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ORIGINAL ARTICLE

Photo oxidative degradation of azure-B by sono-photo-Fenton and photo-Fenton reagents

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Abstract A model for the decomposition of azure-B by photo-Fenton reagent in the presence of ultrasound in homogeneous aqueous solution has been described. The photochemical decomposition rate of azure-B is markedly increased in the presence of ultrasound. It is a rather inexpensive reagent for wastewater treatment. The effect of different variables like the concentration of ferric ion, concentration of dye, hydrogen peroxide, pH, light intensity etc. on the reaction rate has been observed. The progress of the sono-photochemical degradation was monitored spectrophotometrically. The optimum sono-photochemical degradation conditions were experimentally determined. The results showed that the dye was completely oxidized and degraded into CO_2 and H_2O. A suitable tentative mechanism for sono-photochemical bleaching of azure-B by sono-photo-Fenton's reaction has been proposed.

1. Introduction

Among the methods aimed at the degradation of pollutants in aqueous solution, much attention has recently been focused on the so-called advanced oxidation processes for water and wastewater decontamination. In these processes, various techniques (e.g., photolysis, photocatalysis, Fenton reaction, sonochemistry, UV-H_2O_2, UV-O_3 and photo-Fenton) are applied to produce reactive species (often, but not always, the hydroxyl radical) with the purpose of inducing the transformation of water dissolved organic pollutants. This treated water may then be used for washing, cooling, irrigation and cleaning purposes, otherwise it is of no use before treatment.

Treatment method using the Fenton reaction (Fenton, 1894), utilizes hydroxyl radicals produced by interaction of H_2O_2 with ferrous salts-

$$Fe^{2+} + H_2O_2 + H^+ \rightarrow Fe^{3+} + {}^{\cdot}OH + H_2O \text{ (Fenton reaction)} \tag{1}$$

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In the dark, this reaction is stopped after complete conversion of Fe^{2+}–Fe^{3+} ions. The reasons for the rate enhancement of degradation on light irradiation is the photoreduction of Fe^{3+} ions back to Fe^{2+} ions, which produces new $^{\cdot}OH$ radicals with H_2O according to the following mechanism (Carey, 1990).

$$[FeOH]^{2+} + h\nu \rightarrow Fe^{2+} + {}^{\cdot}OH \text{ (photo} - \text{Fenton reaction)} \tag{2}$$

The direct photolysis of H_2O_2 also generates $^{\cdot}OH$ radicals.

$$H_2O_2 + h\nu \rightarrow 2{}^{\cdot}OH \; (\lambda < 400 \text{ nm}) \tag{3}$$

Sonolysis of water yields hydroxyl radicals and hydrogen atoms. Hydroxyl radicals in particular are very reactive and can transform organic pollutants (Makino et al., 1983; Buxton et al., 1988; Riesz and Kondo, 1992).

$$H_2O \xrightarrow{)))} H^{\cdot} + {}^{\cdot}OH \text{ (Ultrasonic irradiation)} \tag{4}$$

Degradation of many organic pollutants occurs in solutions exposed to ultrasounds (Ince et al., 2001; Naffrechoux et al., 2000; Kotronarou et al., 1992). Sonochemical degradation of a triphenyl methane dye was described (Panwar et al., 2007). Characterization of the degradation performance of the sulfamethazine antibiotic by photo-Fenton process has been carried out (Moya et al., 2010). The effect of Fe^{2+2}, Fe^{3+3}, H_2O_2 and the photo-Fenton reaction at near neutral pH on the solar disinfection (SODIS) at low temperature of water containing *Escherichia coli* K12 has been suggested (Spuhler et al., 2010).

Decolorization of solution containing a common textile and leather dye using advanced oxidation processes like Fenton, $UV/H_2O_2/O_2$, $UV/H_2O_2/Fe^{2+}$, $UV/H_2O_2/Fe^{3+}$ and $UV/H_2O_2/Fe^{3+}$/oxalate processes has been studied (Daneshvar and Khataee, 2006; Dutta et al., 2001; Ma et al., 2005; Liou et al., 2004; Prousek et al., 1997; Wu et al., 1998). The photo-Fenton reaction as an effective photochemical process to treat the polluted water was described (Ruppert et al., 1993). The use of photo-Fenton's reaction for the photochemical bleaching of metanil yellow, fast green FCF and rhodamine-B has been reported (Kumar et al., 2008a,b,c) while the photochemical degradation of *p*-dichlorobenzene and *o*-chlorobenzoic acid by photo-Fenton's reagent has also been reported (Mogra et al., 2002, 2003).

In the present study, the sono-photochemical degradation of azure-B by sono-photo-Fenton's reagent using visible light has been carried out and the results show that the sono-photo-Fenton's reagent is a better oxidizing reagent as compared to photo-Fenton's reagent.

2. Experimental

2.1. Materials and methods

All solutions were prepared in doubly distilled water. Azure-B (Qualigen), $FeCl_3$ (CDH) and H_2O_2 (30%, Merck), were used. The concentrations of the solutions were 1.0×10^{-3} and 5.0×10^{-3} mol/L for azure-B and $FeCl_3$, respectively. These were used as stock solutions. Sono-photochemical degradation of azure-B was investigated taking 30 mL of total reaction mixture consisting of dye solution, $FeCl_3$ solution and H_2O_2. The concentration of dye is 1.33×10^{-4} mol/L for sono-photo-Fenton (SPF) and 1.0×10^{-4} mol/L for photo-Fenton (PF), of $FeCl_3$ is 5.0×10^{-4} mol/L for sono-photo-Fenton and 6.67×10^{-4} mol/L for photo-Fenton while H_2O_2 is 0.5 and 1.5 mL for sono-photo-Fenton and photo-Fenton, respectively, in the reaction mixture. The reaction mixture was exposed to the light (75.5 mW cm^{-2}) by a 200 W tungsten lamp (Sylvania Laxman). The light intensity was measured with a Suryamapi (CEL Model SM 201). In sono-photo-Fenton reaction, the reaction mixture was also exposed to the ultrasound (Systronics Ultrasonic Cleaner-392, Italy), with frequency of 40 kHz. A water filter was used to cut off thermal radiations. pH of the solution was adjusted by the addition of standardized sulfphuric acid and sodium hydroxide solutions and measured with a digital pH meter (Systronics Model 335). The necessary condition for the correct measurement of absorbance is that the solution must be free from any suspension. A G 3 sintered glass crucible was used for filtration to obtain the desired accuracy in measurement of absorbance. λ_{max} of the dye was determined using an ultraviolet–visible spectrophotometer (Systronics Model 106).

3. Results and discussion

The photochemical degradation in the presence of ultrasound of azure-B was observed at $\lambda_{max} = 650$ nm. The structure of azure-B has been given in Fig. 1.

The results of a typical run are represented in Fig. 2. It was observed that the absorbance of azure-B solution decreases with increasing time of irradiation; thus, indicating that the dye is consumed. A plot of log (absorbance) against time was linear, following the pseudo-first order kinetics. The rate constant k was calculated from the expression $k = 2.303 \times \text{slope}$. The optimum rate constants for these reactions were determined as $k_1 = 2.99 \times 10^{-3}\ s^{-1}$ for photo-Fenton reaction and $k_2 = 4.39 \times 10^{-3}\ s^{-1}$ for sono-photo-Fenton reaction. The spectral changes for these reactions have been shown in Figs. 3 and 4.

3.1. Effect of pH

The effect of pH on the rate of degradation of azure-B was investigated in the pH range 1.5–2.7. The results are reported in Table 1.

The degradation of azure-B depends strongly on pH of the reaction medium. The reaction rate increases with increasing pH up to 2.2 and 2.1 in photo-Fenton and sono-photo-Fenton reaction, respectively and then decreases. The hydroxyl radicals are generated in two steps;

(i) In the reaction between Fe^{2+} ions with hydrogen peroxide.
(ii) In photochemical reaction of Fe^{3+} ions and water.

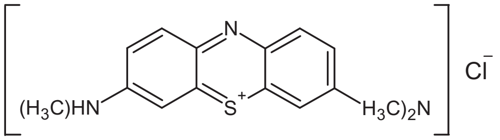

Figure 1 Structure of azure-B.

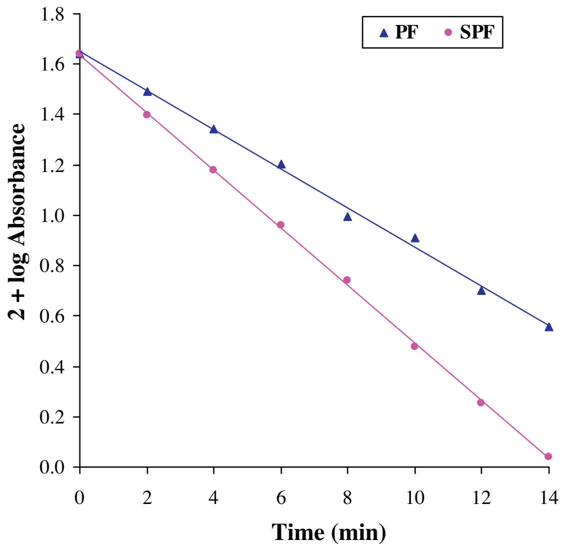

Figure 2 A typical run; photo-Fenton - [azure-B] = 1.0×10^{-4} -mol/L, pH = 2.2, [Fe^{3+}] = 6.67×10^{-4} mol/L, H_2O_2 = 1.5 mL, Llight intensity = 75.5 mW cm^{-2}; sono-photo-Fenton - [azure-B] = 1.33×10^{-4} mol/L, pH = 2.1, [Fe^{3+}] = 5.0×10^{-4} mol/L, H_2O_2 = 0.5 mL, Light intensity = 75.5 mW cm^{-2}, Ffrequency = 40 kHz.

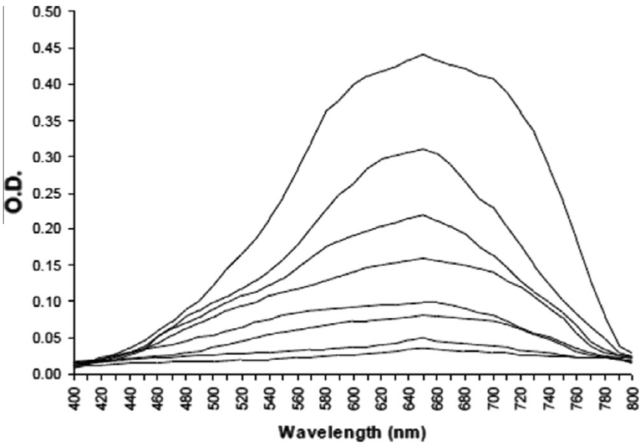

Figure 3 Spectral change for photo-Fenton reaction.

The increase in pH of the medium favors the step (i) where OH^- ions are formed along with hydroxyl radicals, whereas protons are generated in step (ii). Thus, it may be concluded that the step (i) dominates over step (ii) at pH below 2.1 and 2.2 in SPF and PF reaction, respectively. However, the retardation of the reaction above pH 2.1 and 2.2 in SPF and PF reaction, respectively, suggests the dominance of step (ii) over step (i).

3.2. Effect of azure-B concentration

Effect of variation of dye concentration on the reaction rate was also studied by taking different concentrations of azure-B solutions. The results are given in Table 2.

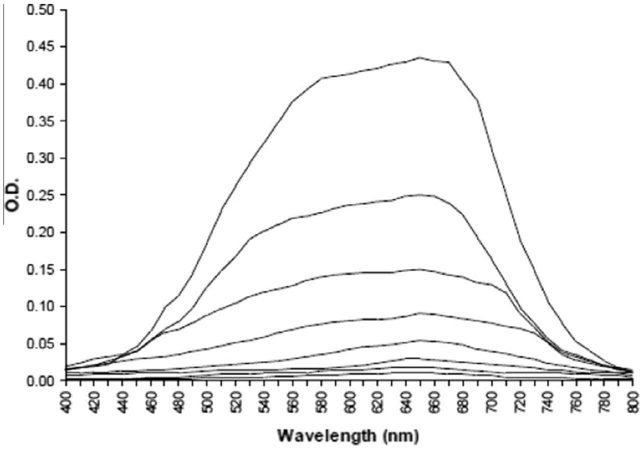

Figure 4 Spectral change for sono-photo-Fenton reaction.

Table 1 Effect of pH.

pH	Photo-Fenton $k \times 10^3$ s^{-1}	Sono-photo-Fenton $k \times 10^3$ s^{-1}
1.5	0.72	3.19
2.0	0.93	3.51
2.1	1.33	**4.39**
2.2	**2.98**	4.21
2.3	1.87	3.87
2.4	1.72	3.51
2.5	1.12	3.19
2.6	0.98	2.83
2.7	0.93	2.34

Photo-Fenton: [Azure-B] = 1.0×10^{-4} mol/L; [Fe^{3+}] = 6.67×10^{-4} mol/L; H_2O_2 = 1.5 mL; light intensity = 75.5 mW cm^{-2}.
Sono-photo-Fenton: [Azure-B] = 1.33×10^{-4} mol/L; [Fe^{3+}] = 5.0×10^{-4} mol/L; H_2O_2 = 0.5 mL; light intensity = 75.5 mW cm^{-2}; frequency = 40 kHz.

Table 2 Effect of concentration of azure-B.

[Azure-B] $\times 10^4$ mol/L	Photo-Fenton $k \times 10^3$ s^{-1}	Sono-photo-Fenton $K \times 10^3$ s^{-1}
0.33	1.59	1.08
0.67	2.27	1.23
1.0	**2.98**	2.30
1.33	2.89	**4.39**
1.67	2.56	3.87
2.0	1.71	2.30
2.33	1.52	1.53
2.67	1.27	0.69

Photo-Fenton: pH = 2.2; [Fe^{3+}] = 6.67×10^{-4} mol/L; H_2O_2 = 1.5 mL; light intensity = 75.5 mW cm^{-2}.
Sono-photo-Fenton: pH = 2.1; [Fe^{3+}] = 5.0×10^{-4} mol/L; H_2O_2 = 0.5 mL; light intensity = 75.5 mW cm^{-2}; frequency = 40 kHz.

The rate of degradation of the dye was found to increase with increasing concentration of azure-B up to 1.33×10^{-4} and 1.0×10^{-4} mol/L in SPF and PF reaction, respectively. On further increasing its concentration, a sudden decrease in the rate of degradation was observed. On increasing the concentration above 1.33×10^{-4} and 1.0×10^{-4} mol/L in SPF and PF reaction, respectively, the reaction rate was found to decrease. This may be attributed to the fact that as the concentration of azure-B was increased, it may start acting as a filter for the incident light. The larger concentrations of dye azure-B will not permit the desired light intensity to reach the dye molecules in the bulk of the solution.

3.3. *Effect of ferric ion concentration*

The effect of concentration of Fe^{3+} ions on the rate of degradation of azure-B was observed by keeping all other factors identical. The results are summarized in Table 3.

It is clear from the data in the table that in both these cases; sono-photodegradation and photodegradation of the dye, the rate of degradation increases on increasing the concentration of Fe^{3+} ions up to 6.67×10^{-4} and 5.0×10^{-4} mol/L for PF and SPF, respectively. This may be explained on the basis that with the increase in Fe^{3+} concentration, there will be an enhanced generation of $^{\cdot}OH$ radicals and as a consequence, the rate of sono-photodegradation and photodegradation increases. However, on increasing the concentration of Fe^{3+} ions further (in sono-photodegradation), the rate of the reaction was found to decrease. This is because of the fact that Fe^{3+} ions imparts a yellow color to the solution and at larger concentrations, it may also act as a filter for the incident light. As the concentration of Fe^{+3} was increased above its optimum concentration, the rate of the reactions (6) and (7) become very fast. In reaction (6), hydroperoxyl radicals ($^{\cdot}OOH$) are generated, which consume more amounts of Fe^{3+} ions in PF but in SPF, an extra reaction (8) also consume Fe^{3+} ions and hence, Fe^{3+} ions are less available for reaction (5) and as a result, less $^{\cdot}OH$ radicals are generated. Therefore, the rate of sono-photodegradation and photodegradation also decreases.

$$Fe^{3+} + H_2O + h\nu \rightarrow Fe^{+2} + {}^{\cdot}OH + H^+ \tag{5}$$

$$Fe^{3+} + H_2O_2 + h\nu \rightarrow Fe^{2+} + HO_2^{\cdot} + H^+ \tag{6}$$

$$Fe^{3+} + HO_2^{\cdot} \rightarrow Fe^{2+} + O_2 + H^+ \tag{7}$$

$$Fe^{3+} + H^{\cdot} \rightarrow Fe^{2+} + H^+ \tag{8}$$

3.4. *Effect of hydrogen peroxide*

The effect of the amount of hydrogen peroxide on the sono-photodegradation and photodegradation of azure-B was also investigated. The results are reported in Table 4.

Table 3 Effect of concentration of ferric ion.

$[Fe^{3+}] \times 10^4$ mol/L	Photo-Fenton $k \times 10^3\ s^{-1}$	Sono-photo-Fenton $k \times 10^3\ s^{-1}$
0.83	0.30	1.07
1.67	0.69	1.43
2.50	0.82	2.01
3.33	1.03	2.09
4.17	1.52	2.51
5.00	2.44	**4.39**
5.83	2.93	2.51
6.67	**2.98**	2.31

Photo-Fenton: [Azure-B] = 1.0×10^{-4} mol/L; pH = 2.2; H_2O_2 = 1.5 mL; light intensity = 75.5 mW cm^{-2}.
Sono-photo-Fenton: [Azure-B] = 1.33×10^{-4} mol/L; pH = 2.1; H_2O_2 = 0.5 mL; light intensity = 75.5 mW cm^{-2}; frequency = 40 kHz.

Table 4 Effect of amount of H_2O_2.

H_2O_2 (mL)	Photo-Fenton $k \times 10^3\ s^{-1}$	Sono-photo-Fenton $k \times 10^3\ s^{-1}$
0.5	1.42	**4.39**
1.0	1.71	3.29
1.5	**2.98**	2.59
2.0	1.71	2.36
2.5	1.42	2.22
3.0	1.14	1.56
3.5	0.75	1.41
4.0	0.58	1.11

Photo-Fenton: [Azure-B] = 1.0×10^{-4} mol/L; pH = 2.2; $[Fe^{3+}]$ = 6.67×10^{-4} mol/L; light intensity = 75.5 mW cm^{-2}.
Sono-photo-Fenton: [Azure-B] = 1.33×10^{-4} mol/L; pH = 2.1; $[Fe^{3+}]$ = 5.0×10^{-4} mol/L; light intensity = 75.5 mW cm^{-2}; frequency = 40 kHz.

It was observed that the reaction rate increases on increasing the amount of H_2O_2, attaining an optimum value of H_2O_2 at 1.5 mL for PF reaction. It is because of the fact that as the amount of H_2O_2 was increased above its optimum value (1.5 mL for PF reaction) the rates of the reactions (6) and (9) increase in PF system. Thereafter, the rate of degradation decreases on increasing the amount of hydrogen peroxide more than 0.5 and 1.5 mL for SPF and PF reaction, respectively. From Eq. (9), $^{\cdot}OH$ radicals are consumed rapidly due to availability of more H_2O_2 molecules. From Eqs. (6) and (9), $^{\cdot}OOH$ radicals are generated in more amounts. This $^{\cdot}OOH$ radical is utilized in Eq. (10) and H^+ ions are produced. As a consequence, the rate of sono-photodegradation and photodegradation decreases.

$${}^{\cdot}OH + H_2O_2 \rightarrow HO_2^{\cdot} + H_2O \tag{9}$$

$$Fe^{3+} + HO_2^{\cdot} \rightarrow Fe^{2+} + O_2 + H^+ \tag{10}$$

3.5. *Effect of light intensity*

The effect of light intensity on the sono-photodegradation and photodegradation of azure-B was also investigated. The results obtained are reported in Table 5.

Plots of the rate constant versus light intensity were linear, which indicate that an increase in the light intensity increases the reaction rate. This may be attributed to the increased number of photons reacting with Fe^{3+} ions and, as a result, increased numbers of active species, the hydroxyl radicals are formed. Therefore, an overall increase in the rate of reaction was observed.

4. Mechanism

On the basis of experimental observations and the existing literature, a tentative mechanism has been proposed for sono-photodegradation of azure-B with the sono-photo-Fenton (SPF) reagent.

$$Fe^{3+} + H_2O + h\nu \rightarrow Fe^{+2} + {}^{\cdot}OH + H^+ \tag{11}$$

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + {}^{\cdot}OH + OH^- \tag{12}$$

$${}^{\cdot}OH + HO_2^{\cdot} \rightarrow H_2O + O_2 \tag{13}$$

Table 5 Effect of light intensity.

Light intensity ($mW\ cm^{-2}$)	Photo-Fenton $k \times 10^3\ s^{-1}$	Sono-photo-Fenton $k \times 10^3\ s^{-1}$
27.7	1.03	1.97
30.6	1.13	2.26
34.6	1.23	2.63
39.6	1.33	3.15
45.6	1.69	3.29
53.2	2.26	3.51
62.9	2.68	4.29
75.5	**2.98**	**4.39**

Photo-Fenton: [Azure-B] = 1.0×10^{-4} mol/L; pH = 2.2; $[Fe^{3+}] = 6.67 \times 10^{-4}$ mol/L; H_2O_2 = 1.5 mL.
Sono-photo-Fenton: [Azure-B] = 1.33×10^{-4} mol/L; pH = 2.1; $[Fe^{3+}] = 5.0 \times 10^{-4}$ mol/L; H_2O_2 = 0.5 mL; frequency = 40 kHz.

$$Fe^{2+} + {}^{\cdot}OH \rightarrow Fe^{3+} + OH^{-} \quad (14)$$

$$HO_2^{\cdot} + HO_2^{\cdot} \rightarrow H_2O_2 + O_2 \quad (15)$$

$$^{\cdot}OH + {}^{\cdot}OH \rightarrow H_2O_2 \quad (16)$$

By ultrasound:

$$H_2O \xrightarrow{\cdot)))} {}^{\cdot}OH + H^{\cdot} \quad (17)$$

$$H^{\cdot} + H_2O_2 \rightarrow {}^{\cdot}OH + H_2O \quad (18)$$

$$Fe^{3+} + H^{\cdot} \rightarrow Fe^{2+} + H^{+} \quad (19)$$

$$Azure - B + {}^{\cdot}OH \rightarrow Products \quad (20)$$

An aqueous solution of ferric ions on exposure to light decomposes water into a proton and $^{\cdot}OH$ radical and ferric ions are reduced to ferrous ions. The ferrous ions decompose H_2O_2 into hydroxide ion and hydroxyl radical, while ferrous ions undergo oxidation to ferric ions. Due to reaction of ferric ions with H_2O_2 in the presence of light, $HO_2^{\cdot}$ radicals are also produced. The reaction of $^{\cdot}OH$ with H_2O_2 also produces $HO_2^{\cdot}$ radicals. Ferrous ions will undergo oxidation to ferric ions by addition of $^{\cdot}OH$ radicals, while ferric ions get reduced to ferrous ions by incorporation of $HO_2^{\cdot}$ radical and producing H^{+} ions. $HO_2^{\cdot}$ radicals are highly unstable in water and undergo facile disproportionation rather than reacting slowly with dye molecules. The participation of hydroxyl radical as an active oxidizing species was confirmed by using a hydroxyl radical scavenger like propan-2-ol, where the rate of photodegradation was drastically reduced.

The $^{\cdot}OH$ radicals are consumed in five reactions. They can induce the dissociation of H_2O_2 into $HO_2^{\cdot}$ and water or they combine (dimerize) to form H_2O_2 molecules. They can also induce the dissociation of $HO_2^{\cdot}$ water and O_2. Secondly, these may react with azure-B to give colorless degradation products.

In SPF reaction, $^{\cdot}OH$ radicals are produced in two reactions. The ultrasonic wave can induce the dissociation of H_2O into $^{\cdot}OH$ and $H^{\cdot}$, and $H^{\cdot}$ can induce the dissociation of H_2O_2 into $^{\cdot}OH$ and water, and this extra generation of $^{\cdot}OH$ radicals enhances the rate of SPF of reaction.

The main advantage of using sono-photo-Fenton's and/or photo-Fenton's reagents is the regeneration of the consumed Fe^{2+} ions on illumination. Each Fe^{2+} ion can produce many $^{\cdot}OH$ radicals in contrast to the dark Fenton reaction. The process is a cyclic one. It means that the amount of ferrous salt required under SPF and PF conditions is very small as compared to Fenton conditions, where ferrous ions are to be added at regular intervals; otherwise, the reaction will stop after complete conversion of ferrous ions to ferric ions. This is important from an industrial point of view because further separation of ferric ions is not required after wastewater treatment (Zepp et al., 1992; Tao et al., 2005; Collins, 2002; Chen et al., 2002; Ma et al., 2003; Bozzi et al., 2004).

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