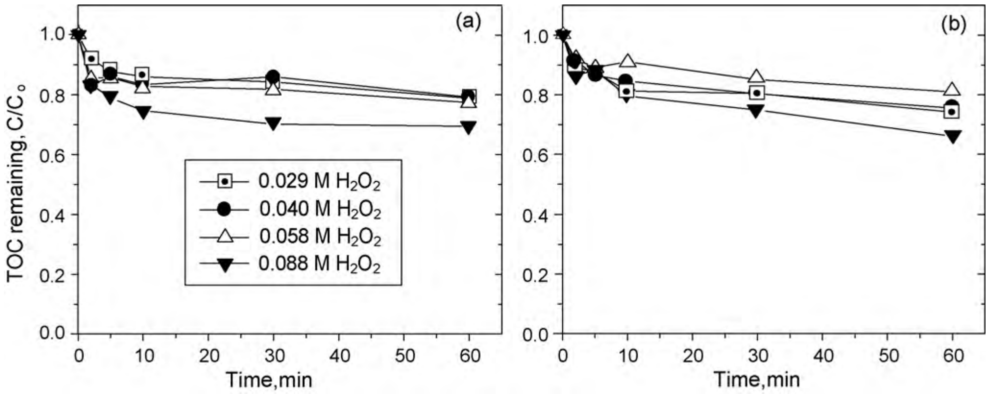

Fig. 6. Effect of H_2O_2 concentration on TOC remaining for the (a) electro-Fenton and (b) fluidized-bed Fenton processes. Initial conditions: 0.01 M aniline solution with 1.07×10^{-3} M Fe^{2+} at pH 3.2.

The results show that mineralization efficiency in the fluidized-bed Fenton process was higher than that of the electro-Fenton process, as H_2O_2 ranged from 0.029 to 0.088 M (Fig. 7). The mineralization efficiency in the electro-Fenton process was decreased by increasing the concentration of H_2O_2. In addition, mineralization efficiency reached a minimum at 0.058 M H_2O_2 in the fluidized-bed Fenton process.

In the electro-Fenton and fluidized-bed Fenton processes, the aniline oxidation was 80–95% and 70–97%, respectively, as shown in Figs. 4(a) and 5(a). However, Fig. 6(a) and (b) shows that removal ratio of TOC was only 20–30% and 18–35%, respectively. This result implies that the aniline was not completely oxidized to carbon dioxide during such a period but transformed into some intermediates, which are similar to aniline itself. The degradation of aromatic compounds often leads to formation of intermediates such as glyoxylic, maleic, oxalic acid, acetic and formic acids [24–26]. Furthermore, oxalic acid is the major intermediate of aromatic compound in the last degradation steps before total conversion to CO_2 [27,28]. The oxalic acid was detected in the degradation solution in order to further realize the oxidative ability of the electro-Fenton and fluidized-bed Fenton processes. Fig. 8 reveals that the fluidized-bed Fenton process has a weaker oxidative ability with regard to producing oxalic acid due to the removal efficiency of aniline in the electro-Fenton process is better than that of the fluidized-bed Fenton.

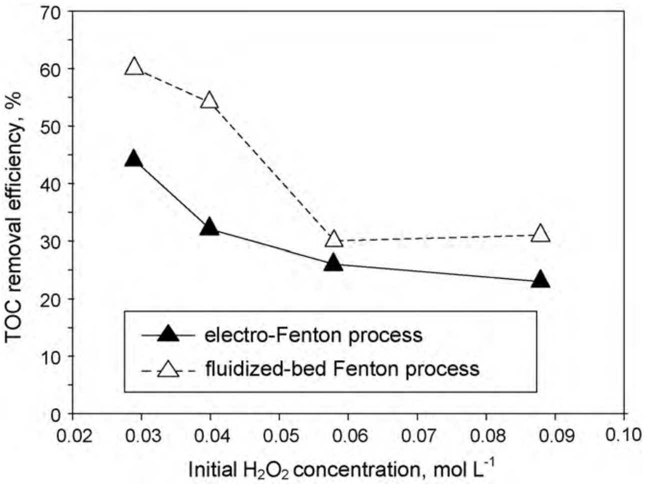

Fig. 7. Mineralization efficiency of electro-Fenton and fluidized-bed Fenton processes.

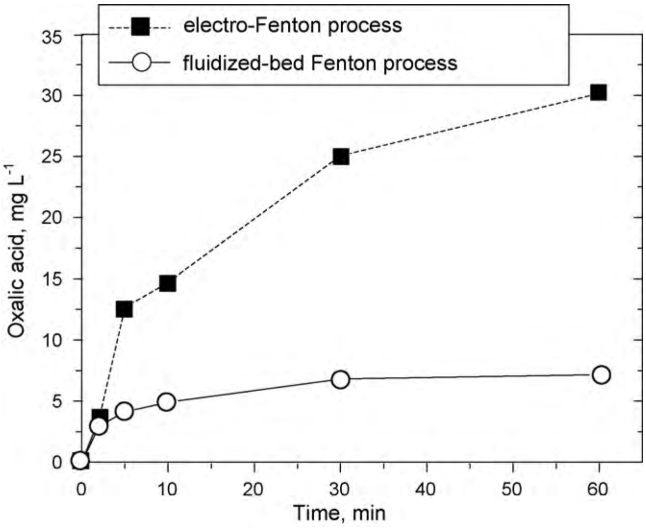

Fig. 8. Intermediate production of aniline degradation by the electro-Fenton and fluidized-bed Fenton processes. Initial conditions: 0.01 M aniline solution with 1.07×10^{-3} M Fe^{2+} at pH 3.2, 74.1 g SiO_2/L and current 4 A.

4. Conclusions

In this study, the oxidation of aniline was investigated by using the electro-Fenton and fluidized-bed Fenton processes. The results showed that hydrogen peroxide feeding mode had no significant influence on the aniline oxidation in the electro-Fenton process. However, the aniline oxidation was slightly decreased by the two-step addition with hydrogen peroxide in the fluidized-bed Fenton process. Presumably, elimination of the remaining Fe^{2+} led to a poor deviation of hydrogen radicals from the Fe^{2+}-catalyzed H_2O_2. The removal efficiency of aniline in the fluidized-bed Fenton process increased with an increase in the H_2O_2 concentration as the H_2O_2 ranged from 0.029 to 0.088 M. In addition, the removal efficiency of aniline kept constant as H_2O_2 conditioned higher than 0.04 M in the electro-Fenton process. Meanwhile, the almost exhausted H_2O_2 caused an increase in the remaining Fe^{2+} in the solution for the electro-Fenton process. This is because the Fe^{2+} is regenerated through the reduction of Fe^{3+} on the cathode. The electro-Fenton process has a stronger oxidative ability with regard to the production of the oxalic acid than the fluidized-bed Fenton process, which was attributed to higher consumption of H_2O_2. Therefore, in the aspect of H_2O_2 depletion, the mineralization efficiency of the fluidized-bed Fenton process was higher than that of the electro-Fenton process.

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