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Application of Fenton Processes for Degradation of Aniline

Chavalit Ratanatamskul[1], Nalinrut Masomboon[2] and Ming-Chun Lu[3]
[1]Department of Environmental Engineering, Chulalongkorn University,
[2] Department of Interdisciplinary programs in Environmental Science, Graduate School,
Chulalongkorn University,
[3]Department of Environmental Resources Management, Chia-Nan University of
Pharmacy and Science,
[1,2]Thailand
[3]Taiwan

1. Introduction

Fenton process is one of advanced oxidation processes (AOPs) which are considered as alternative methods for treatment of non-biodegradable and toxic organic compounds. Fenton process has been widely used in the treatment of persistent organic compounds in water. In general, the mechanism of Fenton reaction included the formation of hydroxyl free radicals, which has $E°$ of 2.8 V, that can oxidize and mineralize almost all the organic carbons to CO_2 and H_2O (Glaze et al., 1987), by the interaction of hydrogen peroxide with ferrous ions (Walling C., 1975). The Fenton's reagent is generally occurred in acidic medium between pH 2 -4 (Rodriguez et al., 2003). The advantage of Fenton process is the complete destruction of contaminants to harmless compounds, for instance, carbon dioxide and water (Neyen E. et al., 2003). However, its application has been limited due to the generation of the excess amount of ferric hydroxide sludge that requires additional separation process and disposal (Chang P.H., 2004). Therefore, electro-Fenton (EF) process is developed for minimizing the disadvantages of conventional Fenton process.

In the electro-Fenton method, the Fenton's regent was utilized to produce hydroxyl radical in the electrolytic cell, and ferrous ion was regenerated via the reduction of ferric ion on the cathode (Zhang et al., 2007). The regenerated ferrous ion will react with hydrogen peroxide and produce more hydroxyl radicals that can destroy the target compounds. However, the electro-Fenton reaction still faces several obstacles that must be overcome first such as the formation of ferric hydroxide sludge. Therefore, the new method which can promote the ferrous ion regeneration was focused in this part of experiment. The efficiency of pollutant removal and the reduction of ferric hydroxide sludge can be improved by using UV-radiation.

The photoelectro-Fenton process involves the additional irradiation of the solution with UVA light. Due to the generation of additional hydroxyl radical from the regeneration of ferrous ion and the reaction of hydrogen peroxide that reacted with UV light, so-called photoelectro-Fenton process (Brillas et al., 2000). Under UV-vis irradiation, the overall

efficiency of the process increases due mainly to the regeneration of ferrous ions and formation of additional hydroxyl radicals. UVA light can favor (1) the regeneration of ferrous ion with production of more amount of hydroxyl radical from photoreduction of $Fe(OH)^{2+}$, which is the predominant ferric ion species in acid medium (Sun and Pignatello, 1993) and (2) the photodecomposition of complexes of ferric ion with generated carboxylic acids (Flox et al., 2007). The maximum adsorption wavelength of $Fe(OH)^{2+}$ species is less than 360 nm, visible irradiation may not drive the reaction of equation (1). An interesting and potentially useful modification of the photoreduction reaction takes advantage of the photo-lability of Fe(III)-oxalate complexes, which has efficiency up to 500 nm (Pignatello et al., 2006).

Aniline has been used as a target chemical. It is one of the main pollutants in wastewater, mainly from the chemical processes of dye, rubber industry, pesticide manufacture and pharmaceutical sectors (Song et al., 2007). In this study, the effects of the initial concentration of 2,6-dimethylaniline concentration, Fe^{2+} concentration, H_2O_2 dosage on 2,6-dimethylaniline and COD removal efficiency were explored. Moreover, this experiment intended to provide important information on the kinetic study of various Fenton processes (Fenton process, electro-Fenton process and photoelectro-Fenton process) on the aniline degradation.

2. Materials and methods

All chemicals used in this study were prepared using de-ionized water from a Millipore system with a resistivity of 18.2 MΩ cm^{-1}. Aniline, perchloric acid, ferrous sulfate and hydrogen peroxide were purchased from MERCK. Sodium hydroxide was purchased from Riedel-da Haën. All the preparations and experiments were realized at the room temperature. Synthetic wastewater containing 1 mM of aniline was dissolved with de-ionized water and then adjusted pH with perchloric acid. After pH adjustment, a calculated amount of catalytic ferrous sulfate was added as the source of Fe^{2+} in this experiment. Then, the H_2O_2 was added into the reactor. The electrical current (for electro-Fenton process and photoelectro-Fenton process) and UVA lamps (for photoelectro-Fenton process) were delivered through out the experimental period. The samples taken at predetermined time intervals were immediately injected into tubes containing sodium hydroxide solution to quench the reaction by increasing the pH to 11(Anotai et al., 2006). The samples were then filtered with 0.45 μm to remove the precipitates formed, and kept for 12 hours in the refrigerator before chemical oxygen demand (COD) analysis was conducted. This work has been carried out to investigate the effect of the concentration of hydrogen peroxide on the COD value.

The samples were also analyzed for aniline and COD. Aniline was analyzed by a gas chromatography (HP 4890II) equipped with a flame ionization detector (FID) and a SUPELCO Equity™ – 5 Capillary Column (length: 15m; id: 0.15 μm) with the rate 65 (°C/min), initial temperature 85°C and flow 10 psi. The solution pH was monitored using a SUNTEX pH/mV/TEMP (SP-701) meter. COD was measured by a closed-reflux titrimetic method based on Standard Methods (APHA, 1992). All experimental scenarios were duplicated. All experiments were carried out at batch mode using an acrylic reactor with a working volume of 5 liters. For the photoelectro-Fenton process, the anodes and cathodes used were mesh-type titanium metal coated with IrO_2/RuO_2 and stainless steel, respectively. The electrodes were connected to a Topward 33010D power supply operated at the desired electric current. The

reactor was also equipped with two mixers to ensure appropriate agitation and the UVA source was turned on to initiate the reaction. The irradiation source was a set of twelve 0.6 W UVA lamps (Sunbeamtech.com) fixed inside a cylindrical Pyrex tube (allowing wavelengths λ>320 nm to penetrate). In addition to all the experimental conditions mentioned above, UV light with maximum wavelength of 360 nm was irradiated inside the reactor, supplying a photoionization energy input to the solution of 7.2 W.

3. Results and discussion

In this step, the degradation of aniline was examined by various processes. Electrolysis, Photolysis, UV + hydrogen peroxide, Fenton, electro-Fenton, photo-Fenton and photoelectro-Fenton experiments in order to investigate the synergistic effect of Fenton's reagent combined with photo and electrochemical methods. As shown in Figure 1, the results show that electrolysis can remove 10% of 2,6-dimethylaniline within one hour. In the electrolysis method, aniline would be destroyed by reaction with adsorbed hydroxyl radical generated at the surface of a high oxygen-overvoltage anode from water oxidation. The same tendency can be found in the research of Brillas et al (Brillas et al., 1998).

Photolysis has lower degradation efficiency compared to electrolysis. The removal efficiency by photolysis was only 8% when using UVA lamps (12 lamps) at pH 3. The degradation of aniline was 12% when using UV+hydrogen peroxide after 60 minutes. Aniline was not well degraded by electrolysis, photolysis and UV+hydrogen peroxide. When using Fenton process, the degradation of aniline increased significantly compared to that when using direct photolysis, electrolysis and UV+hydrogen peroxide. For Fenton process, the degradation of aniline was 88% within one hour. This is due to the fast reaction of ferrous ion and hydrogen peroxide producing hydroxyl radicals (equation 1).
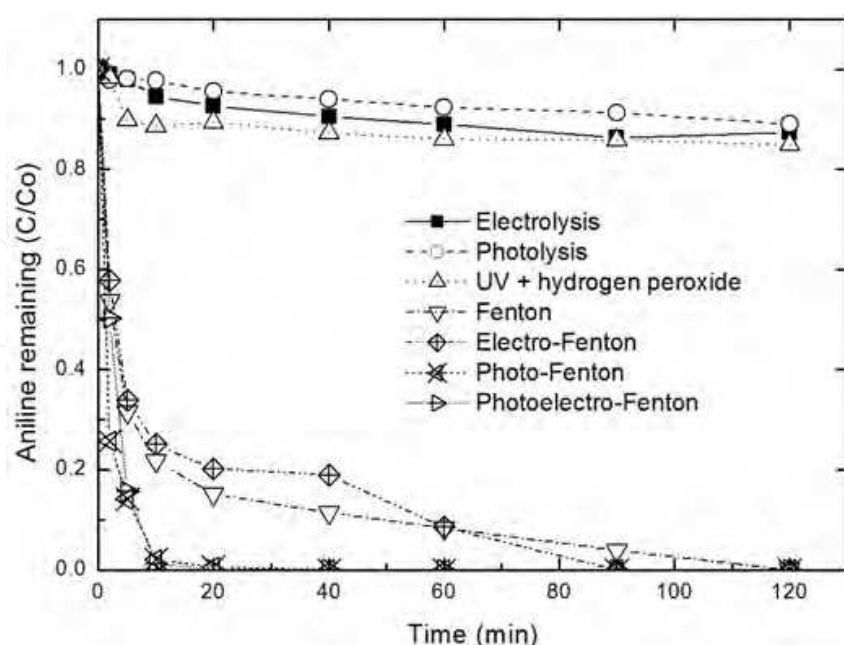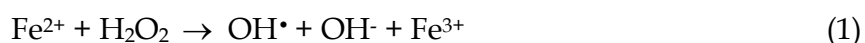
$$Fe^{2+} + H_2O_2 \rightarrow OH^\bullet + OH^- + Fe^{3+} \tag{1}$$

Fig. 1. Effect of different processes on aniline removal efficiency. [Aniline] = 1 mM; [Fe^{2+}] = 0.5 mM; [H_2O_2] = 20 mM; pH = 3; I = 1 A, UVA lamps = 12.

The 90% removal efficiency was achieved by the electro-Fenton process. The reason that electro-Fenton process can remove aniline more than the Fenton process is due to the regeneration of ferrous ion from equation (2) on the cathode side:

$$Fe^{3+} + e^- \rightarrow Fe^{2+} \tag{2}$$

and the ability of electricity that can produce hydroxyl radicals from water oxidation as described in equation (3).

$$H_2O \rightarrow OH^\bullet + H^+ + e^- \tag{3}$$

In photo-Fenton process, 100% of aniline degradation was observed at 60 min. Hence, photo-Fenton process is more efficient than Fenton process and electro-Fenton process. It is obvious that the photo-Fenton system enhanced the photooxidation of aniline. The hydroxyl radical which is a strong oxidant that can degrade aniline occurs by the following equation:

$$H_2O_2 + UV \rightarrow OH^\bullet + OH^\bullet \tag{4}$$

Degradation of aniline is mainly due to hydroxyl radical generated by photochemical reaction. In photo-Fenton process in addition to the above reaction the formation of hydroxyl radical (equation 1) also occurs by equation (4) and (5)

$$Fe^{3+} + H_2O + h\nu \rightarrow Fe^{2+} + OH^\bullet + H^+ \tag{5}$$
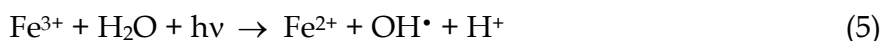
Meanwhile, the utmost removal efficiency was found when applied photoelectro-Fenton process, aniline was removed completely during the first 20 minutes. The degradation of aniline was due to the formation of hydroxyl radical from Fenton's reaction (equation 1) and the ferric ion would be reduced to ferrous ion from photoreduction (equation 5) and the cathode (equation 2). This would induce the formation of hydroxyl radicals efficiently.

The relative efficiencies of the above processes are in the following order: Photoelectro-Fenton > Photo-Fenton > Electro-Fenton > Fenton > UV + hydrogen peroxide > Electrolysis > Photolysis.

The degradation of aniline was monitored by measuring the COD removal. For COD removal, by electrolysis, photolysis, UV+hydrogen peroxide, Fenton, electro-Fenton, photo-Fenton and photoelectro-Fenton processes, it was followed the same trend as aniline degradation. The results revealed that electrolysis could remove COD only 9%, while photolysis was able to remove about 8% as shown in Figure 2. When UVA was combined with hydrogen peroxide, the COD removal increased to 14%. For Fenton process, the COD removal was 43% which is higher than using direct photolysis, electrolysis and UV+hydrogen peroxide .This is due to the fast reaction of ferrous ion and hydrogen peroxide producing hydroxyl radicals (equation 1).

The COD removal efficiency of aniline by the electro-Fenton process and photo-Fenton process were 49% and 52%, respectively. The reason might be due to the formation of hydroxyl radical from Fenton's reaction (equation 1) and the ferric ion would be reduced to ferrous ion from photoreduction (equation 5) in photo-Fenton process and the regeneration of ferrous ion on the cathode (equation 2) in electro-Fenton process. The highest COD removal was found when applied photoelectro-Fenton. It was about 70%. The decrease of COD can be attributed to the mineralization of aniline by the hydroxyl radicals from Fenton's reaction, from the electrochemically generated Fenton's reagent during electro-

Fenton and photoelectro-Fenton processes and the production of hydroxyl radical from photoreduction of $Fe(OH)^{2+}$ in photoelectro-Fenton process.

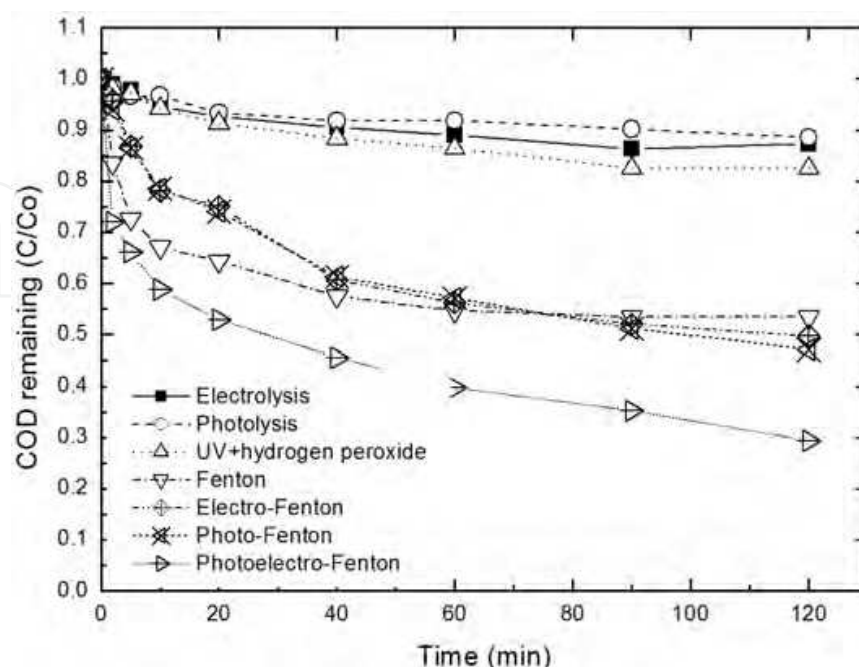Fig. 2. Effect of different processes on COD removal efficiency. [Aniline] = 1 mM; $[Fe^{2+}]$ = 0.5 mM; $[H_2O_2]$ = 20 mM; pH = 3; I = 1 A, UVA lamps = 12.

From this part of experiment, the photoelectro-Fenton was found to be the most efficient treatment to oxidize aniline. Thus, the following part in this chapter will focused on this process.

3.1 Kinetic study of aniline degradation by photoelectro-Fenton process

The kinetic study of aniline degradation by the photoelectro-Fenton was investigated. In this study, the kinetic was performed using initial rate techniques (at the first 10 minute of the reaction) in order to eliminate the interferences from intermediates that might occur during the study period. The test range of each parameter was chosen according to the reality of photoelectro-Fenton process application and the needs of kinetic study in this experiment. The effects of the initial concentration of aniline, initial concentration of ferrous ion and hydrogen peroxide on the kinetic study of aniline degradation will be separately discussed in the following sections.

3.1.1 Effect of initial aniline concentration

The pollutant concentration is one of the important factors in photoelectro-Fenton process. The effect of initial aniline concentration on the removal efficiency of aniline by photoelectro-Fenton processes is shown in Figure 3. The figure clearly reveals that increasing the aniline concentration decreases the removal efficiency of aniline. When increasing the aniline concentration from 0.5 to 5 mM, the degradation of aniline decreased from 100%, 100% to 60%. The main reason for this phenomenon is the hydroxyl radical. Increasing in aniline concentration increases the number of aniline molecules, however, there is not enough hydroxyl radicals to degrade 2,6-aniline, then the removal efficiency decreases.
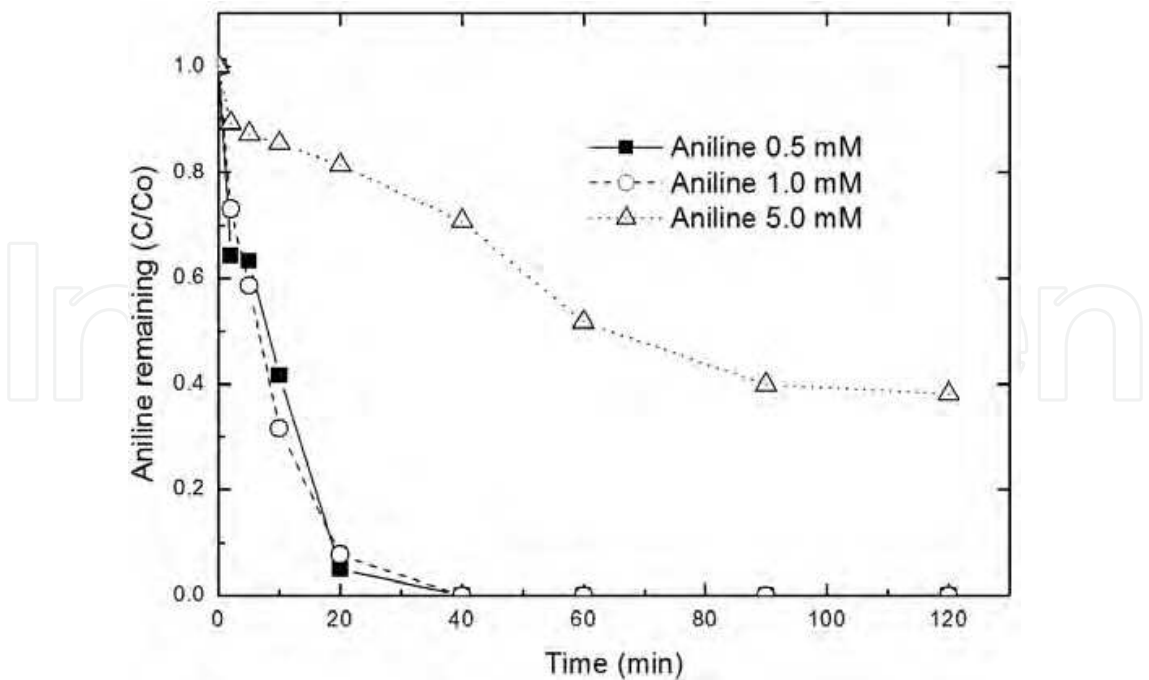
Fig. 3. Effect of initial aniline concentration on the removal efficiency of aniline by Photoelectro-Fenton process . $[Fe^{2+}] = 0.5$ mM, $[H_2O_2] = 20$ mM, I = 1 A, pH = 3, UVA lamp = 12

The effect of the initial aniline concentration on the kinetic was studied by varying the initial concentration from 0.5 to 5 mM and at the experimental condition of 0.5 mM of ferrous ion, 20 mM of hydrogen peroxide, pH 3, an electric current of 1 amperes for and UVA 12 lamps. The study shows the aniline degradation kinetics at the given test condition. The second-order behavior appeared to fit the degradation of aniline by the photoelectro-Fenton process when plot the relationship between aniline degradation and initial aniline concentration was found in a good linear (R> 0.98).

[Aniline]	Initial degradation rate
	Photoelectro-Fenton process
(x10^{-3}) M	(mMs^{-1})
0.5	0.0006
1	0.0008
5	0.0010

Table 1. Initial rate of aniline degradation by varying initial concentration of aniline using photoelectro-Fenton process.

The results on Table 1 shows that when the aniline concentration was increased from 0.5 to 5 mM, the initial degradation rates increased. The high initial degradation rate at a high concentration of aniline was probably due to the excess amount of aniline to react with hydroxyl radical which produced during the process that destroyed the aniline.
For that reason, the initial rate for the higher concentration of aniline increased. These results are similar to the study of *p*-nitroaniline degradation by Fenton oxidation process by Sun and others (Sun et al., 2007) and the results are also similar to the removal of nitroaromatic explosives with Fenton's reagent (Liou and Lu, 2007).

For the effect of initial aniline on the removal efficiency, the results show that increasing the initial concentration of aniline will decrease the removal. The plot between the initial rate and aniline concentration showed a straight line with the slopes of 0.21 for the photoelectro-Fenton process. Therefore, the reaction rate equation became:

$$-\left(\frac{d[Aniline]}{dt}\right)_{photoelectro-Fenton} = r_{PEF,Aniline} [2,6-DMA]^{0.21} \qquad (6)$$

where $r_{PEF,Aniline}$ is rate constant for the photoelectro-Fenton processes with respect to aniline.

3.2 Effect of ferrous ion concentration

The concentration of the catalyst is another important parameter for Fenton processes. The ferrous ion plays the catalyst's role in this Fenton's reaction. Normally, the rate of degradation increases with an increase in the concentration of ferrous ions. The effect of the ferrous ion concentration on the kinetic study of aniline degradation was studied by varying the ferrous ion concentration from 0.1 to 1 mM. The result showed that the removal efficiency of photoelectro-Fenton method was promoted when the ferrous ion concentration increased from 0.1 to 1 mM, as shown in Figure 4. The removal efficiency increased from 94% to 100 % in 40 minutes.
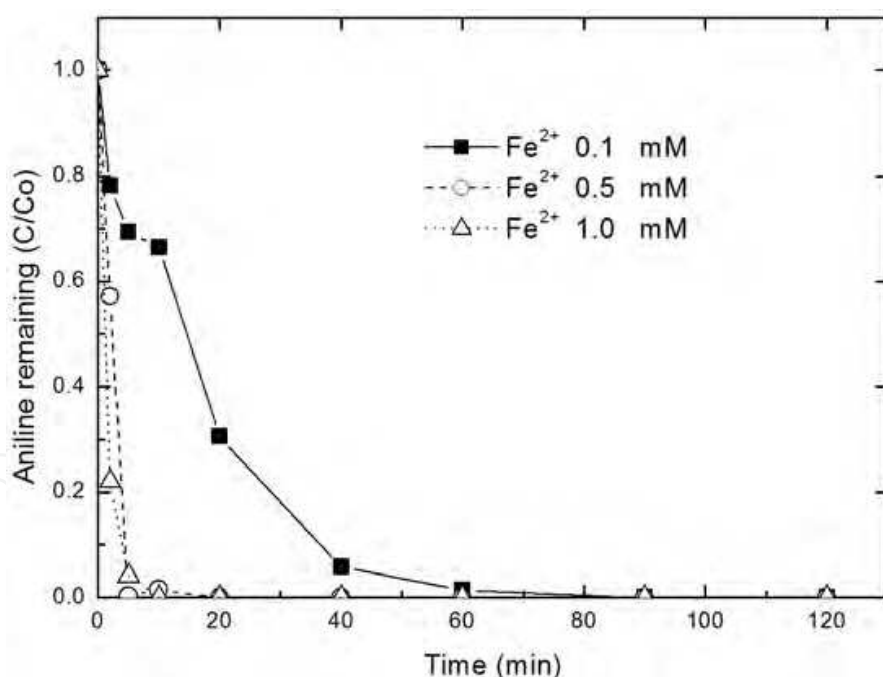

Fig. 4. Effect of initial ferrous ion concentration on the removal efficiency of aniline by Photoelectro-Fenton process . [Aniline] = 1 mM, [H_2O_2] = 20 mM, I = 1 A, pH = 3, UVA lamp = 12

For the initial degradation rate of aniline, the initial rate increased when the initial ferrous ion concentration increased from 0.0005 to 0.0016 mMs^{-1} in the photoelectron-Fenton process as listed in Table 2. Usually, when increasing the initial ferrous ion concentration, it will increase the generation rate of hydroxyl radical due to equation (1).

Therefore, this will enhance the degradation rate of aniline. Accordingly, the results underwent a similar direction. An improvement in removal efficiency by ferrous ion should also consider the supplement of hydrogen peroxide through out the experiment which might lead to more regeneration of ferrous ion from ferric ion. Therefore, the total regenerated hydroxyl radicals and the removal efficiency could increase as the ferrous ion concentration increased.

[Fe^{2+}] (x10^{-3}) M	Initial degradation rate Photoelectro-Fenton process (mMs^{-1})
0.1	0.0005
0.5	0.0014
1.0	0.0016

Table 2. Initial rate of aniline degradation by varying initial concentration of ferrous ion using photoelectro-Fenton process.

In photoelectro-Fenton process, the applied electricity and UVA lamps during experiment will enhance the regeneration of ferrous ion from ferric ion via equation (3) and (5). The supplied electrons from electrical current can regenerate ferrous ions rapidly and, thus, can react with hydrogen peroxide as long as hydrogen peroxide is still available in the reactor (Anotai et al., 2006). For that reason, the degradation of aniline continuously proceeded without the inadequacy of ferrous ions in the solution which in turn increased the efficiency of hydroxyl radical production.

The aniline degradation kinetics at the given test conditions was second–order behavior. The degradation kinetic for the photoelectro-Fenton process when plot the relationship between aniline degradation and aniline concentration was in a good linear (R> 0.95) when using second-order kinetic model. It was seen from the results that the rate of aniline degradation increased with the increase of ferrous ions concentration. The relationship between the initial rate and the ferrous ion concentration in the photoelectron-Fenton process was determined. From the calculation, it showed that the slope of photoelectro-Fenton processes was 0.5289. Thus, the kinetics for aniline degradation on the effect of ferrous ion concentration can be described by following equation:

$$-\left(\frac{d[Aniline]}{dt}\right)_{photoelectro-Fenton} = r_{PEF,Fe^{2+}} \; [Fe^{2+}]^{0.5289} \qquad (7)$$

where $r_{PEF,Fe^{2+}}$ is the rate constant for photoelectro-Fenton process with respect to the ferrous ion. The results indicated that ferrous ions play an important role in degradation of aniline by reacting with hydrogen peroxide to generate hydroxyl radicals. However, higher ferrous ion concentration supplementation by using photoelectro-Fenton processes is not recommended due to the large amount of ferric hydroxide sludge that could occur.

3.3 Effect of hydrogen peroxide concentration
Initial concentration of hydrogen peroxide plays an important role in the photoelectro-Fenton process. It is an oxidizing agent in the Fenton reaction. It has been observed that the percentage degradation of the pollutant increases with an increase in the concentration of

hydrogen peroxide (Pignatello, 1992). The effect of initial hydrogen peroxide concentration on the removal efficiency and kinetic rate of aniline degradation was investigated by varying the initial hydrogen peroxide concentration from 1 to 40 mM under the experiment condition of initial aniline concentration 1 mM, ferrous ion 0.5 mM, pH 3, electrical current 1 A and UVA 12 lamps.
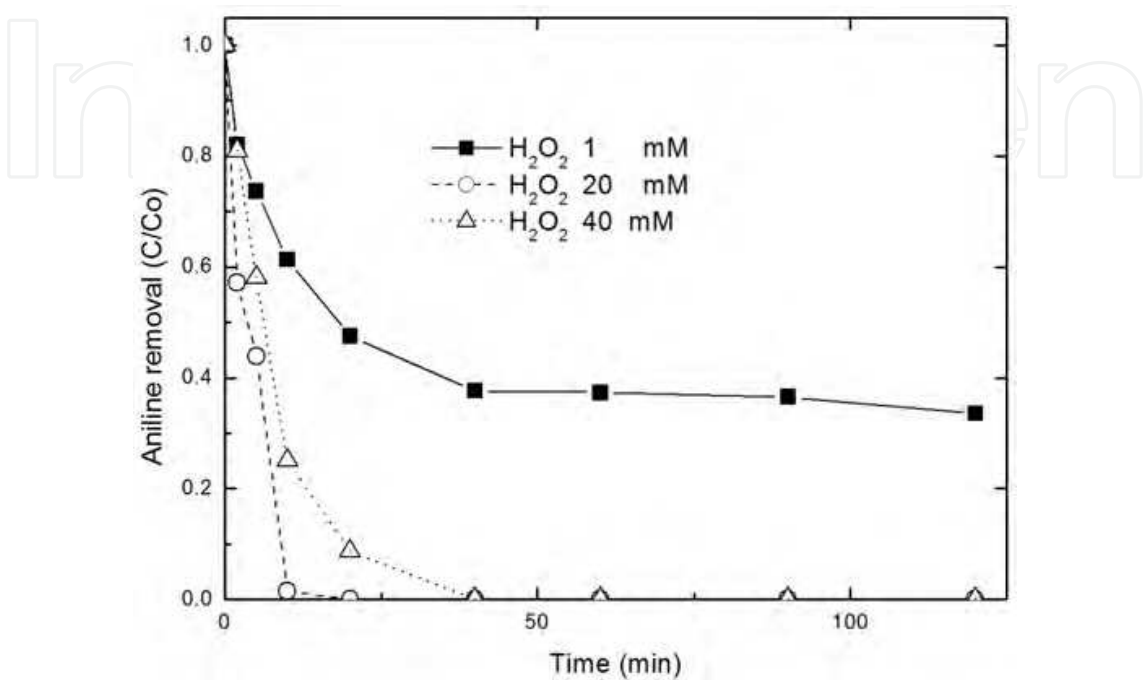

Fig. 5. Effect of initial hydrogen peroxide concentration on the removal efficiency of aniline by Photoelectro-Fenton process . [Aniline] = 1 mM, [Fe^{2+}] = 0.5 mM, I = 1 A, pH = 3, UVA lamp = 12

It can be seen from the results that the removal efficiency of photoelectro-Fenton processes increased when increasing the initial concentration of hydrogen peroxide as shown in Figure 5. The removal efficiency increased from 63 % to 100 % as the hydrogen peroxide concentration increased from 1 to 40 mM in 60 minutes. This was from the production of hydroxyl radicals produced during the experiment by the reaction of ferrous ion and hydrogen peroxide in the solution. Similar result was obtained by Lin et al.,1999.

| [H_2O_2] | Initial degradation rate |
| | Photoelectro-Fenton process |
($\times 10^{-3}$) M	(mMs^{-1})
1	0.0006
20	0.0015
40	0.0012

Table 3. Initial rate of aniline degradation by varying initial concentration of hydrogen peroxide using photoelectro-Fenton process.

Table 3 shows that the initial rate of aniline degradation increased when the hydrogen peroxide concentration was increased from 1 to 20 mM. The initial rate increased from 0.0006 to 0.0015 mMs^{-1} by photoelectro-Fenton process. This increase in the initial rate was

due to the availability of hydrogen peroxide to react with ferrous ions in the solution. However, with the continuous increase in the initial hydrogen peroxide concentration to 40 mM, the increase of hydrogen peroxide leads to the decline of the initial rate. This phenomenon was probably due to the scavenging of hydroxyl radicals by hydroxyl radical as described via equation (5) (Lu et al., 1999). The accumulation of hydroperoxyl radicals also consumed hydroxyl radicals (Sun et al., 2007; Kang et al., 2002).

It was found that the second-order kinetic model is applicable to the aniline degradation quite well under photoelectro-Fenton process with R> 0.98. The relationship between the initial rate and hydrogen peroxide is linear and the slope is 0.2232 for the photoelectro-Fenton process, therefore:

$$-\left(\frac{d[Aniline]}{dt}\right)_{photoelectro-Fenton} = r_{PEF,H_2O_2} \, [H_2O_2]^{0.2232} \tag{8}$$

where r_{PEF,H_2O_2} is rate constant for the photoelectro-Fenton methods with respect to hydrogen peroxide.

3.4 The overall reaction rate equation for aniline degradation by photoelectro-Fenton

From the previous sections, the reaction rate equation of aniline degradation is proposed and it varied with the respect to aniline, ferrous ion and hydrogen peroxide concentrations in photoelectro-Fenton process. The overall degradation kinetic for aniline by the photoelectro-Fenton process can be summarized as shown below:

$$-\left(\frac{d[Aniline]}{dt}\right)_{photoelectro-Fenton} = r_{PEF}[Aniline]^{0.21} \, [Fe^{2+}]^{0.5289} \, [H_2O_2]^{0.2232} \tag{9}$$

where r_{PEF} is the overall rate constant for the photoelectro-Fenton method. From the equation (9), it can be seen that the degradation rate of aniline by photoelectro-Fenton process depended on Fenton's reagent both ferrous ion and hydrogen peroxide. However, from equation (9), the reaction shows that the aniline degradation, when applied with an electrical current and UVA lamp, the degradation rate is still depend on hydrogen peroxide. Moreover, the ferrous ion is also the important species for the degradation of this chemical so the Fenton's reagent still be the major key for this process.

The r_{PEF}, from equation (9) can be calculated using a non-linear least squares method which minimizes the sum of the error squares between the observed initial rates attained from the study and from the calculated initial rates. Accordingly, the rPEF can be proposed by calculated using concentration of aniline, ferrous ion and hydrogen peroxide in millimolar (mM) units. From the calculation, the r_{PEF} was 1.32. Therefore, the final reaction rate equations can be described as:

$$-\left(\frac{d[Aniline]}{dt}\right)_{photoelectro-Fenton} = 1.32[Aniline]^{0.21} \, [Fe^{2+}]^{0.5289} \, [H_2O_2]^{0.2232} \tag{10}$$

4. Conclusions

Fenton, electro-Fenton and photoelectro-Fenton processes were able to oxidize aniline. However, in presence of electrical current and UVA lamps, the removal of aniline was higher. The results show that relative efficiencies of the AOPs processes on aniline degradation are in the following order: Photoelectro-Fenton > Photo-Fenton > Electro-Fenton > Fenton > UV + hydrogen peroxide > Electrolysis > Photolysis. These processes could degrade this compound. The photoelectro-Fenton process was found to give the highest degradation efficiency than the other processes. 100% degradation of 1 mM aniline was achieved in 40 minutes when 1 mM of ferrous ions, 20 mM of hydrogen peroxide, pH 3, 1 A of electric current and 12 lamps of UVA were applied. This was due to the regeneration of ferrous ion from ferric ion that can improve the performance of overall degradation. The overall rate equations for the degradation of aniline by photoelectro-Fenton process was evaluated as shown in equation (10). The kinetics established in this experiment was mathematically determined by considering the three important parameters including aniline, ferrous ion and hydrogen peroxide. The initial rate of aniline was increased with the increase of aniline concentration in all processes and increased with the increase of initial ferrous ion and hydrogen peroxide concentration. It can be indicated that aniline, ferrous ion and hydrogen peroxide have strong influences on the kinetic rate constants for aniline degradation.

5. Acknowledgements

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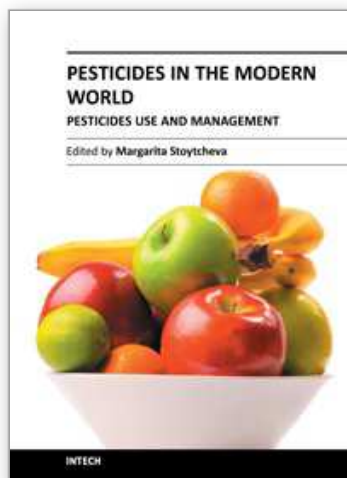
This book brings together issues on pesticides and biopesticides use with the related subjects of pesticides management and sustainable development. It contains 24 chapters organized in three sections. The first book section supplies an overview on the current use of pesticides, on the regulatory status, on the levels of contamination, on the pesticides management options, and on some techniques of pesticides application, reporting data collected from all over the world. Second section is devoted to the advances in the evolving field of biopesticides, providing actual information on the regulation of the plant protection products from natural origin in the European Union. It reports data associated with the application of neem pesticides, wood pyrolysis liquids and bacillus-based products. The third book section covers various aspects of pesticides management practices in concert with pesticides degradation and contaminated sites remediation technologies, supporting the environmental sustainability.

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