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Kinetic modeling of Congo red dye decolorization by US/O_3 process: nonlinear regression analysis

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ABSTRACT

The decolorization and degradation of Congo red dye in the aqueous solution were studied by a combination of sonication and ozonation. The effect of operational parameters such as initial dye concentration, pH value, sonication density and gas flow rate on the decolorization rate has been investigated. The results showed remarkable decolorization efficiency which can be reached up to 100% in less than 12 min. Also, the decolorization efficiency increased with an increase in pH, ultrasonic density, ozone gas flow rate and decreasing initial dye concentration. The reaction was modeled using a pseudo-first-order kinetic model. The effect of different parameters on the rate constant was evaluated using the nonlinear regression method.

Keywords: Decolorization; Sonolysis; Ozonation; Kinetic study; Congo red

1. Introduction

Dyes are extensively used in many industries like cosmetics, carpet, food, pharmaceutical, etc. The textile industry is one of the major consumers of dyes [1,2]. Today, nearly 10,000 dyes are utilized in the textile industries of which about 60% are azo dyes. Azo dyes are sorted as mono-, di-, tri- and tetra-azo dyes.

The wastewater from textile mills are characterized not only by aesthetically objectionable colors but also by high concentrations of chemical and biochemical oxygen demand, total organic carbon and suspended solid [3].

In the past years, different processes by many researchers have been developed for the removal of pollutants and contaminants from various industrial wastewaters such as those in textile industries. These processes include adsorption, photodegradation, membrane filtration, biodegradation, electrocoagulation, oxidation and the ion-exchange process [4,5]. However, some of these processes such as adsorption and coagulation could not destroy or degrade dyes and only change the phase from wastewater to solid waste [6].

Advanced oxidation processes (AOPs) are one of the most effective methods for decolorization of dyes in wastewaters. These processes involve producing an oxidizing agent like hydroxyl radicals or chlorine, which react with organic contaminants in wastewater (like dyes) and decompose them without causing secondary wastes [5,7,8]. AOPs include ozonation, photocatalysis, Fenton, peroxide, O_3/UV, O_3/H_2O_2, UV/O_3/H_2O_2, UV/TiO_2 and UV/ZnO [9–14]. Furthermore, ozonation combined with ultrasonic irradiation (O_3/US) is another effective and novel method for dealing with wastewater [3,15].

Ozone is a powerful oxidant which destroys conjugated double bonds of dyes in aqueous solution such as C=C, C=N and N=N and other functional groups like the complex

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aromatic rings of dyes [8,16,17]. Ozone reacts with an organic compound through two possible degradation routes. (1) Direct reaction: ozone reacts directly with organic substrates in the solution by mass transfer of ozone from the gas phase under acidic condition [1]. (2) Indirect reaction: ozone decomposes and generates hydroxyl and other radical species in the solution under neutral conditions [6].

The rate-limiting parameter in the decolorization of azo dye solutions by O_3 is the poor mass transfer rate of ozone [18], that is, low solubility of ozone in the aqueous solutions [19]. Therefore, combining ozone with UV increases direct and indirect production of hydroxyl radicals because of the O_3 decomposition and H_2O_2 formation [20].

In the past years, much attention has been given to the application of ultrasound for wastewater treatment and destruction of aqueous pollutants including inorganic and organic pollutants [21]. Sonochemical oxidation utilizes ultrasound to generate the cavitation phenomenon, which results in a high temperature (5,000 K) and pressure (1,000 atm) [5,22]. In order to decompose pollutants species using sonication, two pathways occur when a solution is sonicated: (1) pyrolytic reactions inside or near the bubble and (2) reaction with hydroxyl radicals in the solution [21,23].

As the sonication is initiated, the hydroxyl radicals are produced which can react with the dye molecules and most of them are incorporated and produce H_2O_2 inside the cavitation bubble and at the gas–liquid interface. It was reported that hydrophilic and nonvolatile species mainly degrade via the hydroxyl radical in the bulk solution, while hydrophobic and volatile species degrade via pyrolytic reactions inside or near the bubble [24,25]. Since the decomposition rates of azo dyes through the hydroxyl radical are very low, in order to accelerate the decomposition rates of hydrophilic compounds, a combination of sonolysis with other oxidation processes such as ozonation have been developed [21].

The combination of ultrasonic irradiation with ozonation (US/O_3) is an AOP. This synergy is effective for the destruction and degradation of azo dyes from various wastewater, particularly textile effluents. When a liquid is sonolyzed, thermal decomposition of ozone occurs in the vapor phase of a cavitation bubble and hydroxyl radicals generated by this combined process. Also, the mass transfer coefficient is increased which results in increasing the ozone solubility and total rate of decomposition [3].

A simplified reaction scheme for hydroxyl radical production during a combination of US and O_3 treatment of water can be represented as follows [26]:

$$O_3^{\circ} + H_2O \rightarrow 2HOO^{\circ} \tag{1}$$

$$O_3 + HOO^{\circ} \rightarrow OH^{\circ} + 2O_2 \tag{2}$$

$$H_2O+))) \rightarrow OH^{\circ} + H^{\circ} \tag{3}$$

$$O_3+))) \rightarrow O_2(g) \rightarrow O(^3P)(g) \tag{4}$$

$$O(^3P)(g) + H_2O(g) \rightarrow 2OH^{\circ} \tag{5}$$

$$\text{Dye} + O_3 \rightarrow \text{Product} + H_2O_2 \tag{6}$$

$$\text{Dye} + OH^{\circ} \rightarrow \text{Product} \tag{7}$$

Therefore, the mechanism of the US/O_3 process for treatment of wastewater is (1) enhancement in the production of hydroxyl radicals by thermal decomposition of ozone represented in Eqs. (1)–(7) and (2) the mass transfer enhancement of ozone to the aqueous phase, resulting in larger gas diffusion coefficients [18].

In this paper, the decolorization of a model azo dye (Congo red) was investigated using an ultrasound-assisted ozonation process. A mathematical model based on the linear regression was developed to investigate the effect of intrinsic reaction kinetics and mass transfer resistance on the degradation rate.

2. Materials and methods

2.1. Chemicals

Congo red dye (CRD; disodium 4-amino-3-[4-[4-(1-amino-4-sulfunato-naphtalen-2-yl)diazenylphenyl]phenyl]diazenyl-naphtalene-1-sulfonate, $C_{32}H_{22}N_6Na_2O_6S_2$, molecular weight 696.665 g/mol, k_{max} 500 nm) was used as the model dye. This dye was provided from Sigma-Aldrich Company (USA), and used without further purification. The chemical structure of CRD is given in Fig. 1.

Analytical grade HCl (0.1 M) and NaOH (0.1 M) were obtained from Merck (Germany). The stock solution was prepared by dissolving 0.5 g of CRD in 1 L deionized water and HCl and NaOH solutions were used to adjust the initial pH (pH_0) of the dye solutions. Different concentrations of dye samples were prepared from the stock solution.

2.2. Instruments

A WTW-3110 pH meter (Germany) was used for monitoring the pH of the solution which was adjusted by adding HCl solution. Absorbance measurement was done by using the Hach UV–Vis spectrophotometer to monitor decolorization at the maximum absorption wavelength of 500 nm.

Pure oxygen is produced by the Oxy600 oxygen generator (Bitmos GmbH, Germany) and O_3 was generated by passing the pure and dry oxygen through the O_3 generator (LAB 802, Donaly, Iran). Generated ozone was bubbled into the solution through a porous ceramic plate at the bottom of the reactor. The iodimetric method was used for measuring the ozone concentration in air exerts. Sonication was carried out using an Up 400 ultrasonic generator equipped with a

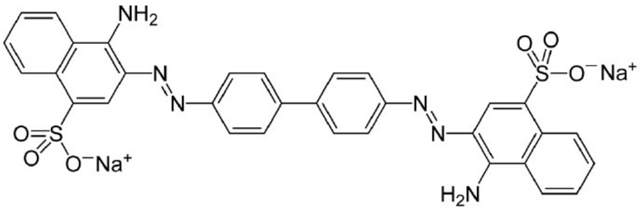

Fig. 1. Chemical structure of Congo red dye (CRD).

titanium probe transducer. The ultrasonic power dissipated into the reactor was calibrated by the calorimetric method to ensure the requested US conditions.

2.3. Experimental procedure

During sonolysis of dye solution with an ultrasonic processor, the generated ozone was bubbled into the solution. The flow rate of ozone was determined via a rotameter connected to the ozone generator. All the experiments were carried out in a thermostat bath fixing the solution temperature at 25°C. The effect of different parameters such as pH, initial dye concentration, ozone dose and ultrasonic density on the degradation of CRD was investigated. A schematic diagram of the process can be seen in Fig. 2.

All the experiments continued for 12 min and the dye concentration was measured at 2 min intervals. The experimental conditions that were performed in this work are shown in Table 1.

The decolorization conversion was calculated using the following equation: [27]

$$\mathrm{DE\%} = \frac{A_0 - A}{A_0} \times 100 \tag{8}$$

in which A_0 and A are the dye concentration at the initial and certain process time, respectively.

3. Results and discussions

3.1. Effect of dye concentration

The decolorization efficiency depends on the initial dye concentration. Fig. 3 shows the decolorization of CRD for the combined US/O_3 system at different initial dye concentrations. The initial concentration of dye was 5, 10, 20, 30 and 50 mg/L and the temperature in all the experiments was 25°C. As can be seen in Fig. 3, the decolorization efficiency was significantly affected by the initial concentration of CRD in the aqueous solutions. The graph shows that the dye removal efficiency decreased with increasing the initial dye concentration. For instance, increasing the dye concentration from 5 to 10 ppm results in decreasing the decolorization efficiency from 99.99% to 48.72% at 12 min. The results are consistent with data obtained by He et al. [3], which indicated that the dye decolorization rate strongly depends on initial dye concentration. However, these results differ from the findings obtained by Lall et al. [28], which indicated the slight effect of initial dye concentration. A possible explanation for this might be that increasing the initial dye concentration generates more intermediates and inorganic anions through the dye oxidation. Therefore, the dye metabolites should compete with carbonaceous organics for reaction with hydroxyl radicals due to the nonselective nature of the radical's reactivity.

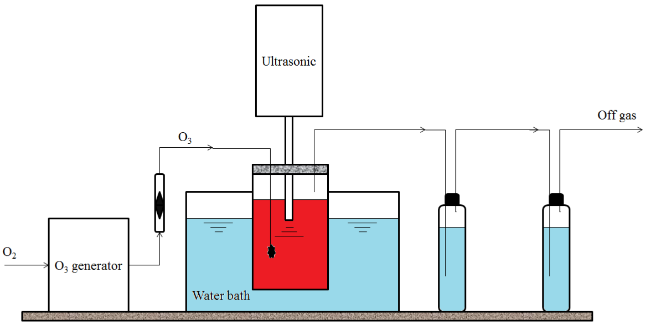

Fig. 2. Schematic diagram of the sono-assisted ozonation process.

Table 1
Experimental condition of US/O_3 system

Parameter	Range
Initial dye concentration (mg/L)	5–50
pH value	3–11
Ozone gas flow rate (L/min)	0.5–2.5
US power density (W/L)	100–400

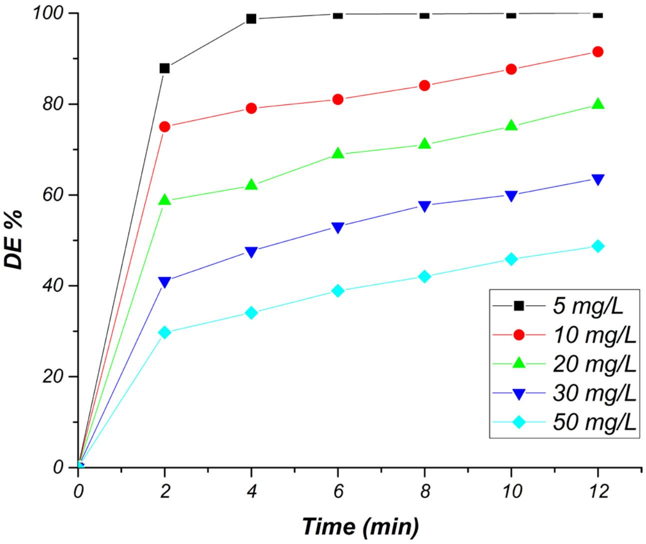

Fig. 3. Effect of initial dye concentration on the decolorization efficiency (pH = 11, ozone flow rate = 2.5 L/min, US power density = 400 W/L).

3.2. Effect of pH

The pH value of the dye solution is an important parameter affecting the overall rate of degradation reaction. In order to investigate the relation between the initial pH of the solution and dye removal efficiency in the combined US/O_3 system, the experiments were carried out at pH values of 3, 5, 7, 9 and 11. Fig. 4 shows the effect of pH on the dye removal efficiency. It is obvious that increasing pH has a positive effect on the rate of decolorization. In fact, the effect of pH on the decolorization rate of different dyes by ozone is not similar. Different researchers reported that an increase in pH enhanced the decolorization rate for Reactive Blue 19 [3] and did not have any effect for the dyes like methylene orange, Acid Orange 7 and Reactive Blue 19 [21,28,29]. Zhou et al. [2] reported that the decolorization rate decreases with increasing pH.

This may be explained by the fact that as the pH of the solution increases, more hydroxyl radicals are expected to

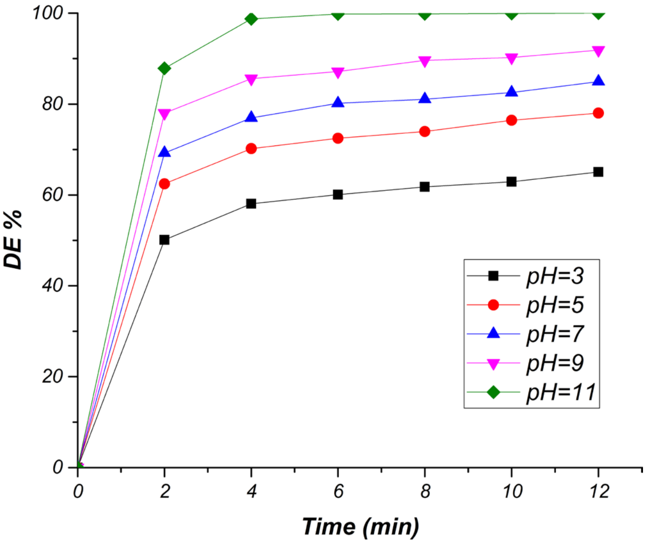

Fig. 4. Effect of pH value on the decolorization efficiency (initial dye concentration = 5 mg/L, ozone gas flow rate = 2.5 L/min, US power density = 400 W/L).

generate and because of the high oxidation potential of the hydroxyl radical, the rate of dye removal is enhanced. At the acidic pH, direct oxidation by ozone is the main mechanism of decolorization and sonication enhances this process via increasing the mass transfer of ozone from the gas to liquid phase. Under the alkali condition, it seems that the decolorization rate is controlled by the $OH^•$ production rate. Ozone decomposes to free radicals and increases the overall reaction rate.

Also, the CRD is in the ionic form in a pH between 4 and 9, which is less degradable in the aqueous solution than the molecular form of the dye. Another possible reason causes enhanced decolorization at a higher pH. So, a higher pH value is favorable for the reaction and the pH of 11 is selected for all the experiments.

3.3. Effect of ozone gas flow rate

Fig. 5 shows the dye removal efficiency of CRD for the combined US/O_3 system at different ozone gas flow rates. The dye removal efficiency is directly affected by the ozone flow rate. It seems that the mass transfer coefficient of ozone from gas phase to aqueous solution was enhanced by increasing the gas flow rate [30]. The use of ultrasonic power also increases the mass transfer of ozone in the solution. It seems that the decolorization process is controlled by mass transfer of ozone in aqueous solution. An increase in the ozone inlet flow rate increases the rate of mass transfer of ozone in the solution, which increases the apparent reaction rate.

3.4. Effect of US power density

Fig. 6 shows the effect of sonication power on the decolorization efficiency. Decolorization efficiencies that were obtained in 12 min in our experiments were 99.99%, 82.96%, 65.07% and 56.8% when power densities were 400, 300, 200 and 100 W/L, respectively. There are three factors that influence the efficiency of US: power density, frequency and amplitude of the system [31]. In our experiments, the ultrasonicator was kept at a constant frequency (20 kHz). By increasing the power density of US, the energy that is supplied to cause cavitation bubbles is increased and therefore the dye removal efficiency should enhance with a power density of US. However, this finding is contrary to that of Zhou et al. [2] who investigated the degradation of triphenylmethane dye by the ultrasonic-assisted ozone oxidation process.

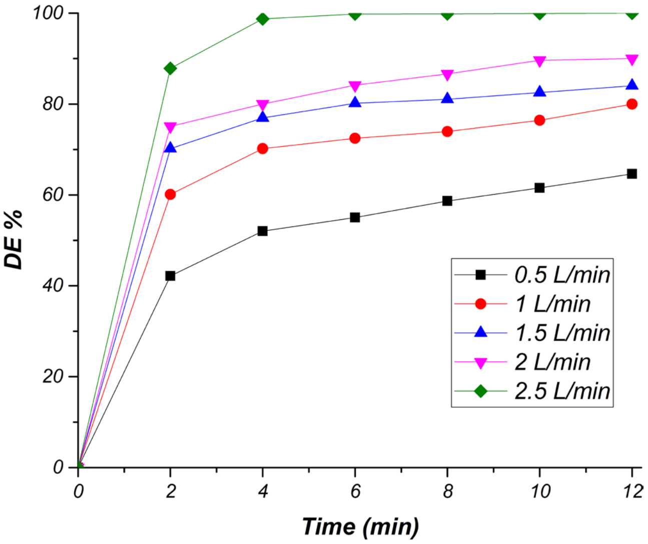

Fig. 5. Effect of ozone flow rate on the decolorization efficiency (initial dye concentration = 5 mg/L, US power density = 400 W/L, pH = 11).

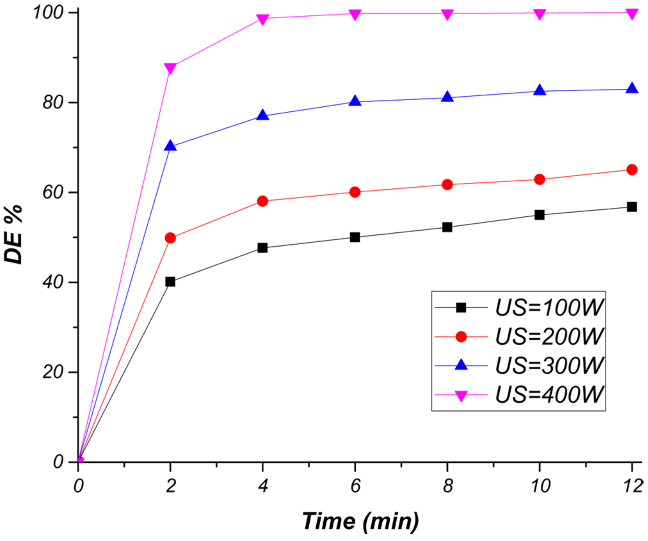

Fig. 6. Effect of ultrasonic density on the decolorization efficiency (initial dye concentration = 5 mg/L, ozone gas flow rate = 2.5 L/min, pH = 11).

Zhang et al. [29] reported that ultrasonic has both chemical and physical effects on the oxidation processes. High temperature and pressure inside the bubbles cause the formation of •H and •OH radicals. However, the yield of radical formation is not much at low ultrasonic frequency.

Previous works have indicated that the thermal decomposition of ozone in collapsing cavitation bubbles appear to be the main mechanism for the enhanced destruction of chemical contaminants at higher pH [3]. Ozone mass transfer from gas bubbles to the liquid phase is considered as a rate-limiting step for the decolorization process. The ultrasonic vibrations increase the gas phase interfacial velocity and decrease the gas bubble size.

3.5. Modeling of decolorization kinetics

The mechanism of dye decomposition by sono-assisted ozonation was explained earlier (Eqs. (1)–(7)). Since, the concentration of O_3 and OH radicals in the solution were controlled by other phenomena other than degradation reaction, the rate of decolorization reaction can be considered first order only with respect to the dye concentration:

$$-\frac{d[\mathrm{CRD}]}{dt} = K[\mathrm{CRD}] \tag{9}$$

where K is the rate constant for the combined US/O_3 system.

The rate constant depends on operational parameters that were introduced above.

$$K = m(\text{operational parameter})^n \tag{10}$$

In the above equation, the relation between K and operational parameters (m, n) were calculated by nonlinear regression analysis of experimental data via Polymath 5.1 software. The obtained results are displayed in Figs. 7–10 and Table 2.

As can be seen from Figs. 8 to 10, the K (rate constant) enhanced by increasing ozone flow rate, US density and pH value. Fig. 7 shows that increasing the dye concentration from 5 to 50 mg/L decreased the rate constant from 0.3 to 0.056 min^{-1}

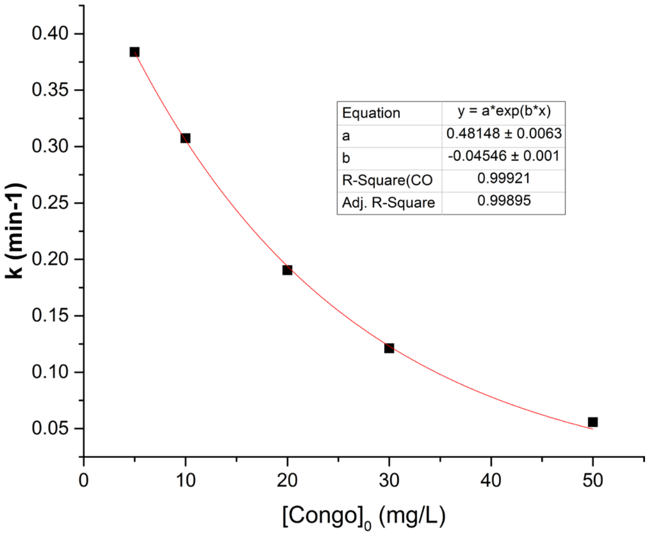

Fig. 7. Effect of initial dye concentration on the decolorization rate (K) (ozone gas flow rate = 2.5 L/min, US power density = 400 W/L, pH = 11).

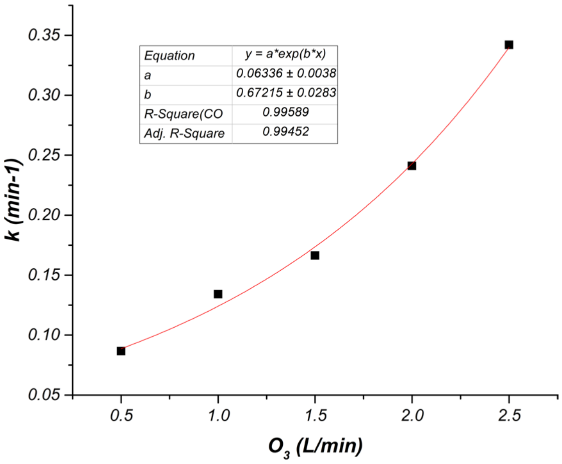

Fig. 8. Effect of ozone gas flow rate on the decolorization rate (K) (initial dye concentration = 5 mg/L, US power density = 400 W/L, pH = 11).

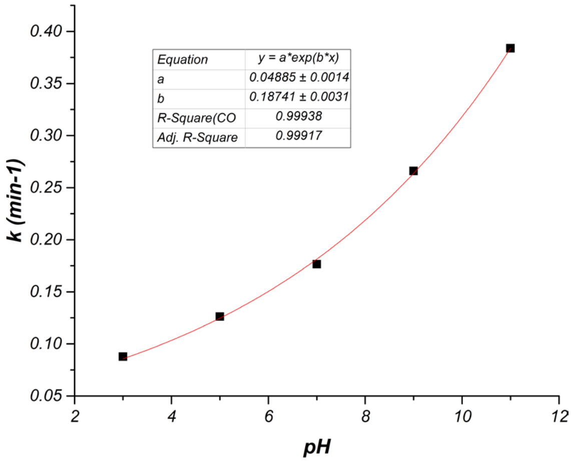

Fig. 9. Effect of US power density on the decolorization rate (K) (initial dye concentration = 5 mg/L, ozone gas flow rate = 2.5 L/min, pH = 11).

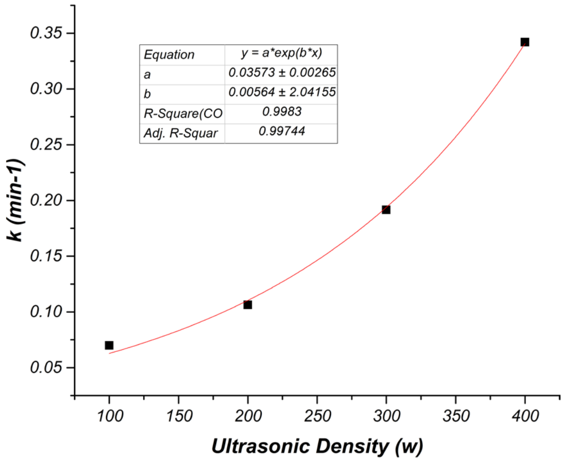

Fig. 10. Effect of pH value on the decolorization rate (K) (initial dye concentration = 5 mg/L, ozone gas flow rate = 2.5 L/min, US power density = 400 W/L).

Table 2
Kinetic model constants obtained from nonlinear regression analysis

Parameter	m	n	R^2
Initial dye concentration (mg/L)	0.4645	–0.043	0.9997
pH value	0.0498	0.185	0.9991
Ozone gas flow rate (L/min)	0.064	0.6669	0.9925
US power density (W/L)	0.039	0.0054	0.9944

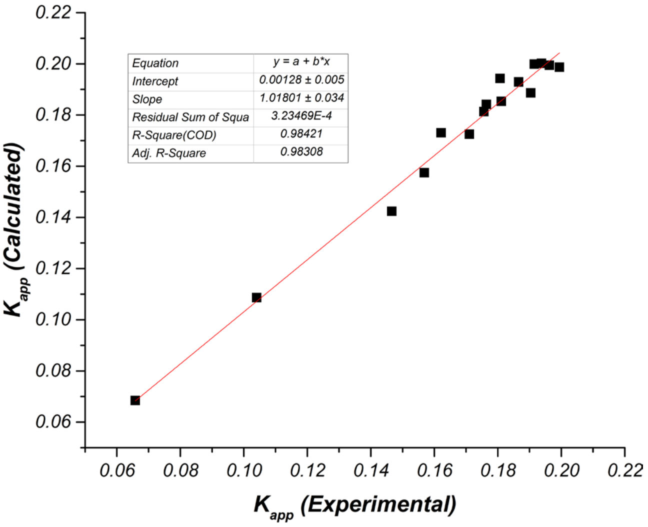

Fig. 11. Comparison between experimental and calculated pseudo-first-order rate constants (min^{-1}) for decolorization of CRD in the US/O$_3$ process at different operational conditions (for experimental details refer to Table 1).

which is an 81.3% decrease in the rate constant. The effect of ozone flow rate on the rate constants can be seen in Fig. 8. Increasing the ozone flow rate from 0.5 to 3 L/min causes a four time increase of the rate constant. An increase in ozone concentration in the presence of the ultrasonic field leads to an increase in ozone generated free radicals at the boundary between cavitation-induced bubbles and the bulk liquid.

Fig. 9 shows that an increase in ultrasonic density increases the formation of free radicals and results in cavitation in the liquid and rate of reaction. It also increases turbulences which reduce the mass transfer film thickness and also the overall rate of reaction. Increasing US density from 100 to 400 W leads to an enhancement of the rate constant from 0.069 to 0.34 min^{-1}.

Increasing the solution pH seems to have a direct effect on the rate constant (Fig. 10). These results may be due to the fact that increasing pH increases the rate $^{\bullet}$OH radicals destruction of the dye due to the enhanced trapping effect. It can be seen that increasing pH from 3 to 11 increases the rate constant from 0.087 to 0.38 min^{-1}.

3.6. Development of the kinetic model

Figs. 7–10 show the effect of operational parameters such as initial dye concentration, ozone concentration, power density and pH on the decolorization rate. A nonlinear regression analysis was used in order to evaluate the relation between the overall rate constant and the operational parameters as follows:

$$K = m[\mathrm{CRD}]_0^a[\mathrm{O}_3]^b[\mathrm{US}]^c[\mathrm{pH}]^d \tag{11}$$

The value of m in Eq. (11) can be calculated using these parameters and the experimental data (average value: 0.07054 min^{-1}).

$$K = 0.07054[\mathrm{CRD}]_0^{-0.043}[\mathrm{O}_3]^{0.67}[\mathrm{US}]^{0.0054}[\mathrm{pH}]^{0.185} \tag{12}$$

Fig. 11 shows the rate constant from the experimental data vs. calculated rate constant which was obtained from the mathematical model. As can be seen in this figure, the calculated results are in good agreement with the experimental data.

4. Conclusion

In this work, the decolorization of CRD as a model of azo dye was investigated using a combination of the ozonation and sonication system (O$_3$/US) in the aqueous solutions. In order to study the effect of various operational parameters on the decolorization process and dye removal efficiency, different experiments were carried out under different operational parameters. The experimental results indicate that dye removal efficiency increased with enhancing initial pH value, ozone flow rate and US power density and declined with increasing initial dye concentration. The decolorization reaction was successfully modeled using a pseudo-first-order kinetic model. Nonlinear regression analysis was used to evaluate the relation between the rate constant and operational parameters.

A comparison between the theoretically calculated data for the rate constant (K) and experimental data shows that these values are in good agreement.

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