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DECOLORIZATION OF C.I. REACTIVE YELLOW 84 IN AQUEOUS SOLUTION BY ELECTROCOAGULATION ENHANCED WITH OZONE: INFLUENCE OF OPERATING CONDITIONS

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ABSTRACT

We studied the use of a combination of electrocoagulation (EC) and ozonation for the decolorization of the azo dye, C.I. Reactive Yellow 84 (RY84). The effects of initial dye concentration, initial pH, current density, salt concentration, ozone flow rate, temperature and interelectrode distance on the decolorization of RY84 were investigated. The results showed that a synergistic effect was achieved by combining EC with ozone for the decolorization of RY84. Under the experimental conditions, the decolorization rate increased with an increase in current density. However, the decolorization rate decreased with an increase in initial dye concentration and salt concentration. The interelectrode distance did not substantially affect the decolorization rate. Other operating parameters such as initial pH, ozone flow rate and temperature had both positive and negative effects. Over 97% of the color decay was achieved within 10 min reaction time under reasonable experimental conditions. When the decolorization of RY84 was complete, the efficiency of removal of total organic carbon (TOC) was more than 85%.

Keywords: Electrocoagulation, ozone, C.I. Reactive Yellow 84, decolorization, iron electrode

INTRODUCTION

Due to rapid changes in customers' demands, some high quality textiles need to be obtained by using dyestuffs, auxiliaries and other chemicals. Thus, the textile dyeing and finishing industry produces large volumes of discharge effluent, which is considered to be one of the major industrial polluters. Color is a visible pollution; even small quantities ($\geq$0.005 mg l^{-1}) of dyes in water are unacceptable [1]. In addition, the release of effluents containing dyes into the environment can interfere with the transmission of sunlight into flowing liquid, and hence cause perturbations in aquatic life and the food web [2-5]. Under certain conditions, some dyes are toxic, mutagenic and carcinogenic [4, 6]. Azo dyes, characterized by nitrogen-to-nitrogen double bonds (N=N), constitute over 50% of all textile dyes and have been widely used in many industries, such as the production of textiles, paint, ink and cosmetics [7]. It is known that soluble azo dyes, when incorporated into the body, are split by liver enzymes and intestinal microflora into the corresponding aromatic amines, which can cause cancer in humans [8-10]. Therefore, it is necessary to find an effective method for removal of azo dyes from textile effluents.

The traditional techniques used for treating dye effluents include adsorption [11], filtration [12], coagulation [12], precipitation [13], photodegradation [14], biodegradation [15], etc. However, due to the stability of dyes, these methods have several drawbacks, such as high operational costs, limited applicability and ineffective degradation [16-18].

The decomposition of the chromophores in organic dyes can be achieved by injecting a strong oxidizing agent such as ozone. However, a large dose of ozone is usually required for decolorization due to the significant amounts of auxiliary chemicals present, and this is not economically feasible [19]. Among electrochemical treatments, electrochemical oxidation and electrochemical coagulation are more effective than others in decolorizing textile effluents [19]. Some electrochemical processes involve the direct oxidation of pollutants at the anode surfaces. Electrocoagulation (EC) has been proposed in recent years as an effective method for treating textile wastewaters with high removal efficiency. In the study of iron or steel anodes, two mechanisms for the production of the metal hydroxide have been proposed [20]. When ozone is injected into a dyebath, active species are produced to react with the target pollutants. Unfortunately, there has been little research into the

decolorization of azo dyes by the combination of EC and ozonation technology.

In the present work, C.I. Reactive Yellow 84 (RY84), used as a representative azo dye, was decolorized by ozone assisted EC. The effects of initial pH, initial concentration, current density, salt concentration, temperature, ozone flow rate and distance between electrodes have been investigated.

MATERIALS AND METHODS

Materials

RY84 (molecular mass 1628.2 g mol^{-1}), obtained from Wujiang Taoyuan Dyestuffs Plant (Wujiang, China), is over 99% pure and was used without further purification. The solubility of RY84 is 100 g l^{-1} in water at 323 K.

The conductivity of the solutions was increased and adjusted to different values by the addition of K_2SO_4 (Shanghai Zhenxin Reagent Factory, China). The initial pH of the solutions was adjusted by adding KOH (Shanghai Shengyu Chemical Co., Ltd., China) or H_2SO_4 (Quzhou Juhua Group Co., Ltd., China) solutions. Iron plates with the dimensions 40 mm × 50 mm × 1 mm and total areas of 3×10^{-3} m^2 were used as the anode and the cathode.

METHODS

Setup

The system consisted of an electrocoagulator, which was made of plexiglass with the dimensions 50 mm × 50 mm × 100 mm, and an ozone generator (CHYF-3A, rated flow 50 mg min^{-1}, Hangzhou Rongxin Electronic Equipment Co., Ltd.). The ozone flow rate from the generator was monitored with a rotameter. The electrodes were connected to a digital DC power supply equipped with current- and voltage-reading meters to supply the current options. A schematic diagram of the setup is presented in Figure 1.

Analytical Methods

Analytical analysis of RY84 in aqueous solution was performed using an ultraviolet/visible spectrophotometer (T6, Beijing Purkinje General Instrument Co., Ltd.) coupled with a photoelectric detector at 406 nm. The percent color removal efficiency is given by

$$\text{color removal efficiency}(\%) = \frac{abs_0 - abs_t}{abs_0} \times 100(\%) \qquad \text{(i)}$$

where abs_t is the absorbance of RY84 after reaction for t min and abs_0 is the initial absorbance of RY84.

The pH of the solutions were measured with a pHs-25 instrument (Rex Analytical Instrument Co. Ltd., Shanghai, China). Total organic carbon (TOC) was determined with a TOC-V_{CPH} total organic carbon analyzer (Shimadzu, Japan).

Procedure

All the runs were performed at a constant temperature. Before each run, the iron electrodes were immersed for 5 min in dilute H_2SO_4 solution to remove oxide, and then they were rinsed with pure water; finally, they were polished by sands to further remove oxide. For each run, an aqueous solution of 250 ml containing RY84 was placed into the electrolytic cell. During all the experiments, the initial pH, initial concentration, current densities, salt concentration, temperature, ozone flow rate and distance between the electrodes were adjusted to a desired value before the experimental run was started. At the end of the run, all the samples were centrifuged at 2000 rpm for 7 min and then analyzed.

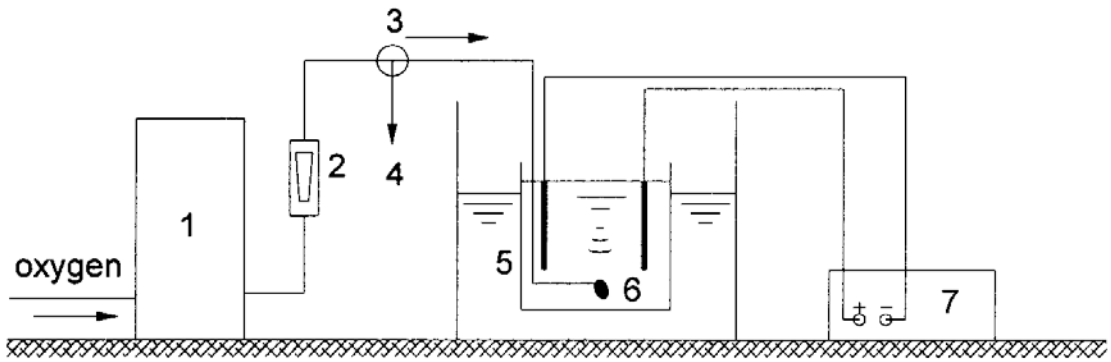

Figure 1. The experimental set-up. (1) Ozone generator; (2) Rotameter; (3) Three-way valve; (4) To input ozone gas detection; (5) Thermostatic bath; (6) Reactor; (7) DC Power.

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RESULTS AND DISCUSSION

Comparison of the Three Processes (EC/O_3, EC, and O_3) for Decolorization of RY84

As shown in Figure 2, the color removal efficiency at 10 min in the O_3, EC and EC/O_3 treatments was 3%, 92% and 97%, respectively. EC alone was more effective than ozonation alone for the color removal of RY84, and the combination of the two resulted in a synergetic increase in the overall rate of decolorization. It is likely that the yield of free radicals is responsible for the increase in decolorization efficiency.

Ozone is a strong oxidant, which reacts with wastewater compounds in two different ways, namely, through direct molecular and indirect radical type chain reactions [21].

$$O_3 + H_2O \rightarrow 2HO_2\bullet \quad \text{(ii)}$$

$$O_3 + HO_2\bullet \rightarrow HO\bullet + 2O_2 \quad \text{(iii)}$$

When ozone is bubbled into a coagulation system, Fe^{2+} catalyzes ozone decomposition to generate hydroxyl radicals. The catalytic O_3/Fe^{2+} system involves direct reaction of Fe^{2+} with ozone to give the intermediate FeO^{2+}, a species that evolves to ·OH [22, 23].

$$Fe^{2+} + O_3 \rightarrow FeO^{2+} + O_2 \quad \text{(iv)}$$

$$FeO^{2+} + H_2O \rightarrow Fe^{3+} + HO\bullet + OH^- \quad \text{(v)}$$

Hence, electrocoagulation coupled with ozone is more efficient for the decolorization of RY84 than using EC alone or ozonation alone.

Effect of the Initial pH

Due to the formation of iron hydroxide complexes during the electrocoagulation process, and the possible importance of radical reactions in the ozonation of dyes, the study of the effect of pH on the color removal efficiency was very important. To determine the optimum pH, the pH was varied from 2.0 to 12.0 as illustrated in Figure 3(a). At low pH (2.0 or 4.0), the color removal efficiency increased with increasing pH, achieving 23% and 87% color decay at 10 min, respectively. By increasing the pH to 6.5 we obtained the highest color removal efficiency (97% after 10 min reaction). For pH values above 6.5, the color removal efficiency decreased. The effect of pH could be attributed [24, 25] to the

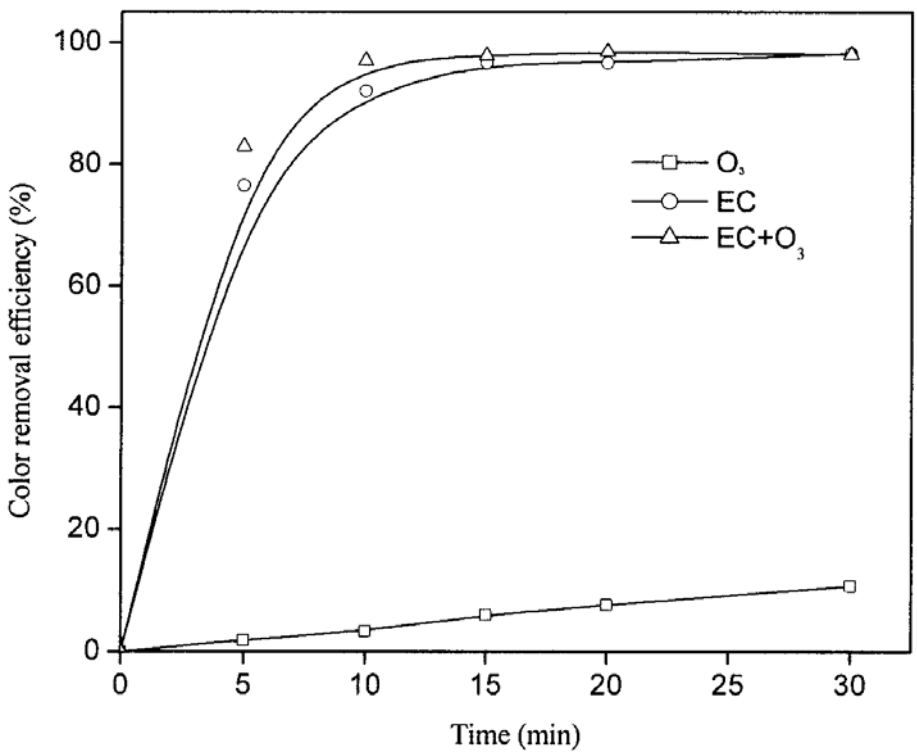

Figure 2. Comparison of the efficiencies of the three processes for decolorization of RY84: RY84 initial concentration 100 mg l^{-1}, K_2SO_4 concentration 2000 mg l^{-1}, pH 6.5, ozone flow rate 20 ml min^{-1}, temperature 30 °C, current density 15 mA cm^{-2}, interelectrode distance 3 cm.

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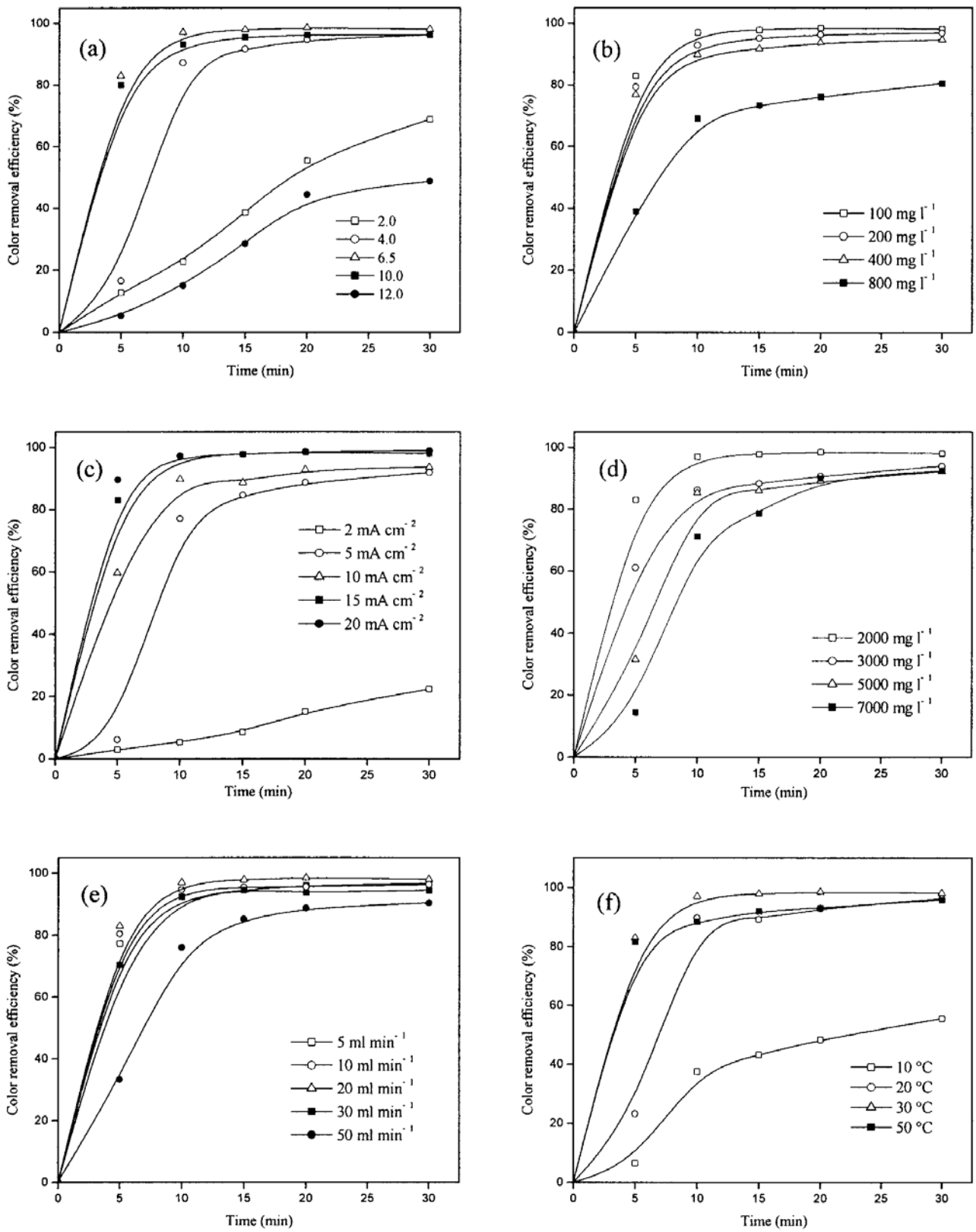

Figure 3. Effect of process variables on the color removal efficiency of RY84. (a) Effect of initial pH; (b) Effect of the initial dye concentration; (c) Effect of the current density; (d) Effect of the salt concentration; (e) Effect of the ozone dose; (f) Effect of the temperature. (RY84 initial concentration 100 mg l^{-1}, K_2SO_4 concentration 2000 mg l^{-1}, ozone flow rate 20 ml min^{-1}, current density 15 mA cm^{-2}, and interelectrode distance 3 cm).

fact that, at acidic pH, the protons in the solution are reduced to H_2 at the cathode, preventing the production of the same proportion of hydroxide ions. Moreover, under these conditions, the concentration of hydroxyl radicals, which can enhance the indirect attack rate on the dye, is lower. However, at high pH, some of the hydroxide ions are oxidized, reducing the production of the same proportion of iron ions at the anode. Meanwhile, the concentration of hydroxyl radicals also decreased, according to equations (iv) and (5). Therefore, the pH that optimized color removal efficiency was found to be 6.5. Under the optimum initial pH conditions, measurements taken before and after the experiments showed that the maximum pH change was less than 2.5 units.

Effect of the Initial Dye Concentration

The effect of initial dye concentration on the color removal efficiency of RY84 was studied for the initial concentrations of 100, 200, 400 and 800 mg l^{-1} (Figure 3(b)). As can be seen, when the concentration of RY84 < 400 mg l^{-1}, the color removal efficiency did not change significantly with an increase in initial concentration. Thus, the effect of the dye concentration was small. At concentrations lower than 400 mg l^{-1}, the adsorption capacity of the flocs is not exhausted and the rate of color removal is relatively constant. However, beyond this concentration, the color removal efficiency falls from 90% to 69% at 10 min reaction time with an increase in the concentration of the dye from 400 to 800 mg l^{-1}. This is likely due to the formation of an insufficient number of iron hydroxide complexes at the electrode and hydroxyl radicals produced by the reaction of Fe^{2+} with ozone to coagulate and oxidize the larger number of dye molecules at higher concentrations [20]. In conclusion, under the experimental conditions, the electrocoagulation process coupled with ozone was more effective in decolorizing at lower concentrations of the dye.

Effect of the Current Density

Current density directly determines both coagulant dosage and bubble generation rates, as well as strongly influencing both solution mixing and mass transfer at the electrodes. In addition, it has an influence on the generation of hydroxyl radicals. Thus a set of experiments was carried out to quantify the impact of the operating current on the color removal efficiency. The experiments were done at 2, 5, 10, 15 and 20 mA cm^{-2}. As shown in Figure 3(c), at 10 min reaction time, the color removal efficiency increased from 5% to 97% with an increase in current density from 2 to 20 mA cm^{-2}. Between 2 and 5 mA cm^{-2}, the effect of the current density was significant. However, when the current density was increased from 15 to 20 mA cm^{-2}, the color removal efficiency only increased slightly. This is because, when the current density increases, the efficiency of ion production at the anode and cathode increases. Hence, there is sufficient floc production in the solution to coagulate the RY84 dye molecules [26]. Moreover, the Fe^{2+} production enhances the generation of hydroxyl radicals, which has the highest oxidation potential (2.8 V) to oxidize the dye molecules. Considering the power consumption, a reasonable current density in our experiments was 15 mA cm^{-2} since it ensures the quickest color removal efficiency at the lowest cost.

Effect of the Salt Concentration

In this study, K_2SO_4 was added as an electrolyte, and the effect of electrolyte concentration on the color removal efficiency of RY84 was studied. The results are shown in Figure 3(d). It can be seen that the color removal efficiency decreased from 97% to 71% as the salt concentration increased from 2000 to 7000 mg l^{-1} at 10 min reaction time. At high salt concentrations, the excess SO_4^{2-} ion is expected to react with the hydroxyl radicals to generate $SO_4^{-}\bullet$, which is less reactive than a hydroxyl radical [27]. Therefore, the excess SO_4^{2-} reduces the effect of the hydroxyl radicals. In addition, the high salt concentration leads to a high ionic strength, which affects the kinetics and equilibria of the reactions between the charged species during EC. Therefore, we selected the salt concentration of 2000 mg l^{-1} as the optimum condition.

Effect of the Ozone Dose

The effect of the dosage of ozone was examined at 5, 10, 20, 30 and 50 ml min^{-1}. Figure 3(e) depicts the trends of color removal efficiency at various doses of ozone. It can be observed that when the dosage of ozone was increased to 20 ml min^{-1}, the color removal efficiency reached approximately 97% at 10 min reaction time. This may be attributed to the fact that there was sufficient concentration of hydroxyl radicals to react with the RY84 dye molecules. With a dosage of ozone greater than 20 ml min^{-1}, the color removal efficiency decreased. It is likely that, at high doses of ozone, the high flow rate of gas through the solution may cause the destabilization of the flocs and, hence, may cause the precipitated dye molecules to become soluble, resulting in a decrease in color removal efficiency.

Effect of the Distance Between the Electrodes

In our experiments, the interelectrode distance does not have a significant effect on the efficiency of color removal as reported by Daneshvar et al. [28]. There may be two reasons for this. Firstly, with increasing distance a slower movement of the ions produced is expected, resulting in a greater opportunity for the ions to aggregate and produce flocs. Hence, the color removal efficiency would increase. Secondly, when the distance between the electrodes increases, the electrostatic attraction force increases to reduce the interaction of the ions with the hydroxyl polymers. Hence, the color removal efficiency would decrease. Therefore, the interelectrode distance has only a slight effect on the color removal of RY84.

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Effect of the Temperature

The effect of the temperature of the solution on the color removal of RY84 was investigated in the range of 10 to 50 °C. It can be seen in Figure 3(f) that when the temperature was lower than 30 °C, the color removal efficiency increased gradually, with the temperature enhancing the EC/O_3 process. The color removal efficiency was about 38% at 10 °C and 97% at 30 °C after 10 min reaction time. When the temperature was varied from 30 °C to 50 °C, the color removal efficiency decreased from 97% to 88%. This could be explained by the generation of hydroxyl radicals in the bulk being facilitated by the increasing temperature due to the higher mass transfer of different species at high temperature that leads to an enhancement of the rate of reaction of the radicals with the dye molecules. Moreover, at high temperature the movement of the ions is enhanced to facilitate reaction with the hydroxyl polymers. However, at higher temperature, the formation of unsuitable flocs or an increase in the solubility of the precipitates would cause the color removal efficiency to decrease [29].

TOC Reduction

The results for dye concentrations reported so far were based on the spectrophotometer analysis of the dye solutions. The reduction of color does not imply necessarily the complete removal of a dye, which is expected to reduce the TOC most effectively. Subsequent to decolorization by a simple EC cell under optimized conditions, the TOC of the treated solution was measured again. The TOC was reduced by more than 85%.

CONCLUSIONS

Electrocoagulation combined with ozonation was employed as an effective way to decolorize RY84 in aqueous solution. When initial RY84 concentration was 100 mg l^{-1}, the best operational parameters were: K_2SO_4 concentration 2000 mg l^{-1}, pH 6.5, ozone flow rate 20 ml min^{-1}, temperature 30 °C, current density 15 mA cm^{-2} and interelectrode distance 3 cm. Over 97% removal of color was achieved after 10 min reaction time. The decomposition of RY84 was accompanied by the removal of total organic carbon. When the decolorization of RY84 was complete, the removal efficiency of TOC was more than 85%. We found that ozone can enhance the rate of decolorization of RY84 during the electrocoagulation process. The effects of operational variables on the decolorization of RY84 by EC and ozonation treatment, either alone or in combination, need to be further studied. More work is needed to determine the precise mechanisms for the electrocoagulation enhanced with ozone process reported here, but the clear synergy achieved by the use of the combined techniques may form the basis of important techniques for environmental treatment.

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