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Decolorization of antraquinonic dye, C.I. Acid Blue 25, in aqueous solution by direct UV irradiation, UV/H_2O_2 and UV/Fe(II) processes

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In this work, the decolorization of C.I. Acid Blue 25 (AB25), a commercially important anthraquinonic dye, by direct UV irradiation alone and UV/H_2O_2 and UV/Fe(II) processes was investigated. Experiments were conducted in batch mode using a low-pressure mercury lamp emitting mainly at 253.7 nm. For direct UV photolysis, the decolorization rate increased with decreasing pH and initial dye concentration. The decolorization of AB25 was investigated using UV irradiation in the presence of H_2O_2 as function of hydrogen peroxide concentration, dye concentration and pH. Additionally, the effect of salts on the bleaching of this dye was also studied. Decolorization rate was increased in the presence of UV/H_2O_2 compared to UV irradiation alone. The results showed that the bleaching increases as the initial H_2O_2 concentration increased up to a certain limit at which hydrogen peroxide reduced the bleaching by scavenging the OH radicals. The bleaching of AB25 decreased when the dye concentration increased. Decolorization rate by means of Fe(II) homogeneous photocatalysis under UV irradiation was increased compared to direct UV irradiation alone. The best working condition was found for an initial Fe(II) concentration of 30 mg L^{-1}. The results revealed that the tested advanced oxidation processes are very effective for the decolorization of AB25 in aqueous solutions.

1. Introduction

Synthetic dyes are widely used in the textile, cosmetic, printing, drug and food processing industries. Factories generate waste effluents containing color compounds which cause serious environmental problems. Dyes can have acute and/or chronic effects on exposed organisms depending on the exposure time and dye concentrations [1]. Dyes are inherently highly visible meaning that concentrations as low as 0.005 ppm capture the attention of both the public and the authorities [2]. Dyes absorb and reflect sunlight entering water and so can interfere with the growth of bacteria and hinder photosynthesis in aquatic plants [3]. There are many structural varieties of dyes, such as azo, anthraquinone, phthalocyanine, direct, reactive, dispersive, acidic, basic, neutral and metal complex [4–6]. The three most commonly used groups of dyes are azo, anthraquinone and phthalocyanine [7,8].

A wide range of methods has been developed for the removal of synthetic dyes from waters and wastewaters to decrease their impact on the environment. Insufficient treatment of wastes of the dyestuff industries leads to dye contamination of the environment such as soil and natural water bodies. One promising strategy is the use of advanced oxidation processes (AOPs), which are based on the generation of hydroxyl radicals ($^{\bullet}OH$) that can oxidize the contaminants powerfully and non-selectively.

Ultraviolet photolysis combined with hydrogen peroxide (UV/H_2O_2) is one of the most appropriate AOP technologies for removing dyes from water because it may occur in nature itself. This process involves the production of reactive and non-selective hydroxyl radicals and can initiate the decolorization reactions by reacting with the dye molecules. The use of hydrogen peroxide shows some advantages such as the complete miscibility of H_2O_2 with water, the stability and commercial availability of hydrogen peroxide, no phase transfer problems, no sludge formation, simplicity of operation and lower investment costs [9]. Additionally, the present work is focused on the adoption of an innovative AOP which uses oxygen as oxidative agent. This process is based on the same photocatalytic iron cycle on which photo-Fenton relies (Fe(II)/Fe(III)). However, the presence of oxygen under the UV irradiation employed makes possible a photochemical reoxidation of Fe(II) followed by the photolysis of Fe(III) aquacomplex which generates OH radicals [10].

The objectives of the present investigation were the decolorization of AB25 by direct UV irradiation alone and UV/H_2O_2 and UV/Fe(II) processes. Anthraquinonic dyes represent the second most important class of commercial dyes after azo-compounds and are mainly used for dying wool, polyamide and leather. AB 25 was chosen because of its known wide applications (wool, nylon,

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silk, paper, ink, aluminum, detergent, wood, fur, cosmetics, biological stain) and it often serves as model compound for removing anthraquinonic dyes from aqueous solutions. The effects of operating parameters, such as initial dye concentration, pH, hydrogen peroxide dosage, Fe(II) concentration on the decolorization kinetics were studied. Additionally, the effect of salts on the bleaching of dye by UV/H_2O_2 process was investigated.

2. Materials and methods

2.1. Materials

Acid Blue 25 (abbreviation: AB25; C.I. number: 62055; chemical class: anthraquinone; molecular formula: $C_{20}H_{13}N_2NaO_5S$) was used as a model solute. Acid Blue 25 [1-amino-9,10-dihydro-9,10-dioxo-4-(phenylamino)-2-anthracenesulfonic acid, monosodium salt] (dye content 45%, molecular weight: 416.39 g mol^{-1}) was purchased from Aldrich and was used as received. The molecular structure of Acid Blue 25 (C.I. 62055) is shown in Fig. 1.

The aqueous solutions of AB25 were prepared by dissolving the required amount in distilled water. The pH of the solution was adjusted using sodium hydroxide (NaOH) or sulfuric acid (H_2SO_4). Hydrogen peroxide (30%, w/w) purchased from Merck was used as received.

Sodium chloride, calcium chloride and sodium bicarbonate were supplied by Acros Organics.

All reagents used in the present study were purchased among the products of high purity (analytical grade).

2.2. Photoreactor

All experiments were conducted in a 200 mL capacity batch photoreactor. The temperature of the solution was monitored using a thermocouple immersed in the reacting medium. The temperature inside the reactor was kept constant (20 °C) by circulating water within the jacket surrounding the reactor. The UV irradiation was provided by a low-pressure mercury lamp (Oriel 6035, 15 mW cm^{-2}) emitting at a predominant wavelength of 253.7 nm. The UV lamp was encased in a quartz tube and immersed in the aqueous solution.

2.3. Procedures

Photolytic decolorization of AB25 was carried out in a cylindrical water-jacketed glass reactor using a constant solution volume of 100 mL. The temperature of the solution was maintained constant at 20 °C with circulating water. During irradiation, the solution was agitated in a constant rate. Aqueous samples were taken from the solution and the concentrations were determined. The concentrations of AB25 in the solution before and during the bleaching were determined using a UV–vis spectrophotometer (Jenway 6405) at 602 nm.

In UV/H_2O_2 or UV/Fe(II) experiments, precise amount of hydrogen peroxide or ferrous sulfate was mixed with the dye solution by means of a magnetic stirrer to obtain a total volume of 100 mL in the photoreactor. Then the lamp was switched on to initiate the reaction. At certain reaction intervals, the concentration of the dye was determined by means of a spectrophotometer.

For studying the effect of pH on dye decolorization, the pH of the dye solution was modified by adding necessary amounts of H_2SO_4 or NaOH. For experiments examining the effects of different salts on the bleaching of AB25, a final concentration of 1 g L^{-1} of various salts (NaCl, $CaCl_2$ and $NaHCO_3$) was added to the dye solution before the addition of hydrogen peroxide. None of the salts used had any effect on the dye spectra in the absence of UV light. The solution was then subjected to UV light and change in absorbance value was noted to determine the decolorization kinetics.

3. Results and discussion

3.1. Direct UV photolysis

3.1.1. Effect of initial dye concentration

The decolorization of AB25 with initial concentration varying from 10 to 150 mg L^{-1} was investigated. The pH values of these dye solutions with different initial concentrations were all about 5.7. Application of UV irradiation to 100 mL of AB25 solution resulted in the decline of the initial concentration (Fig. 2). Irradiation of aqueous AB25 solutions with the initial concentration of 10, 25, 50, 75, 100 and 150 mg L^{-1} for 20 min resulted in decolorization efficiency of 100, 57, 26, 16, 11 and 7%, respectively, reflecting the low bleaching effectiveness at high dye concentration. The results presented in the form of initial rate of AB25 decolorization as a function of initial dye concentration are shown in Fig. 3. It was observed that the initial decolorization rate increased with the increase of initial AB25 concentration. It can be noticed that the bleaching rate does not obey the first kinetic order process. During this study, different runs were compared using the initial decolorization rate (mg L^{-1} min^{-1}), rather than the pseudo-first-order kinetic constant. A 15-fold increase in the initial AB25 concentration leads to a 2-fold increase of the initial rate of the process. The observed slight increase can be explained in terms of the less availability of photons as the color of the solution gets more intense. Thus, a rise in dye concentration induces an inner filter effect and

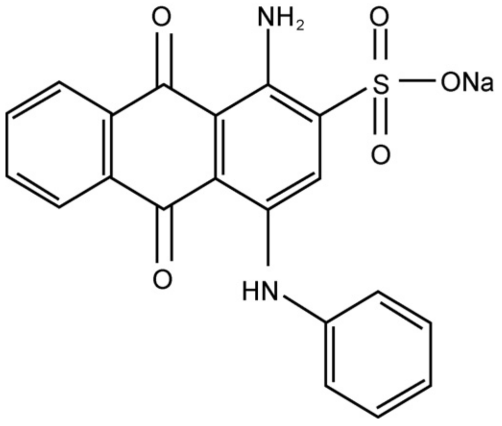

Fig. 1. Chemical structure of Acid blue 25 (AB25).

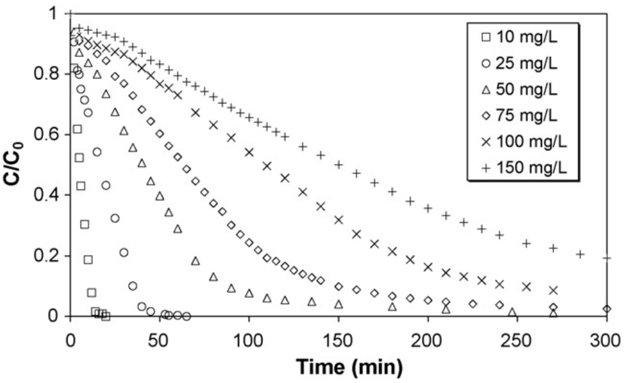

Fig. 2. Kinetics of AB25 decolorization by direct UV irradiation for different initial dye concentrations (conditions: 100 mL of AB25 solution, initial concentration 10–150 mg L^{-1}, pH 5.7, temperature 20 °C).

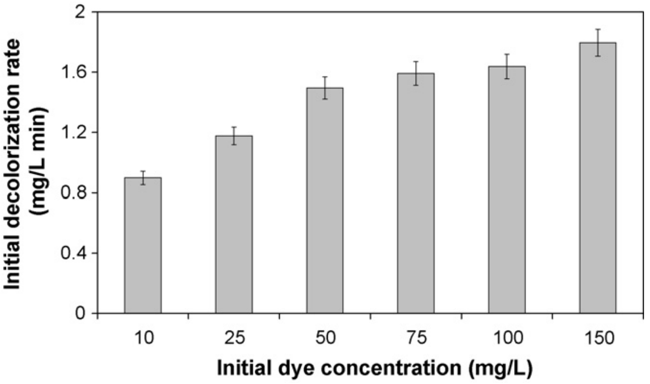

Fig. 3. Effect of dye concentration on the initial rate of AB25 decolorization by direct UV irradiation (conditions: 100 mL of AB25 solution, initial concentration 10–150 mg L^{-1}, pH 5.7, temperature 20 °C).

hence the solution becomes more and more impermeable to UV irradiation.

Similarly, Kusic et al. [11] have indicated that colored solutions of Reactive Blue 49 and Reactive Blue 137 were completely and rather fast decolorized by direct photolysis.

3.1.2. *Effect of pH*

The pH value was considered as one of the important factors affecting dye decolorization by UV irradiation. For an initial dye concentration of 50 mg L^{-1}, the initial decolorization rate at different solution pH values are shown in Fig. 4. It was found that the rate of AB25 decolorization was strongly pH dependent. With the decrease of pH from 5.7 to 1.4, the increased decolorization rates indicate an enhanced photolysis of AB25 at decreasing solution pH. The absorbance data of dye solution determined spectrophotometrically indicated that, change of the initial pH of dye solution has no effect on the λ_{max} of AB25 (pH 1.4–11.8). The initial bleaching rate in acidic solutions (1.4–3) are higher, especially at pH 1.4, and decrease from pH 1.4 to 5.7, and there is nearly no change in the pH range of 5.7–9.3. Relatively higher decolorization rates are observed in basic media (10.5–11.8). This may be because there is a greater facility in the generation of free radicals $R^{\bullet}$ at these pH values. The acceleration of decolorization in strong acidic conditions (pH 1.4) is probably associated with the effect of protonation of negatively charged $-SO_3^-$ group in acidic medium. The results indicated that the photolysis of AB25 molecules was markedly inhibited in anionic form and promoted in neutral form.

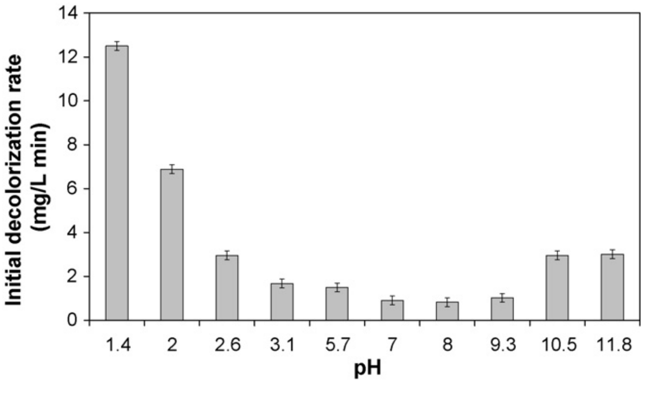

Fig. 4. Initial decolorization rate of AB25 by direct UV irradiation as a function of solution pH (conditions: 100 mL of AB25 solution, initial concentration 50 mg L^{-1}, pH 1.4–11.8, temperature 20 °C).

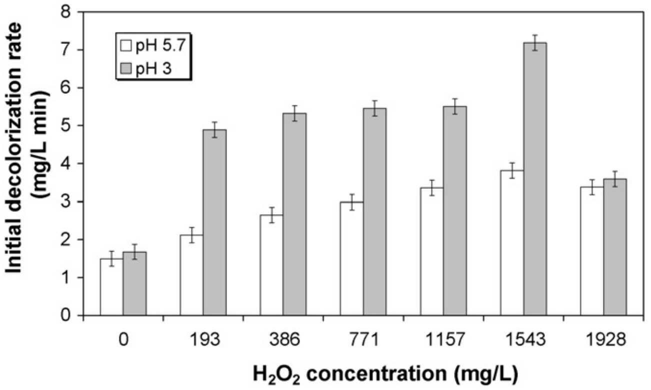

Fig. 5. Initial decolorization rate of AB25 as a function of H_2O_2 concentration (conditions: 100 mL of AB25 solution, initial concentration 50 mg L^{-1}, temperature 20 °C, H_2O_2 concentration 0–1928 mg L^{-1}).

3.2. *UV/H_2O_2*

3.2.1. *Effect of H_2O_2 dosage*

To optimize the decolorization kinetics of the dye, a systematic study varying the concentration of H_2O_2 was conducted. The initial decolorization rate of AB25 at different H_2O_2 concentrations in the presence of UV irradiation for two different pH values (3.1 and 5.7) is shown in Fig. 5. As can be seen from this figure, the decolorization rate for both pH values increases with increasing H_2O_2 concentration until 1543 mg L^{-1}. The reason for this is that at higher concentrations of hydrogen peroxide, more UV is absorbed by hydrogen peroxide and the rate of generation of hydroxyl radicals is higher, which leads to a higher rate of decolorization of AB25. The decolorization of the dye solution is due to the reaction of hydroxyl radicals generated by hydrogen peroxide in solution upon irradiation by UV light (reaction (1)).

$$H_2O_2 + h\nu \rightarrow 2^{\bullet}OH \quad (1)$$

Since hydroxyl radicals are very strong oxidizing reagents, they can react with the dye molecule to produce intermediates which can cause the decolorization of the original solution. The enhancement of H_2O_2 concentration above 1543 mg L^{-1} retards the process. This is due to the fact that at high concentrations, the solution undergoes self-quenching of $^{\bullet}OH$ radicals by added amounts of H_2O_2 to produce $HO_2^{\bullet}$ radicals (reaction (2)). Additionally, $^{\bullet}OH$ radicals generated at high concentration, dimerize to H_2O_2 (reaction (3)). The peroxy radicals produced as a result of reaction (2) can also enter in other reaction pathways (reactions (4)–(6)) [12]. A similar behavior was reported for the decolorization of rhodamine B [13,14] and reactive azo dyes [15] by UV/H_2O_2 process.

$$H_2O_2 + {}^{\bullet}OH \rightarrow H_2O + HO_2^{\bullet} \quad (2)$$

$$^{\bullet}OH + {}^{\bullet}OH \rightarrow H_2O_2 \quad (3)$$

$$HO_2^{\bullet} + H_2O_2 \rightarrow {}^{\bullet}OH + H_2O + O_2 \quad (4)$$

$$2HO_2^{\bullet} \rightarrow H_2O_2 + O_2 \quad (5)$$

$$HO_2^{\bullet} + {}^{\bullet}OH \rightarrow H_2O + O_2 \quad (6)$$

As a result, there is a critical hydrogen peroxide concentration for the AB25 bleaching in UV/H_2O_2 process. At this favorable concentration, the rate of decolorization is maximized. For subsequent decolorization studies, the concentration of H_2O_2 was kept at an optimum level of 1543 mg L^{-1}.

3.2.2. *Effect of initial dye concentration*

The effect of initial dye concentration varying from 10 to 150 mg L^{-1} on the decolorization of AB25 by UV/H_2O_2 pro-

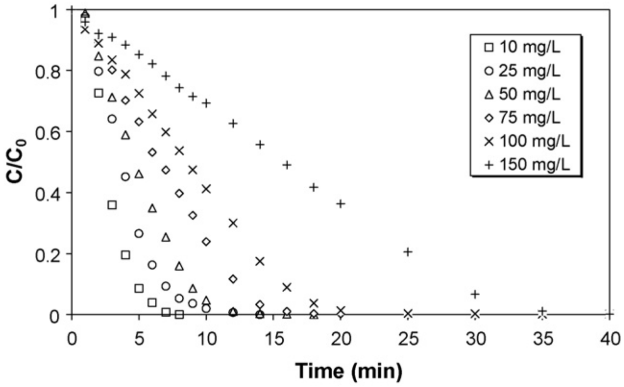

Fig. 6. Kinetics of AB25 decolorization by UV/H_2O_2 process for different initial dye concentrations (conditions: 100 mL of AB25 solution, initial concentration 10–150 mg L^{-1}, pH 5.7, temperature 20 °C, H_2O_2 concentration 1543 mg L^{-1}).

cess was investigated using hydrogen peroxide concentration of 1543 mg L^{-1}. With different initial concentrations, the pH values of these dye solutions were all about 5.7. Fig. 6 shows that the complete bleaching of 10 mg L^{-1} AB25 solution was possible after 8 min of irradiation, the decolorization time with an increasing initial dye concentration to 150 mg L^{-1}, increased to 40 min. The decolorization efficiency after 8 min was 100, 95, 84, 60, 46 and 26% for initial dye concentration of 10, 25, 50, 75, 100 and 150 mg L^{-1}, respectively. The decrease in the decolorization kinetics with the increase of dye initial concentration (Fig. 6) can be explained by an internal filter effect. An increase in dye concentration induces a rise of the internal optical density and the solution becomes more and more impermeable to UV irradiation. By this way, hydrogen peroxide can only be irradiated by a smaller portion of UV light to form lower free radicals and the color removal decreases. Additionally, at higher concentrations of AB25, higher intermediates are formed. These intermediates are also highly reactive towards hydroxyl radicals. Thus, AB25 and its intermediates compete effectively for hydroxyl radicals, reducing the bleaching efficiency.

Fig. 7 depicts the initial decolorization rate versus concentration for each concentration. It can be noticed that, the higher the substrate concentration, the higher the initial bleaching rate, but the decay of AB25 decolorization was not found to follow pseudo-first-order reaction kinetics. With an increase in the solution concentration of dye the probability of OH radical attack on dye molecules would increase, thus leading to an increase in the initial decolorization rate.

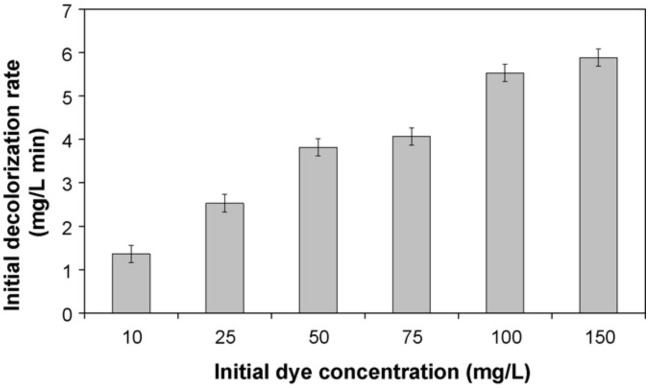

Fig. 7. Effect of dye concentration on the initial decolorization rate of AB25 by UV/H_2O_2 process (conditions: 100 mL of AB25 solution, initial concentration 10–150 mg L^{-1}, pH 5.7, temperature 20 °C, H_2O_2 concentration 1543 mg L^{-1}).

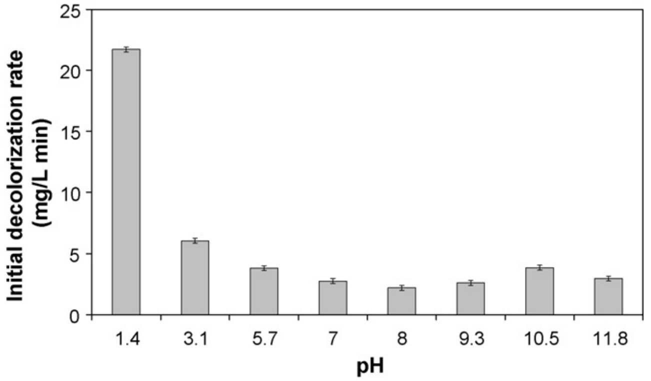

Fig. 8. Initial decolorization rate of AB25 as a function of solution pH for the UV/H_2O_2 process (conditions: 100 mL of AB25 solution, initial concentration 50 mg L^{-1}, pH 1.4–11.8, temperature 20 °C, H_2O_2 concentration 1543 mg L^{-1}).

3.2.3. *Effect of pH*

The effect of pH on the decolorization efficiency in the pH range 1.4–11.8 was studied. The initial concentrations of AB25 and hydrogen peroxide used were, respectively, 50 and 1543 mg L^{-1}. The AB25 initial decolorization rate at various pH values is shown in Fig. 8. The decolorization efficiency of the AB25 solution decreased from pH 1.4 to 8. After this value (pH 8), the efficiency increased slightly as the pH increased toward 11.8. The low bleaching under alkaline conditions may be due to the hydrogen peroxide deprotonation with formation of the H_2O_2/HO_2^- equilibrium ($pK_a = 11.75$ [16]). The HO_2^- species react with a non-dissociated molecule of H_2O_2 according to reaction (7), which leads to dioxygen and water, instead of producing hydroxyl radicals under UV irradiation. Therefore, the instantaneous concentration of $^{\bullet}OH$ is lower than expected.

$$HO_2^- + H_2O_2 \rightarrow H_2O + O_2 + OH^- \quad (7)$$

Furthermore, the deactivation of OH radicals is more important when the pH of the solution is high. The reaction of OH radicals with HO_2^- is approximately 100 times faster than its reaction with H_2O_2. Additionally, the self-decomposition rate of hydrogen peroxide (reaction (8)) increased in alkaline conditions leading to the decrease of OH radical formation [16].

$$2H_2O_2 \rightarrow 2H_2O + O_2 \quad (8)$$

Some researchers [16,17] have indicated that hydroxyl radicals react with dye through hydrogen abstraction mechanism. This may be true for the decolorization of AB25 because of the higher rate at pH 1.4. The enhancement of bleaching at pH 1.4 is probably associated with the effect of protonation of negatively charged $-SO_3^-$ group. In the deprotonated form, dye exhibits low reactivity due to the absence of a labile hydrogen atom [16]. Indeed, in the molecule of AB25, the nitrogen atom carries an H atom and this labile H atom makes the molecule of dye especially vulnerable to attack by OH radicals [16].

3.2.4. *Effect of salts*

The effects of various salts on the decolorization of AB25 by UV/H_2O_2 process were examined for an initial dye concentration of 50 mg L^{-1} and hydrogen peroxide concentration of 1543 mg L^{-1} at 20 °C. The AB25 decolorization in the presence of 1 g L^{-1} of NaCl, $CaCl_2$ and $NaHCO_3$ was presented in Fig. 9. Both NaCl and $CaCl_2$ drastically decrease of the removal of dye. This is due to the hydroxyl radical scavenging effect of chloride ion. The anion Cl^- is able to react with hydroxyl radical leading to inorganic radical ions (reactions (9) and (10)). These inorganic radical anions show a much lower reactivity than $^{\bullet}OH$, so that they do not take part in

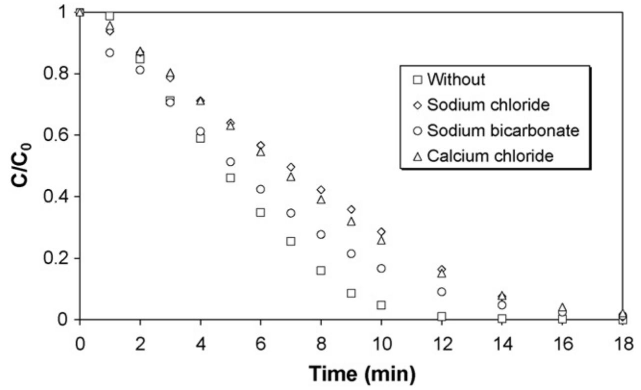

Fig. 9. Effects of various salts on AB25 decolorization by UV/H_2O_2 process (conditions: 100 mL of AB25 solution, initial concentration 50 mg L^{-1}, temperature 20 °C, H_2O_2 concentration 1543 mg L^{-1}, salt concentration 1 g L^{-1}).

the dye decolorization. There is also a drastic competition between the dye and the anions with respect to ${}^{\bullet}OH$ [18].

$$Cl^- + {}^{\bullet}OH \rightarrow HClO^{\bullet -} \tag{9}$$

$$HClO^{\bullet -} + H^+ \rightarrow Cl^{\bullet} + H_2O \tag{10}$$

$$HCO_3{}^- + {}^{\bullet}OH \rightarrow H_2O + CO_3{}^{\bullet -} \tag{11}$$

In the presence of $NaHCO_3$, the slight decrease of dye decolorization was due to the formation of $CO_3{}^{\bullet -}$ (reaction (11)), reactive toward organic compounds although less than ${}^{\bullet}OH$ [18,19].

3.3. UV/Fe(II)

To investigate the effect of ferrous ion on the decolorization of AB25 in the presence of UV irradiation, a series of experiments were conducted by varying Fe(II) concentration from 10 to 50 mg L^{-1}. The experiments were carried out using an initial dye concentration of 50 mg L^{-1}, a solution pH of 3 and a temperature of 20 °C. The AB25 initial decolorization rate for various concentrations of Fe(II) was presented in Fig. 10. The decolorization rate of AB25 increased with increasing the amount of iron up to 30 mg L^{-1}. The enhancement of dye bleaching is due to the oxidation of Fe(II) to Fe(III) (reaction (12)) which can generate H_2O_2 and OH radicals (reactions (13) and (14)) [20].

$$Fe(II) + O_2 + h\nu \rightarrow Fe(III) + O_2{}^{\bullet -} \tag{12}$$

$$Fe(II) + O_2{}^{\bullet -} + 2H^+ \rightarrow Fe(III) + H_2O_2 \tag{13}$$

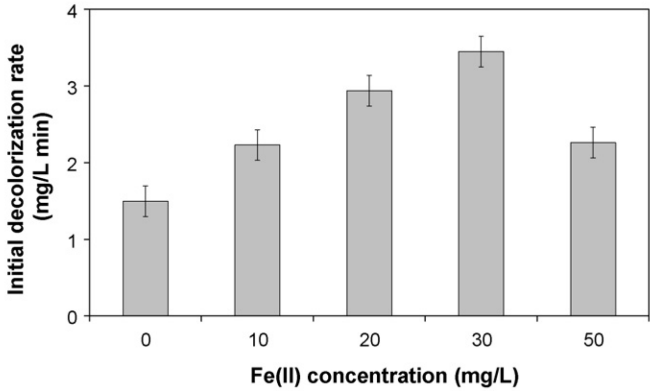

Fig. 10. Effects of Fe(II) concentration on the initial decolorization rate of AB25 in the presence of UV irradiation (conditions: 100 mL of AB25 solution, initial concentration 50 mg L^{-1}, pH 3, temperature 20 °C, Fe(II) concentration 0–50 mg L^{-1}).

$$Fe(II) + H_2O_2 \rightarrow Fe(III) + {}^{\bullet}OH + OH^- \tag{14}$$

$$Fe(II) + {}^{\bullet}OH \rightarrow Fe(III) + OH^- \tag{15}$$

Addition of iron above 30 mg L^{-1} decreases the decolorization of dye. This is due to the fact that, at a Fe(II) concentration higher than the optimum, the initial formation rate of hydroxyl radicals was so high that a lot of hydroxyl radicals were consumed by Fe(II) before they could be utilized effectively for the decolorization of dye [21]. Moreover, it resulted in brown turbidity that hindered the absorption of the UV light required for photolysis [22].

4. Conclusion

The present work shows that the UV/H_2O_2 and UV/Fe(II) processes provide good performance in the decolorization of anthaquinonic dye in water. In the direct UV irradiation experiments, the decolorization rate increased with decreasing initial dye concentration, but the initial bleaching rate increased by increasing initial AB25 concentration. The best pH for dye decolorization was obtained in strong acidic pH (1.4). Bleaching rate was increased in the presence of UV/H_2O_2 compared to direct UV irradiation alone. The results showed that the decolorization increases as the initial H_2O_2 concentration increased up to a certain point at which hydrogen peroxide reduced the bleaching by scavenging the ${}^{\bullet}OH$ radicals. The higher the dye concentration, the higher the initial bleaching rate is. The higher decolorization was obtained at pH 1.4. The effects of various salts on the decolorization of AB25 by UV/H_2O_2 process were examined. Both NaCl and $CaCl_2$ drastically decrease the decolorization rate. In the presence of $NaHCO_3$, the slight decrease of decolorization was due to the formation of $CO_3{}^{\bullet -}$. Decolorization rate using Fe(II) homogeneous photocatalysis under UV irradiation was increased compared to direct UV irradiation alone. The best working conditions were obtained for an initial Fe(II) concentration of 30 mg L^{-1}. The results of the present investigation indicate that the UV/H_2O_2 and UV/Fe(II) techniques are highly efficient for the decolorization of AB25 in aqueous solutions.

The best favorable conditions for getting the higher decolorization rate by using the three studied oxidation processes are: strong acidic pH (1.4), H_2O_2 concentration of 1543 mg L^{-1} and initial Fe(II) concentration of 30 mg L^{-1}.

This investigation has discussed the decolorization of only one acid anthraquinonic dye (Acid Blue 25) but this method could be used for the decolorization of other acid anthraquinonic dyes such as Acid Blue 40, 41, 51, 53, 129 and 230 that have very similar chemical structures with Acid Blue 25.

Acknowledgement

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