Desalination and Water Treatment
www.deswater.com

doi: 10.1080/19443994.2014.988657

57 (2016) 4100–4109
February

Taylor & Francis
Taylor & Francis Group

Degradation of anthraquinonic dye in water by photoactivated periodate

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Received 3 May 2014; Accepted 7 November 2014

ABSTRACT

In this work, the degradation of Acid blue 25 (AB25), an anthraquinonic dye and a widely used colorant in many industries, by photoactivated periodate (UV/IO_4^-) process was investigated. Experiments were conducted in batch mode using a low-pressure mercury lamp emitting mainly at 253.7 nm. The effects of the operational conditions such as periodate ions concentration, initial dye concentration, initial pH of the solution, and tert-butanol addition on the degradation of AB25 were examined. The results have shown that the degradation rate was increased in the presence of UV/IO_4^- compared to UV irradiation alone. The degradation rate increased with the increase in periodate concentration to 1 mM and decreased slightly at higher values (>1 mM). The degradation of AB25 decreased when the dye concentration increased. The extent of degradation was found to be inversely proportional to the initial concentration of dye. AB25 degradation was significantly affected by varying the initial solution pH. The fastest degradation was obtained at natural pH (6.4). The degradation of the dye was not appreciably affected by the addition of tert-butanol, known as an effective scavenger of $^{\cdot}OH$ radicals, even at high concentration. Thus, the degradation of AB25 by photoactivated periodate technique was not dominated by an $^{\cdot}OH$ pathway.

Keywords: Acid blue 25; Advanced oxidation process; Periodate; Ultraviolet irradiation

1. Introduction

The dye pollutants taken from various industries play an important role in the environmental contaminations because of their resistance to biological degradation. It is evaluated that 1–15% of the dye is lost during dyeing processes and released into wastewater [1], which results a serious issue for the industry and create an important threat to the environment [2]. Several chemical and physical processes such as chemical precipitation, coagulation, electrocoagulation, adsorption on activated carbon, and reverse osmosis are used for color removal from dye-bearing effluents. However, these processes merely transfer the contamination from one stage to another to produce secondary wastes and leave the main problem unsolved [3,4]. Recent developments of chemical treatment of wastewater resulted in a considerable improvement in the oxidative degradation of organic compounds dissolved in aqueous media [5–8]. Among these methods mentioned, advanced oxidation processes that are focused on the hydroxyl radicals' production ($^{\cdot}OH$) may oxidize contaminants.

Lately, the photoactivated periodate (UV/IO_4^-) system was suggested as a modern advanced oxidation technology, which was appeared to remove aqueous

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organic compounds in many studies [9–11]. Even though, the periodate ion (IO_4^-) itself is considered as a famous oxidant whose reduction potential was determined to be +1.60 V, oxidation reactions initiated by this ion are selective and significantly slow compared with those involving $^{\cdot}OH$ [12]. Periodate is effectively applicable to the oxidation of the compounds in which the carbonyl group or hydroxyl group is adjacent to a second carbonyl or hydroxyl group, such as α-diols, α-ketols, α-diketones, and α-ketonic aldehydes. Even if the selective reactivities of periodate are substrate with specific functional groups, UV light can extremely improve the reactivity and minimize the periodate oxidation reaction's selectivity because periodate produces $^{\cdot}OH$ and other reactive radical and non-radical oxidants, through the photodecomposition that occurs when it absorbs UV light at wavelengths of less than 300 nm. Although the photochemistry of the periodate ion in the aqueous stage has been extensively examined and relatively well elucidated in several previous studies [13–17], some reports on the application of photoactivated periodate to water treatment are remained. Additionally, although the degradation of dyes by photocativated periodate have been examined [18–23], comprehensive studies on the effects of process variables are yet to be carried out.

The main aim of this work concerned the degradation of Acid blue 25 (AB25) by UV/IO_4^- process. After azo compounds, anthraquinone dyes are classified as the second most important class of commercial dyes and are mainly used for dyeing wool, nylon, and leather. The reasons that stand behind the AB25 choice are its wide applications known (wool, nylon, silk, paper, ink, aluminum, detergent, wood, fur, cosmetics, and biological task) as well as its use as a model compound for removing anthraquinone dyes in aqueous media. The effects of process parameters such as periodate concentration, initial dye concentration, initial solution pH, and addition of tert-butyl alcohol on the destruction of this dye were examined.

2. Materials and methods

2.1. Materials

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

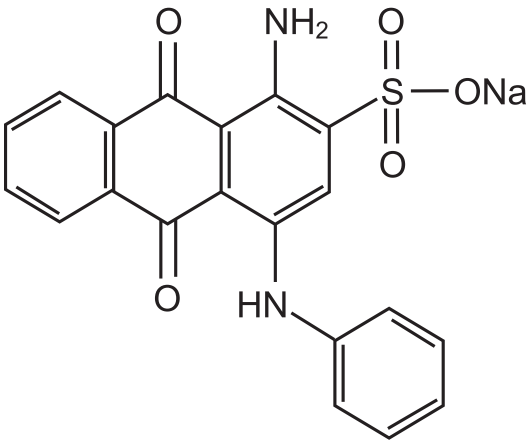

Fig. 1. Chemical structure of AB25.

The aqueous solutions of AB25 were prepared by dissolving the required amount in distilled water. The solution's pH was adjusted using sodium hydroxide (NaOH) or sulfuric acid (H_2SO_4).

Sodium periodate was supplied by Acros Organics. All reagents used in the present study were purchased among the products of high purity (analytical grade).

2.2. Reactor

Experiments were conducted in a 500 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 at 20 ± 1°C by circulating water within the jacket surrounding the reactor. The UV irradiation was provided by a low-pressure mercury

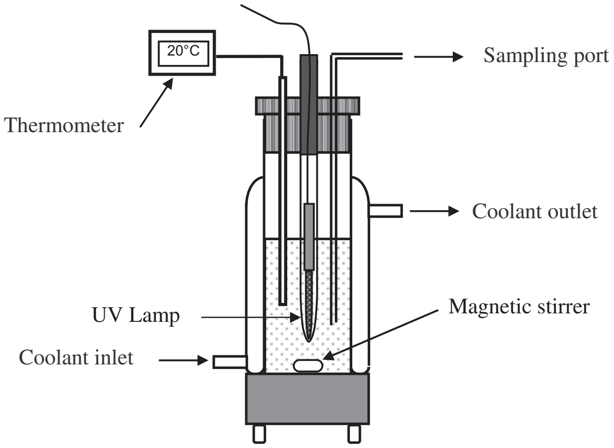

Fig. 2. Scheme of the photoreactor used for AB25 degradation.

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 (Fig. 2).

2.3. Procedure

Photolytic degradation of AB25 was carried out in a cylindrical water-jacketed glass reactor using a constant solution volume of 300 mL. The temperature of the solution was maintained constant at 20℃ with circulating water. During irradiation, the solution was agitated at 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 degradation were determined using a UV–visible spectrophotometer (Jenway 6405), at 603 nm.

In UV/IO_4^- experiments, precise amount of sodium periodate was mixed with the dye solution using a magnetic stirrer to obtain a total volume of 300 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 using a spectrophotometer. In order to study the effect of pH on dye degradation, the pH of the dye solution was modified by adding necessary amounts of H_2SO_4 or NaOH. The absorbance data of dye solution determined spectrophotometrically indicated that change in the initial pH of dye solution has no effect on the λ_{max} of AB25 (pH 2–10).

All experiments were conducted in triplicate and the mean values were reported.

2.4. Analytical method

A well-known procedure for determining AB25 concentrations, based on Beer's law calibration plots, was applied using a UV–visible spectrophotometer (Jenway 6405). The wavelength resolution and the bandwidth were, respectively, 1 and 0.5 nm. The maximum absorption wavelength was determined as equal to 602 nm.

Chemical oxygen demand (COD) was valued according to the method presented by Thomas and Mazas [24] by means of a dichromate solution (Aldrich) as the oxidizer in a strong acid medium. Test solution (2 mL) was transferred into the dichromate reagent, and digested at 150℃ for 2 h. The optical density for the color change of dichromate solution was determined using a UV–visible spectrophotometer (Jenway 6405).

3. Results and discussion

3.1. Photodecomposition of periodate ions

Application of UV irradiation to 300 mL of periodate ions aqueous solution with an initial concentration of 0.5 mM at natural pH caused a decrease in the periodate concentration with time. The UV–visible spectrophotometry was used to evidence the photodecomposition of periodate ions under UV irradiation. Fig. 3 shows the evolution of the shape of the UV spectra as a function of UV irradiation time. As can be observed from these spectra, before the decomposition, the absorption spectrum of periodate ions in water was characterized by a band with a maximum absorption at 221 nm. Following a period of irradiation, this characteristic absorption band decreased in intensity and underwent a change in their spectral shape, indicating the decomposition of periodate ions.

The photolysis of periodate in aqueous solution have been extensively studied, and the reactions involved in periodate photolysis have been discussed in several studies [13–17]. In spite of the complicated nature of periodate photolysis, Wagner et al. [15] proposed the simple reaction mechanism for the periodate photolysis, where periodate can be photodecomposed by two photo-initiated pathways. Periodate can be photodecomposed to $IO_3^{\bullet}(I^{VI})$ and $O^{\bullet-}$ by one electron transfer (reaction (1) in pathway I) or to IO_3^- (I^{VI}) and $O(^3P)$ by two electron transfer (reaction (9) in pathway II). The $O^{\bullet-}$ generated by reaction (1) is present as its conjugate acid, $^{\bullet}OH$, in the pH range of <11.9 (p*K*a of $^{\bullet}OH$ [25]). On the other hand, the atomic oxygen, $O(^3P)$ generated by reaction (9) rapidly reacts with dissolved oxygen to produce O_3 by reaction (10) [26]. The primary products of periodate photolysis, such as $^{\bullet}OH$, $IO_3^{\bullet}$, and O_3, undergo self-reaction or reactions with other products, as shown in reactions (3)–(8). The reaction mechanism shows that the potential oxidants in the UV/IO_4^-system can be classified into radical species ($^{\bullet}OH$, $IO_3^{\bullet}$, and $IO_4^{\bullet}$) and non-radical species (O_3, IO_4^-, and IO_3^-). It has been suggested that the periodate ions can be decomposed into two reaction schemes [15]:

Pathway I

$$IO_4^-(I^{VII}) + h\nu \rightarrow IO_3^{\bullet}(I^{VI}) + O^{\bullet-} \tag{1}$$

$$O^{\bullet-} + H^+ \rightarrow {}^{\bullet}OH \tag{2}$$

$${}^{\bullet}OH + IO_4^- \rightarrow OH^- + IO_4^{\bullet} \tag{3}$$

$$O_3 + IO_3^{\bullet} \rightarrow IO_4^{\bullet} + O_2 \tag{4}$$

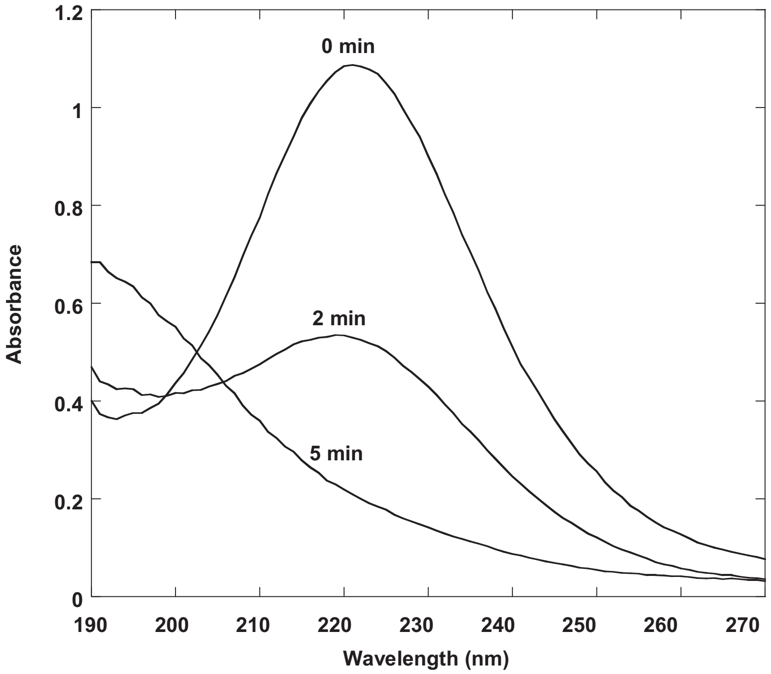

Fig. 3. Changes in the UV absorption spectra observed during UV irradiation of periodate ions aqueous solution (conditions: periodate ions concentration: 0.5 mM, temperature: 20 ± 1 °C).

$$2IO_4^{\bullet} \rightarrow I_2O_8 \quad (5)$$

$$I_2O_8 + H_2O \rightarrow IO_3^- + IO_4^- + 2H^+ + O_2 \quad (6)$$

$$2IO_3^{\bullet} \rightarrow I_2O_6 \quad (7)$$

$$I_2O_6 + H_2O \rightarrow IO_4^- + IO_3^- + 2H^+ \quad (8)$$

Pathway II

$$IO_4^-(I^{VII}) + hv \rightarrow IO_3^-(I^V) + O(3P) \quad (9)$$

$$O_2 + O(^3P) \rightarrow O_3 \quad (10)$$

3.2. *Effect of periodate concentration on AB25 degradation*

Preliminary experiments conducted on the degradation of AB25 (30 mg/L) by direct UV irradiation alone have indicated that complete fading of the dye requires a treatment time of about 150 min. In order to overcome this drawback and achieve better results, it was suggested to examine the degradation of the dye by photoactivated periodate ions.

The effect of different periodate initial concentrations ranging from 0.05 to 5 mM on the degradation of 30 mg/L AB25 solution by UV irradiation is shown in Fig. 4. As can be seen from this figure, the degradation rate of AB25 solutions was considerably enhanced by the addition of periodate ions. The destruction of AB25 increased with increasing periodate concentration and reached a maximum at 1 mM. The increase in periodate concentration above 1 mM retards the process, but the destruction rate was higher than that obtained in the absence of periodate ions. The enhancement of AB25 degradation is due to the formation of highly reactive intermediates, such as $IO_4^{\bullet}$, $IO_3^{\bullet}$, $^{\bullet}OH$, and $IO^{\bullet -}$, produced from the photodecomposition of periodate ions [27,28].

The concentration of AB25 appears to decrease exponentially with time. It is clear that the degradation does not follow a first-order kinetics and cannot be characterized by a single rate constant expressed in 1/time. Consequently, in this work, results were presented using the initial degradation rate (mg/L min), during the first minutes, rather than a pseudo-first-order kinetic constant. Initial degradation rates were computed as $\Delta C/\Delta t$ over the first minutes of UV irradiation from the results showing the evolution of solute concentration as a function of time.

The initial degradation rate of AB25 at different periodate initial concentrations in the presence of UV irradiation is shown in Fig. 5. This figure shows that the degradation rate increased with increasing

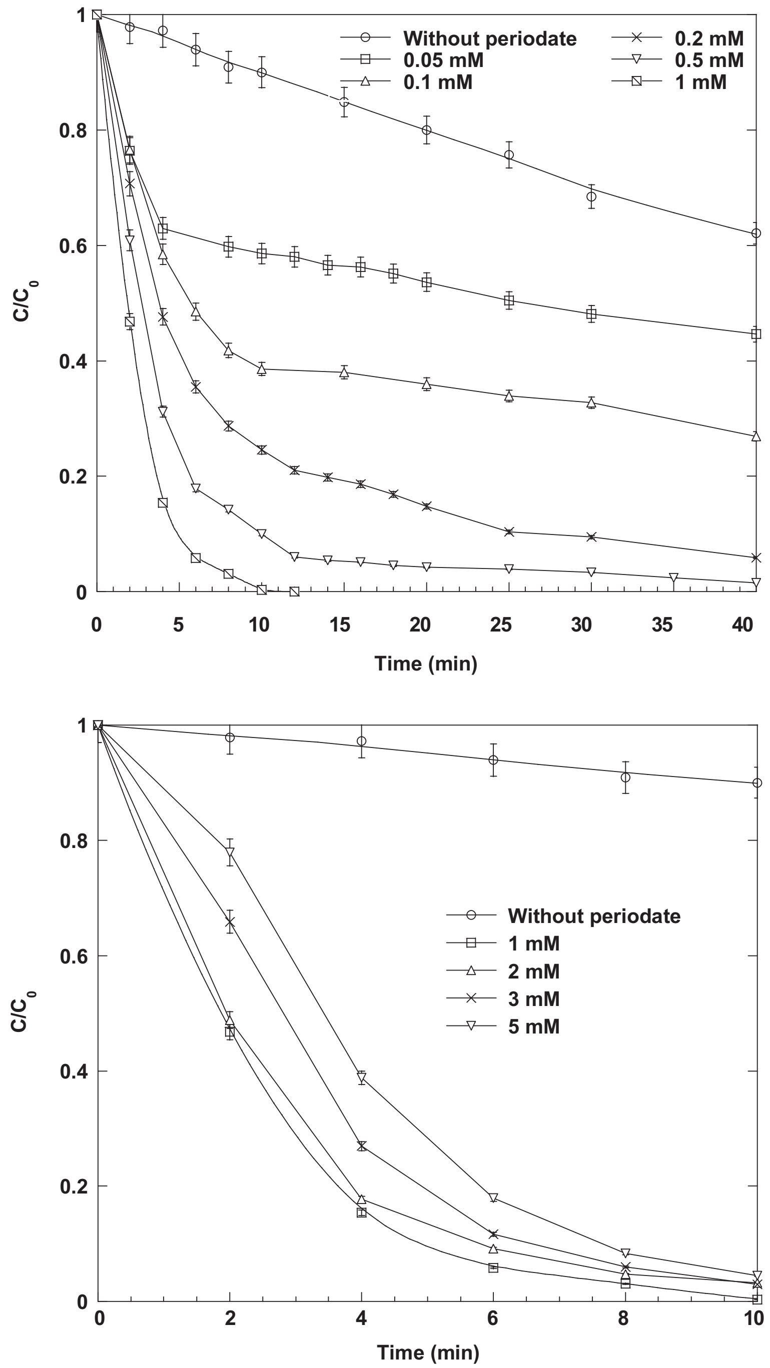

Fig. 4. Effect of initial periodate concentration on AB25 degradation (conditions: initial dye concentration: 30 mg/L, initial pH 6.4, temperature: 20 ± 1℃).

periodate concentration until 1 mM because at higher concentrations of periodate, more UV is absorbed by periodate ions and the rate of generation of reactive species is higher, which leads to a higher rate of degradation of AB25. Besides, the enhancement of periodate concentration above 1 mM retards the process because at high concentrations, the solution undergoes self-quenching of ·OH radicals, since periodate can act as an ·OH scavenger by reaction (5) [28,29].

3.3. *Effect of initial dye concentration on AB25 degradation*

The influence of initial dye concentration varying from 5 to 50 mg/L on the degradation of AB25 by

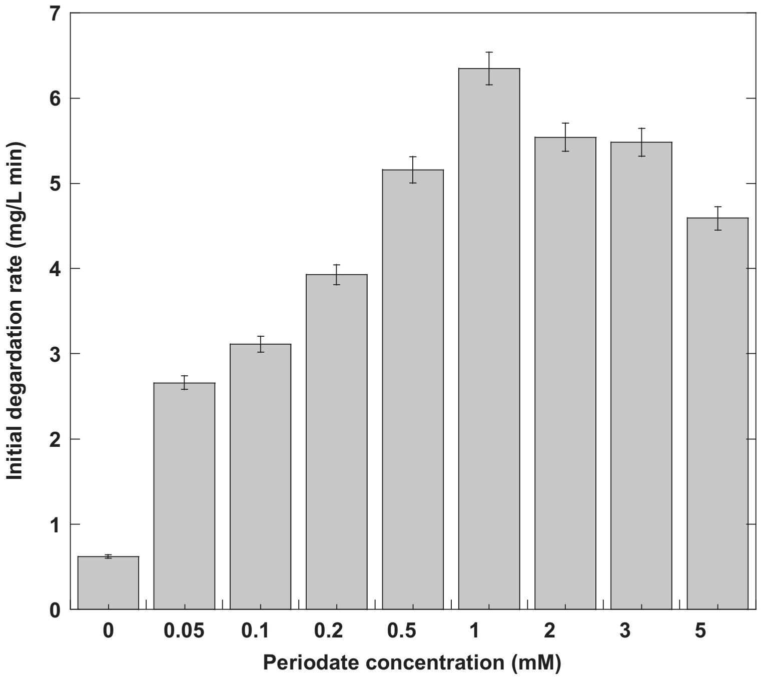

Fig. 5. Initial degradation rate of AB25 as a function of periodate concentration (conditions: initial dye concentration: 30 mg/L, initial pH 6.4, temperature: 20 ± 1°C).

UV/IO_4^- process was studied using periodate concentration of 1 mM. All experiments were conducted using various initial dye concentrations at natural pH (about 6.4). The results presented in the form of initial rate of AB25 degradation as a function of periodate concentration for different dye concentrations are shown in Fig. 6. From this figure, it was observed that the initial degradation rate of AB25 increased with increasing dye concentration from 5 to 50 mg/L. Assuming a constant rate of the formation of highly reactive radicals and non-radicals intermediates produced from the photodecomposition of periodate ions over the used dye concentrations, with an increase in the solution concentration of dye, the probability of attack on dye molecules would increase, thus, leading to an increase in the initial degradation rate. However, the extent of degradation was found to be inversely proportional to the initial concentration of AB25. The complete degradation of 5 mg/L AB25 solution was possible after 5 min, whereas the necessary time for the total removal of dye at an initial concentration of 50 mg/L was 30 min. The degradation efficiency after 5 min was 100, 96.7, 94.2, and 66.4% for initial dye concentration of 5, 10, 30, and 50 mg/L, respectively. The decrease in the degradation kinetics with the increase in dye initial concentration can be explained by an inner filter effect. An increase in dye concentration leads to a rise of the internal optical density and the solution becomes more and more impermeable to UV irradiation. This way, periodate can only be irradiated by a smaller portion of UV light to form lower free radicals and the color removal decreases. Besides, higher intermediates are formed at higher concentrations of AB25. These intermediates are also highly reactive towards reactive species formed by periodate photodecomposition. Thus, AB25 and its intermediates compete effectively for reactive species, reducing the degradation efficiency.

Control experiments were conducted without UV irradiation in order to evaluate the effect of 1-mM periodate ions alone on the degradation of AB25 at various initial dye concentrations ranging from 5 to 50 mg/L. AB25 was slightly degraded by direct reaction with periodate ion. The initial degradation rates of AB25 by periodate ions in the absence of UV irradiation were 0.016, 0.019, 0.029, and 0.055 mg/L min for initial concentrations of dye of 5, 10, 30, and 50 mg/L, respectively. These degradation rates were very low compared with those obtained in the case of photoactivated periodate (1.28, 2.60, 6.35, and 6.52 mg/L min for AB25 concentrations of 5, 10, 30, and 50 mg/L, respectively).

3.4. *Effect of initial pH on AB25 degradation*

To investigate the effect of initial pH on AB25 by UV/IO_4^- technique, 30 mg/L dye solution was

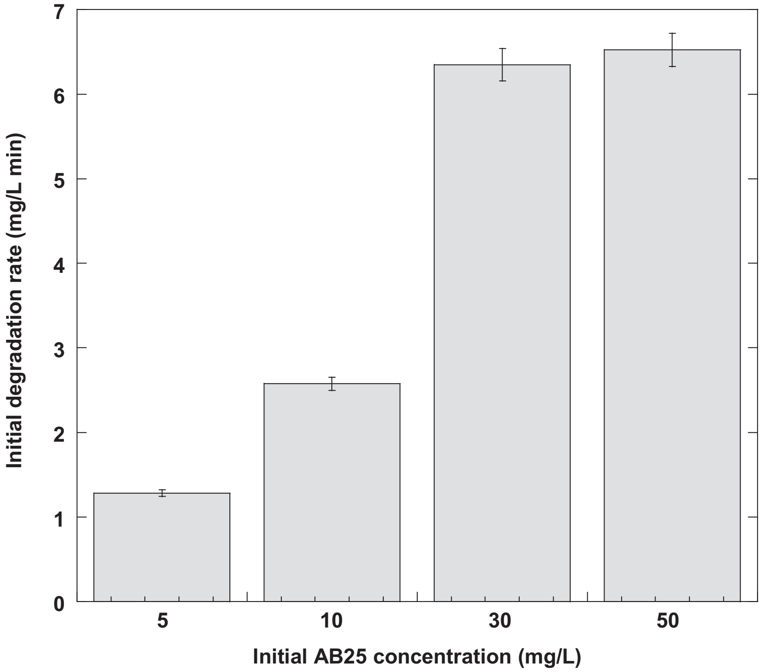

Fig. 6. Effect of dye concentration on the initial degradation rate of AB25 by UV/IO_4^- process (conditions: initial dye concentration: 5–50 mg/L, initial: pH 6.4, temperature: 20 ± 1°C, periodate concentration: 1 mM).

irradiated at various initial pH values (2, 6.4, 7, and 10) in the presence of 1 mM of periodate. The obtained results are shown in Fig. 7. As it was shown from this figure, AB25 degradation was significantly affected by varying the initial solution pH. The fastest degradation was obtained at natural pH (6.4). The degradation rate at pH 10 was lower than those obtained at other pH values. IO_4^- species dominated at pH < 8, whereas the dimerized form, $H_2I_2O_{10}^{4-}$, was the dominant species at a pH higher than 8 [28]. It seems that the ions from the addition of sulfuric acid (H^+ and SO_4^{2-}) or caustic soda (Na^+ and OH^-) in order to adjust the pH, generate a slower degradation of AB25 by UV/IO_4^- technique.

In the control experiments carried out in the absence of UV irradiation, the direct oxidation of AB25 (30 mg/L) by periodate ion (1 mM) at various pH values of 2, 6.4, 7, and 10 was studied. Periodate ions alone without UV irradiation did not significantly affect the oxidation of dye at pH values of 6.4, 7, and 10. The initial degradation rates of AB25 by periodate ions alone without UV irradiation were 0.026, 0.030, and 0.032 mg/L min at initial pH values of 6.4, 7, and 10, respectively. However, AB25 was slightly degraded at pH 2 by its direct reaction with periodate ion alone in the absence of UV irradiation (the initial degradation rate was 0.57 mg/L min). The initial degradation rate of AB25 by photoactivated periodate was 4.25 mg/L min at pH 2, 6.35 mg/L min at pH 6.4, 5.02 mg/L min at pH 7, and 3.29 mg/L min at pH 10.

3.5. Effect of tert-butanol addition

If, indeed, the hydroxyl radical is a major participant in the degradation of AB25 by photoactivated periodate, we should be able to suppress this reaction using a known $^{\cdot}OH$ radical scavenger in the solution. Experiments were performed using tert-butyl alcohol which is a common $^{\cdot}OH$ radical scavenger.

In the present work, the effect of tert-butanol addition on the degradation of AB25 by photoactivated periodate (1 mM) was investigated and the obtained results are shown in Fig. 8. As shown in this figure, the degradation of the dye was not significantly affected by the addition of tert-butanol even at high concentration. This is demonstrating the importance of reactive species other than $^{\cdot}OH$ in the photoactivated periodate process. Similarly, the degradation rate of 4-chlorophenol experienced only a minor decline in the presence of the $^{\cdot}OH$ scavenger, tert-butanol [30]. Therefore, the degradation of AB25 by photoactivated periodate was not dominated by an $^{\cdot}OH$ pathway.

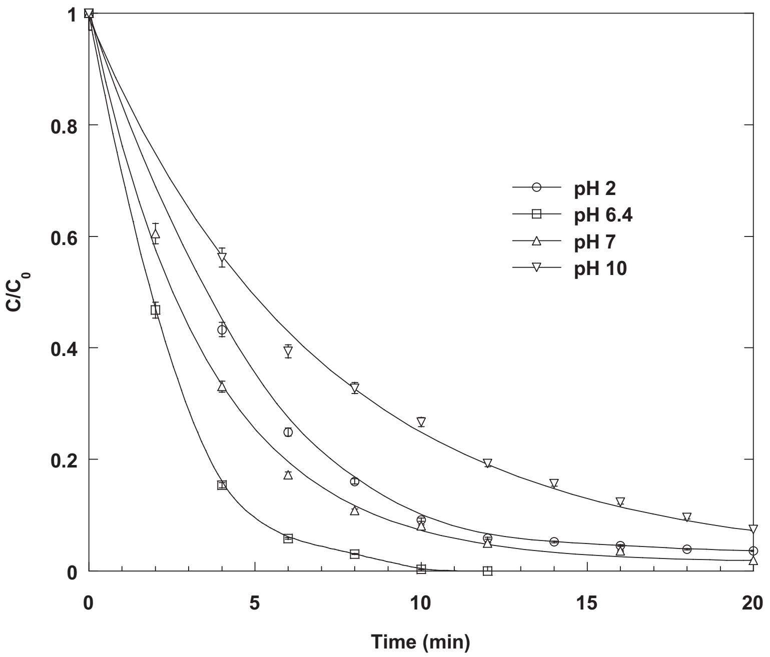

Fig. 7. Effect of initial pH on the degradation of AB25 by UV/IO_4^- process (conditions: initial dye concentration: 30 mg/L, temperature: 20 ± 1˚C, periodate concentration: 1 mM).

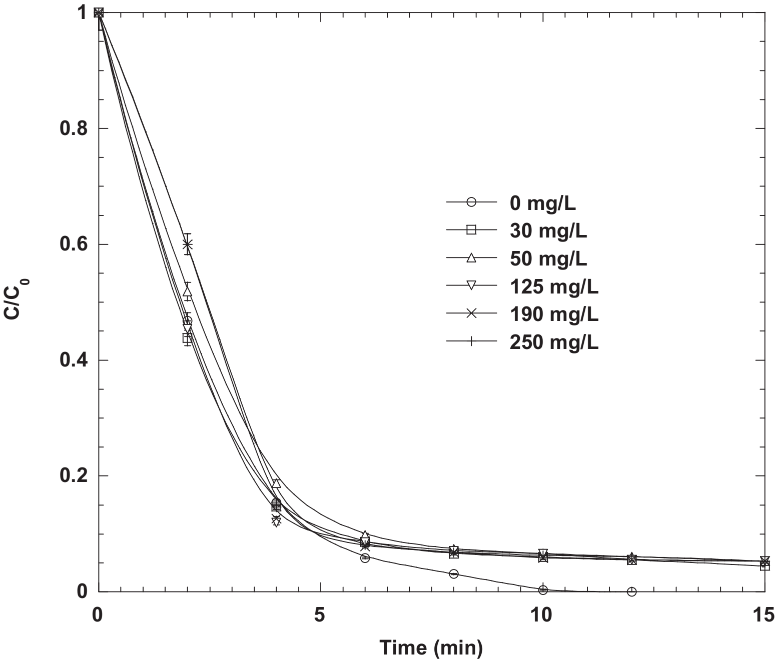

Fig. 8. Effect of tert-butyl alcohol addition on the degradation of AB25 by photoactivated periodate (conditions: initial dye concentration: 30 mg/L, temperature: 20 ± 1˚C, periodate concentration: 1 mM).

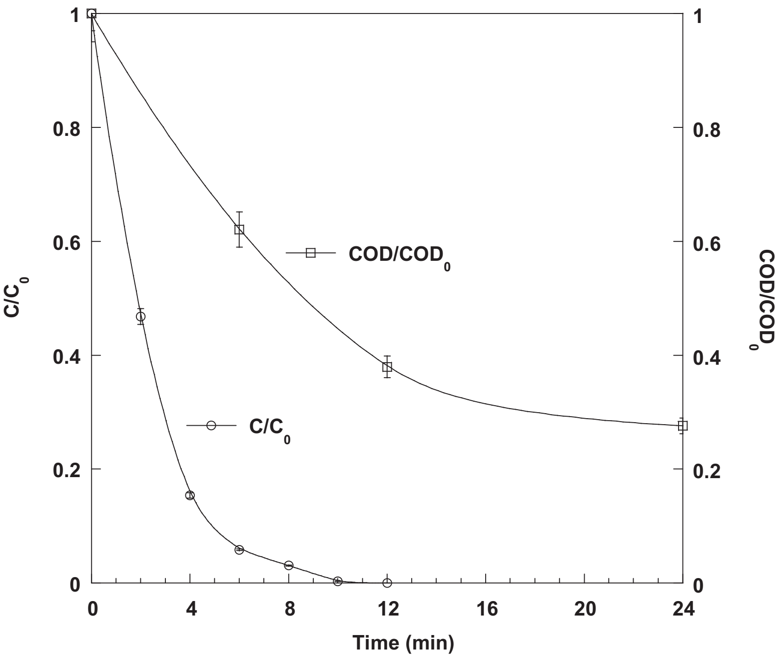

Fig. 9. AB25 removal and COD profile during photoactivated periodate technique (conditions: initial dye concentration: 30 mg/L, temperature: 20 ± 1˚C, periodate concentration: 1 mM).

3.6. COD abatement

It is known that complete degradation of AB25 does not mean that the dye is completely oxidized, and so the degradation of dye in terms of COD removal was investigated. Fig. 9 depicts the evolution of COD/COD_0 during photoactivated periodate treatment of 30 mg/L AB25 in the presence of 1 mM periodate. It can be observed from this figure that the AB25 aqueous solution was degraded after 12 min of irradiation. COD abatement after 12 min of treatment was 62.1% and it was 72.4% after 24 min. COD was not completely removed by the photoactivated periodate process. These results underline the fact that degradation products of AB25 are recalcitrant towards UV/IO_4^- treatment. This is because the intermediate products have very low probabilities of making contact with reactive species. Thus, the UV/IO_4^- action that gives rise to products bearing more hydroxyl (or carboxylic) groups is of low efficiency towards COD abatement.

4. Conclusion

The present work revealed that the UV/IO_4^- process supplies good performance in the degradation of anthaquinonic dye AB25 in water. The degradation rate of dye by UV/IO_4^- process was drastically increased compared to direct UV irradiation alone. This significant improvement in the degradation of the dye is due to the formation of radical ($^{\cdot}OH$, $IO_4^{\cdot}$, and $IO_3^{\cdot}$) and non-radical (O_3, IO_4^-, and IO_3^-) species coming from the photodecomposition of periodate ions that are actively involved in the degradation of AB25. The destruction of AB25 increased with increasing periodate concentration and reached a maximum at 1 mM. The increase in periodate concentration above 1 mM retards the process, but the destruction rate was higher than that obtained in the absence of periodate ions. The initial degradation rate of AB25 increased with increasing dye concentration from 5 to 50 mg/L. However, the extent of degradation was found to be inversely proportional to the initial concentration of dye. AB25 degradation was significantly affected by varying the initial solution pH. The fastest degradation was obtained at natural pH (6.4). The degradation of the dye was not appreciably affected by the addition of tert-butanol, known as an effective scavenger of hydroxyl radicals, even at high concentration. Thus, the degradation of AB25 by photoactivated periodate technique was not dominated by an $^{\cdot}OH$ pathway.

Acknowledgment

This work was financially supported by the Ministry of Higher Education and Scientific Research of Algeria (Project No. J0101120120098).

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