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Separation Science and Technology

Publication details, including instructions for authors and subscription information:
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Online publication date: 19 July 2010

To cite this Article Vujević, Dinko , Papić, Sanja , Koprivanac, Natalija and Božić, Ana Lončarić(2010) 'Decolorization and Mineralization of Reactive Dye by UV/Fenton Process', Separation Science and Technology, 45: 11, 1637 — 1643

To link to this Article: DOI: 10.1080/01496395.2010.487734

URL: http://dx.doi.org/10.1080/01496395.2010.487734

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Separation Science and Technology, 45: 1637–1643, 2010
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ISSN: 0149-6395 print/1520-5754 online
DOI: 10.1080/01496395.2010.487734

Decolorization and Mineralization of Reactive Dye by UV/Fenton Process

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The photochemical degradation of C. I. Reactive Blue 2 (RB2) in aqueous solution has been studied using a homogeneous UV/Fenton process. The effects of applied H_2O_2, Fe^{2+} salt and RB2 concentrations at constant UV irradiation characteristics, the pH, and temperature were determined. By the application of 0.5 mM Fe^{2+} and 2.5 mM of H_2O_2 the complete color removal and mineralization extent of 73.8% were achieved. According to the color and TOC removal, it could be suggested that the studied UV/Fenton type process appeared as a viable technique for the treatment of wastewater containing such types of reactive dyes.

Keywords decolorization; mineralization; reactive dye; UV/Fenton

INTRODUCTION

Reactive dyes have been identified as the emerging compounds in textile dye effluents and the environment as well. These types of dyes, due to hydrolysis effect, easily lead to the high portion of unfixed dyes which have been usually washed off during the dyeing process. Moreover, reactive dye wastewater has limited biodegradability in an aerobic environment. Therefore, the conventional aerobic wastewater treatment plants cannot be used for the decolorization of these wastewaters (1–3). Consequently, it is necessary to develop systems which are much more effective than those adopted in the conventional purification processes. An intensive research has been addressed to this aim pointing out the prominent role of a special class of oxidation techniques defined as advanced oxidation processes (AOPs) which usually operate at ambient temperature and pressure (4,5). Oxidative degradation of dyes has recently received considerable attention since colored aromatic compounds have proven to be degraded effectively by a variety of homogeneous and heterogeneous AOPs. The Fenton and Fenton "like" processes employing iron salts with the assistance of UV irradiation have been studied due to the high oxidation ability of various organic compounds, including dyes (6–11). The capability of the Fenton process for the oxidative degradation of organic dyes is attributed to the generation of hydroxyl radicals formed during the catalytic decomposition of hydrogen peroxide in acidic media as represented by the following reaction (5,12):

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

Hydroxyl radicals are extremely reactive oxidant species able to degrade almost all recalcitrant organic compounds under the mild experimental conditions and in short reaction time. In addition, the rate of dye degradation via the Fenton process is strongly accelerated by irradiation with UV-vis light called a photo-Fenton process. When UV light is utilized, the degradation (decolorization and mineralization) is accelerated due to the enhancement of Fe^{2+} regeneration from the additional photo-reduction of Fe^{3+} species and the photolysis of hydrogen peroxide as demonstrated by equations below (13):

$$Fe^{3+} + H_2O + h\nu \rightarrow HO^{\bullet} + Fe^{2+} + H^{+} \qquad (2)$$

$$H_2O_2 + h\nu \rightarrow HO^{\bullet} + HO^{\bullet} \qquad (3)$$

Besides the high efficiency of the UV/Fenton process, it should be emphasized that iron ions are considered as non-toxic species and can be easily separated from treated wastewater by coagulation, while H_2O_2 is consumed during the degradation process and also considered as an environmentally safe compound (14). Nunez et al. (11) studied decolorization and mineralization of reactive azo dyes under Fenton's and UV/Fenton's conditions and found out that the Fenton's process run under solar light was the most effective. Also, Huang et al. (5) published results obtained for comparison of different processes of Fenton type used for the oxidative destruction of reactive dye. The UV/Fenton process allowed even faster and more

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Received 10 December 2009; accepted 9 February 2010.

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complete destruction and yielded with more than 98% of mineralization extent.

The aim of this article is to evaluate the efficiency of the UV/Fenton process for destructive treatment of a commercial reactive dye, C. I. Reactive Blue 2 (RB2) in the prepared aqueous solutions. This dye was chosen as the model compound in this study because of its wide use in the textile industry. The consumption of reactive dyes with similar chemical structure is evident in other dye application industries as well. The influence of different operational parameters of studied UV/Fenton process on RB2 decolorization and mineralization at constant UV irradiation characteristics, pH, and temperature was investigated.

MATERIALS AND METHODS

The monochlorotriazine anthraquinone reactive dye, C. I. Reactive Blue 2, C.N. 61211 (Fig. 1) was obtained from Ciba-Geigy (Switzerland) as commercially available dye and used without further purification. Hydrogen peroxide (H_2O_2 30%, w/w), ferrous sulfate ($FeSO_4 \times 7H_2O$), sulphuric acid (H_2SO_4), and sodium hydroxide (NaOH) were analytical reagent grade, used without any further purification and obtained from Kemika (Croatia). Aqueous solutions containing 25, 50, 100, and 150 $mg\,L^{-1}$ of dye were prepared with distilled water. All experiments were performed in a batch reactor of 0.5 L capacity. The reactor was made of borosilicate glass, with sampling ports on the top, and a magnetic stirrer, and a water jacket for temperature control. A low pressure mercury lamp (125 W, UV-C 254 nm) was placed axial in quartz tube inside of the reactor (15,16) for the performance of the UV/Fenton process. The incident light flux of the UV-C lamp was found to be $I_0 = 3.42 \times 10^{-6}$ $Ein^{-1}L^{-1}s^{-1}$ (17).

Experiments of UV/Fenton oxidation were carried out as follows: a required amount of $FeSO_4 \times 7H_2O$ was added into the 0.5 L of dye solution; pH was adjusted to pH = 3 using 1 M H_2SO_4 and 1 M NaOH, than the required amount of H_2O_2 was added and the UV lamp was turned on; the solution was mixed for 60 minutes. The temperature of the reaction mixture ($T = 23 \pm 1°C$) was kept at constant value by recirculation of cooling water. At specific time intervals 1–2 ml of the sample was withdrawn and analyzed immediately. Decolorization of RB2 during the treatment was monitored using a UV-vis spectrophotometer Lambda EZ 201, Perkin-Elmer (USA) at a maximum absorbance value of 620 nm. The mineralization extent of RB2 in the solution after 1 hour of treatment was determined by the means of total organic carbon (TOC) analysis using a TOC V_{CPN} 5000 A analyzer, Shimadzu (Japan). The initial absorbance at maximal absorption wavelength and TOC values for aqueous solutions containing 25, 50, 100, and 150 $mg\,L^{-1}$ of RB2 were evaluated and presented in Table 1.

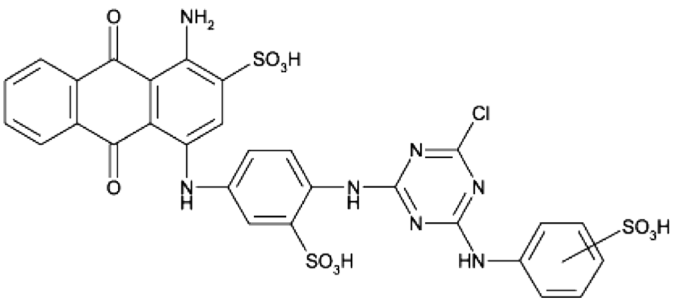


FIG. 1. Chemical structure of RB2.

TABLE 1
Initial absorbance and TOC values for studied RB2 aqueous solutions

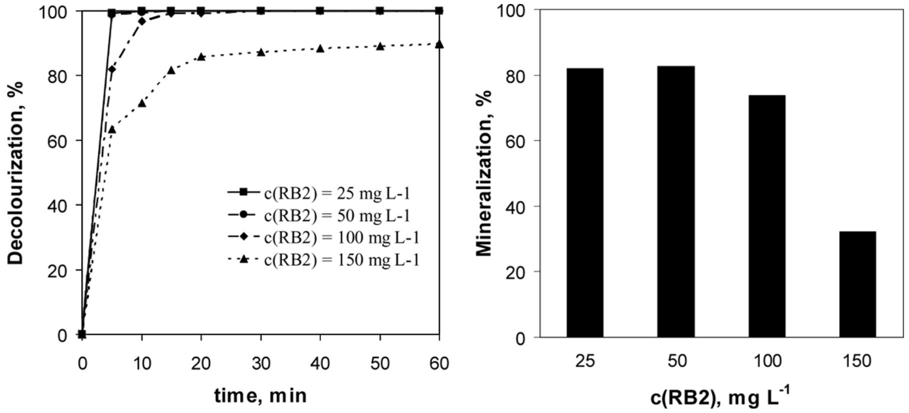
c(RB2), $mg\,L^{-1}$	A_{620}	TOC, $mg\,L^{-1}$
25	7.28	0.293
50	13.8	0.567
100	28.14	1.140
150	42.28	1.735

RESULTS AND DISCUSSION

Degradation of RB2 Dye by UV/Fenton Process

Effect of Initial RB2 Concentration

Under the term "dye degradation" decolorization and mineralization of RB2 in aqueous solution was considered. Decolorization presents destruction of the chromophoric part of the dye molecule while further destruction of organic by-products towards CO_2 and H_2O was considered as mineralization. Since the pollutant concentration is an important parameter in wastewater treatment the effect of initial dye concentration of aqueous solution of RB2 on the efficiency of UV/Fenton process was investigated, using a constant molar ratio of $Fe^{2+}/H_2O_2 = 1{:}5$ ($[Fe^{2+}] = 0.5$ mM, $[H_2O_2] = 2.5$ mM). The influence of the RB2 concentration on decolourization and mineralization efficiency is shown on Fig. 2. It can be seen that the extent of dye degradation decreases with the increase of the initial dye concentration. Within the first 10 minutes of the treatment, the dye solution of 25 $mg\,L^{-1}$ was completely decolorized. By increase of the initial dye concentration to 50 $mg\,L^{-1}$ the decolorization efficiency was not affected. However, a further increase to 100 $mg\,L^{-1}$ required 30 minutes for complete decolorization. When the initial dye concentration was increased to 150 $mg\,L^{-1}$ the complete decolorization was not achieved. Decolorization extents of 71.5% and 89.9% were obtained after 10 and 60 minutes, respectively. Correspondingly, the mineralization efficiency was negatively affected. Mineralization extents obtained after



(a) (b)

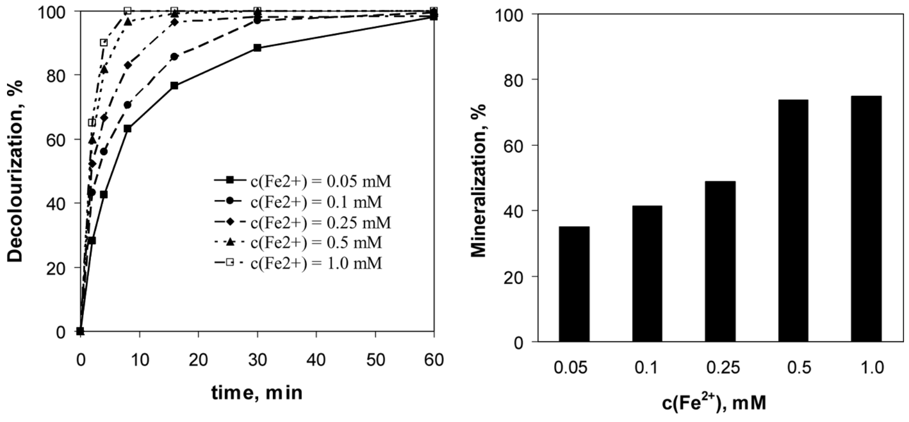
FIG. 2. Effect of RB2 concentration on decolorization (a) and mineralization (b) by UV/Fenton process. Experimental conditions: $[Fe^{2+}] = 0.5\,mM$; $[H_2O_2] = 2.5\,mM$; pH = 3; t = 60 min.

60 minutes of treatment decreased from 81.9% to 32.2% with increasing of the initial dye concentration from 25 to $150\,mg\,L^{-1}$. By increasing the initial dye concentration at constant Fe^{2+} and H_2O_2 concentration, the pollutant/oxidant ratio was also increased, thereby negatively influencing the overall process efficiency. Moreover, due to the lower transparency at higher dye concentration, the penetration of photons into the solution is attenuated. Consequently, the generation of OH radicals through reactions (2) and (3) is inhibited, thus enhancing the negative effect of the initial dye concentration (10). In our previous research dealing with the degradation of azo dye by UV/Fenton "like" process, the maximal mineralization extent was obtained in 45 minutes of the treatment. It was shown that prolonging the treatment time has not contributed to the further increase of the mineralization extent (18).

Further experiments were performed using the initial dye concentration of $100\,mg\,L^{-1}$ as the maximal threshold concentration reaching complete decolorization within 1 hour of treatment by the UV/Fenton process.

Effect of Initial Fe^{2+} Concentration

The amount of ferrous ions is one of the main parameters influencing the Fenton type processes due to its fundamental role in the generation of OH radicals, Eq. (1). According to the literature (19), a minimal threshold concentration of ferrous ions which allows the reaction to proceed within a reasonable period of time regardless of the concentration of organic material is $3–15\,mg\,L^{-1}$. Therefore, in this study the effect of Fe^{2+} concentration on dye degradation efficiency has been investigated within the range from 0.05 to 1.0 mM. Experiments were performed at concentrations of $[RB2] = 100\,mg\,L^{-1}$ and $[H_2O_2] = 2.5\,mM$. The results presented in Fig. 3a, indicate that the degradation rate increases with the increase in the initial Fe^{2+} concentration. The addition of Fe^{2+} in the



(a) (b)

FIG. 3. Effect of Fe^{2+} concentration on decolorization (a) and mineralization (b) of RB2 by UV/Fenton process. Experimental conditions: $[RB2] = 100\,mg\,L^{-1}$; $[H_2O_2] = 2.5\,mM$; pH = 3; t = 60 min.

range from 0.05 mM to 0.5 mM increases color removal from 63.2% to 96.8% within 8 min of the treatment time. However, it should be noted that the complete decolorization was achieved after 1 hour of treatment in all cases. The increase of ferrous concentration within the investigated range yielded with the increase of final mineralization extents from 35% to 73.8%. From the results presented in Fig. 3b, it can be concluded that further addition of iron above 0.5 mM becomes inefficient. When present in excess, ferrous ions may act as hydroxyl radical scavenger according to the reaction presented by Eq. (4). Although typical for the Fenton reagent, this ferrous threshold concentration depends on wastewater characteristics and therefore should be optimized for each particular treatment system (10,20).

$$Fe^{2+} + HO^{\bullet} \rightarrow HO^{-} + Fe^{3+} \qquad (4)$$

Effect of Initial H_2O_2 Concentration

Figure 4 shows the relationship between the degradation of the dye and the initial concentration of H_2O_2. The objective of this evaluation was to select the most effective dosage of H_2O_2 in the UV/Fenton process within the investigated concentration range of 0.3 – 3.0 mM. Increase of H_2O_2 concentration positively affected the decolorization of RB2. When 0.3 mM H_2O_2 was applied, the decolorization extent after 8 minutes of treatment was 51.7%, while 3.0 mM yielded with 100% of color removal at the same treatment period. From Fig. 4a, it can be observed that by the addition of 2.5 and 3.0 mM of H_2O_2 complete decolorization was achieved. The mineralization extents after 1 hour of treatment increased from 10.8% to 73.7% with an increase of H_2O_2 dosage within the investigated range, Fig. 4b. It should be noted that similar results were obtained with 2.5 and 3.0 mM H_2O_2. Such behavior is due to the increase in hydroxyl radical concentration by addition of H_2O_2, Eq. (3). However, H_2O_2 may react with OH radicals through the reaction given by Eq. (5) (10,20). This scavenging effect was more pronounced at higher H_2O_2 dosages. As a consequence, the further addition of H_2O_2 resulted in low or no TOC reduction.

$$H_2O_2 + HO^{\bullet} \rightarrow H_2O + HO_2^{\bullet} \qquad (5)$$

Within the investigated ranges of operating parameters 0.5 mM Fe^{2+} and 2.5 mM H_2O_2 were found to be the most effective in degradation of 100 mg L^{-1} of RB2 in aqueous solution. At these parameters, after 1 hour of treatment by UV/Fenton process 100% of color and 73.7% of TOC removal was achieved.

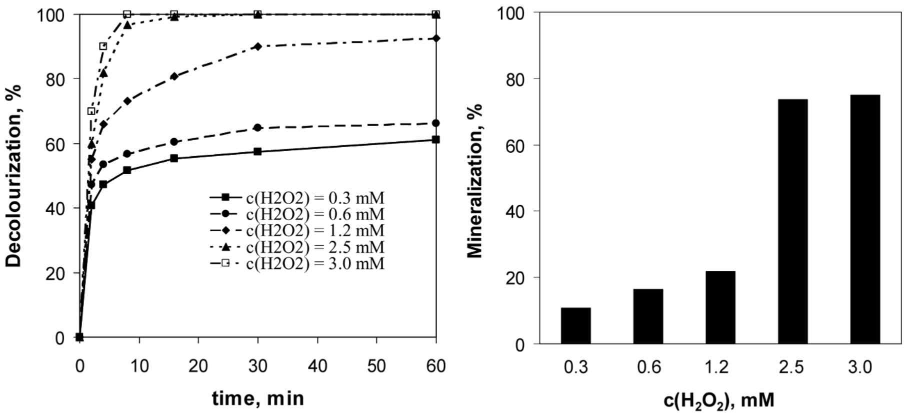
Effect of Different Experimental Conditions

To evaluate the efficiency and contribution of UV light, ferrous salt and hydrogen peroxide in overall dye degradation efficiency by UV/Fenton process, experiments were carried out under the following conditions:

1. dye/H_2O_2,
2. dye/UV,
3. dye/UV/H_2O_2,
4. dye/UV/Fe^{2+},
5. dye/Fe^{2+}/H_2O_2 (Fenton process),
6. dye/UV/Fe^{2+}/H_2O_2 (UV/Fenton process).

The results of RB2 decolorization and mineralization are presented in Figs. 5a. and 5b, respectively.

It can be observed that the decolorization extent of RB2 solution obtained after the treatment by H_2O_2 alone is almost negligible. These results speak in favor of the stability of the dye molecule despite the relatively high oxidation power of hydrogen peroxide (21), and suggest that for the degradation of RB2 a stronger oxidant such OH



(a) (b)

FIG. 4. Effect of the addition of H_2O_2 on decolorization (a) and mineralization (b) of RB2 by UV/Fenton process. Experimental conditions: [RB2] = 100 mg L^{-1}; [Fe^{2+}] = 0.5 mM; pH = 3; t = 60 min.

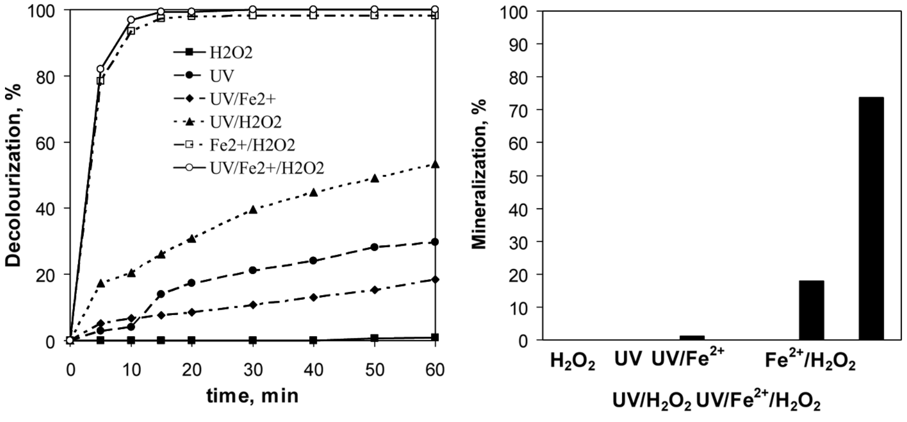


FIG. 5. Effect of the different conditions on the decolorization (a) and mineralization (b) of RB2. Experimental conditions: [RB2] = 100 mg L^{-1}; [Fe^{2+}] = 0.5 mM; [H_2O_2] = 2.5 mM; pH = 3; t = 60 min.

radical is required. When RB2 solution was irradiated by UV light for 60 minutes, 29.7% decolorization extent was achieved while TOC was unaffected. In direct photolysis, the contaminant to be destroyed must absorb the incident radiation and undergo degradation from its excited state. It can be concluded that anthraquinone chromophore is a photoactive part of RB2 dye molecule. By absorption of UV light, a photochemical reaction was initiated resulting with the formation of colorless organic by-products. The combined action of UV irradiation and H_2O_2 resulted with 53.3% bleaching within 1 hour of treatment. The observed enhancement in the decolorization efficiency by UV/H_2O_2 process in comparison to direct UV-photolysis can be contributed to the activity of hydroxyl radicals formed through the reaction given by Eq. (3). With the simultaneous application of UV irradiation and ferrous salt observed decolorization extent was 18.4% in 60 min, which is lower than that obtained by UV irradiation alone. Maloney et al. (22) suggested that the presence of iron may contribute to attenuation of UV irradiation either directly or by interactions with dissolved organics, thus negatively influencing the overall process efficiency. A slight reduction in the TOC content can be contributed to the possible ferric coagulation (Fig. 5b). In this work Fenton and UV/Fenton processes were found to be the most effective according to the obtained 98.3% and 100% decolorization extents within 1 hour of treatment.

It should be noted that among the investigated systems, only the application of Fenton reagent yielded with the reduction of TOC after 1 hour of treatment. Since in these processes a complete decolorization has been achieved it can be concluded that the initial step in oxidative degradation of RB2 is the cleavage of anthraquinone chromophore and formation of colorless by-products which can be further degraded to CO_2 and H_2O. The positive effect of UV irradiation on dye degradation efficiency by Fenton type processes can be observed from the results presented in Fig. 5b. The UV assistance resulted in significant increase in the mineralization extent, from 17.9% to 73.8%. Such behavior is an obvious result of the synergistic effect of the several mechanisms; direct photolysis of RB2, oxidative degradation of the parent dye molecule, and its colorless by-products by OH radicals. Those radicals can be generated through the main Fenton reaction (Eq. 1), as well as photo-assisted reduction of ferric ion (Eq. 2), and photolysis of hydrogen peroxide (Eq. 3). Moreover, dye degradation by-products tend to form Fe^{3+}-complexes that are stable in the dark. By the assistance of UV irradiation, formed complexes are usually degraded, allowing Fe^{3+} ions to participate in the Fenton catalytic cycle, thus positively influencing the overall process efficiency (Eq. 6) (20,23).

$$Fe^{3+}\text{-complex} + h\nu \rightarrow Fe^{2+} + \text{organic products} \qquad (6)$$

From all of the above presented results it can be concluded that hydroxyl radicals play a major role in decolorization and particularly mineralization of RB2.

Kinetic Analysis

The decolorization kinetic of each applied system can be assessed on the basis of the calculated values of pseudo-first order decolorization rate constants as presented in Table 2. Initial decolorization rate constants were determined from the slope of $-\ln(A/A_0)$ vs t (min) plots, where A_0 and A are dye absorbances at zero and t time, respectively. It can be seen that calculated decolorization rate constants for Fenton and UV/Fenton process are three order magnitudes higher than in other studied processes which is in accordance with previously discussed results. According to the results presented in Table 1, the relative decolorization rates follow the increasing order: $H_2O_2 <$ UV/Fe^{2+}

TABLE 2
Pseudo-first order decolorization rate constants

Oxidation process	k_d, min^{-1}
H_2O_2	–
UV	6.2×10^{-3}
UV/Fe^{2+}	3.4×10^{-3}
UV/H_2O_2	1.18×10^{-2}
Fe^{2+}/H_2O_2	0.3328
UV/Fe^{2+}/H_2O_2	0.3323

$< UV < UV/H_2O_2 < Fe^{2+}/H_2O_2$ (Fenton) $\approx UV/Fe^{2+}/H_2O_2$. In all the studied systems (except H_2O_2) certain concentrations of OH radicals were generated. The value of decolorization rate increases with the increase of OH radicals formed which was confirmed by the calculated values of the decolorization rate constants.

UV-vis Spectra of the RB2 Solution during Degradation

To study decolorization of RB2, UV-vis absorption spectra of 100 mg L^{-1} RB2 solution were recorded before and during the treatment by UV/Fenton process at the parameters determined as optimal ones, $[Fe^{2+}] = 0.5$ mM, $[H_2O_2] = 2.5$ mM, (Fig. 6). Before the treatment, UV-vis spectra of RB2 consisted of two main characteristic bands. One is in the UV region (254 nm) and another one is in the visible region (620 nm). The band in the UV region is characteristic for aromatic rings, whereas the band in the visible part of the spectra is connected to anthraquinone part of the RB2 dye (Fig. 1). When 100 mg L^{-1} of RB2 solution was oxidized by the UV/Fenton process it was clearly observed that the intensity of absorption maximum in visible region (620 nm) disappears within 2 to 10 min and the solution becomes colorless. It was observed that the maximum in UV region at 254 nm also diminishes but at a lower rate than that of the visible one. So, from Fig. 6 it can be observed that even though complete decolorization was achieved, a partial degradation of the aromatic parts of the molecule was obtained.

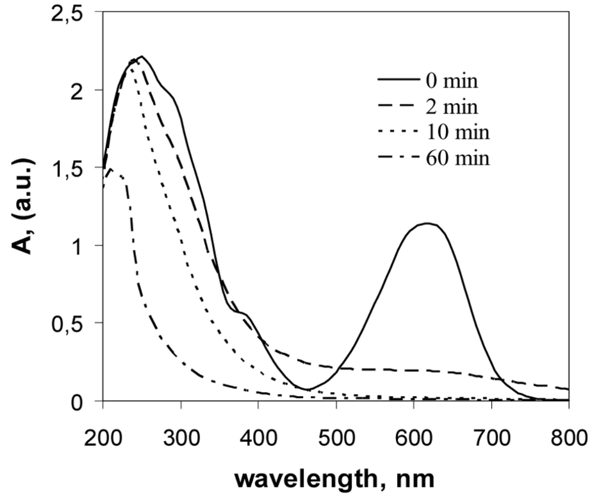


FIG. 6. UV-vis absorption spectra of RB2 solution during degradation with UV/Fenton process. Experimental conditions: $[RB2] = 100$ mg L^{-1}; $[Fe^{2+}] = 0.5$ mM; $[H_2O_2] = 2.5$ mM; pH = 3; t = 60 min.

CONCLUSIONS

Degradation of monochlorotriazine anthraquinone reactive dye RB2 with homogeneous UV/Fenton process was studied. The influence of initial dye, Fe^{2+} and H_2O_2 concentrations at pH 3, $T = 23 \pm 1°C$ and constant UV irradiation characteristics within 60 minutes of treatment time were determined. The molar ratio of the Fenton reagent 1:5 was found as optimal for the studied photo-Fenton process. By the application of 0.5 mM Fe^{2+} and 2.5 mM of H_2O_2 the complete color removal and mineralization extent of 73.8% were achieved. Hydroxyl radical degradation mechanism plays a significant role in both decolorization and mineralization processes. It was found out that the UV/Fenton process can be successfully applied for the oxidative degradation of RB2 in aqueous solution.

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