Effect of Inorganic Ions on the Discoloration of Rhodamine B by the Fenton Process

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Research Article

Keywords: Optimisation, Oxydation, Rhodamine B, Fenton Process, discoloration rate, inorganic ions.

Posted Date: January 17th, 2022

DOI: https://doi.org/10.21203/rs.3.rs-982974/v1

Abstract

This study showed that the oxidation of rhodamine B by the Fenton process is a very fast method because the reaction takes place within the first 20 minutes. The mixture of Fe^{2+} and H_2O_2 produces hydroxide radicals responsible for the degradation of rhodamine B. The study of pH influence on the rhodamine B oxidation reveals that for maximum oxidation of rhodamine B, the pH must be less than or equal to 2. For pH > 2, there is a decrease in rhodamine B oxidation. This is due to side reactions that occur if the concentration of Fe^{2+} is high. This reduces the amount of oxidized rhodamine B. We note that the oxidation of rhodamine B is faster for low concentrations than for high concentrations of rhodamine B. According to our results, for a maximum oxidation of 5 mg / L of rhodamine, it takes $8.4.10^{-4}$ mg / L of Fe^{2+}, 3.10^{-3} M of H_2O_2 and pH = 2. This work also showed that the presence of inorganic ions strongly slows down the rate of degradation of rhodamine in the following order: $Cl^{-} \boxtimes NO_3^{-} \boxtimes SO_4^{2-} \boxtimes PO_4^{3-}$.

1. Introduction

Water is the central element of all economic and social processes, and its human need grows in line with the increase in agro-industrial activities and the degree of development (Wyman 2013). The water used in the various industrial sectors contains more and more pollutants (Sadia et al. 2016). Dyes are among the most dangerous pollutants. They are used in many industrial sectors such as food, cosmetic and clinical products, in particular in the textile industries (Mansour et al. 2011; Lellis et al. 2019). The molecules of these dyes are hardly biodegradable and difficult to degrade by conventional methods (Deepika et al. 2017; Radia et al. 2019). Thus, for the industrial world, the treatment of dyes has become a priority. The development of effective methods of treating these dyes has been and continues to be the subject of much research. Currently, the most recent advances in wastewater treatment have been made in advanced oxidation processes (AOP) which appear to be more efficient techniques for the destruction of organic pollutants (Fu et al. 2021 ; Sydorchuk et al. 2021 ; Ranjitha et al. 2021).

The Fenton process is one of the advanced oxidation techniques, based on the formation of hydroxyl radicals ($HO^{\bullet}$) possessing an oxidizing power superior to that of traditional oxidants such as Cl_2, ClO_2 or O_3. These radicals are capable to partially or totally mineralizing most organic compounds (Jiahai et al. 2006; Sheng et al. 2017; Zhihui et al. 2021). In the literature, the process has been used to degrade rhodamine B (RhB). However, this work did not completely degrade RhB (Wei et al. 2016; Mei-Fang et al. 2011). Moreover, in the literature very few authors have studied the influence of inorganic ions on RhB oxidation. Thus our objective is to optimize the RhB oxidation by the Fenton process and to study the influence inorganic ions on RhB oxidation.

2. Materials And Methods

2.1. Rhodamine B concentration determination

The spectrophotometric method of RhB qantification is a method which makes it possible to follow only the RhB concentration and all the compounds absorbing at the same frequency as RhB. In this study, absorbance was measured using a HACH DR-6000 UV-visible spectrophotometer.

RhB used is a cationic dye. It is also used as a tracer dye in water to determine volumes, rates and directions of flow and transport. All the information on RhB is given by Table 1 and Fig. 1.

Table 1
Rhodamine B characteristics

Parameters	Values
Suggested name	Rhodamine B
Aspect	Solid green
Color	Red
Odeur	Odorless
C.I. number	45170
C.I. name	Basic violet 10
Class	Rhodamine
λmax	554 nm
Molecular formula	$C_{28}H_{31}N_2O_3Cl$
Formula weight	479.02

2.2. Reagents

Hydrogen peroxide (H_2O_2) were manufactured SCHARLAU. Ferrous sulfate ($FeSO_4$), sodium chloride (NaCl), sodium hydroxide (NaOH), sodium sulfate (Na_2SO_4), sodium nitrate ($NaNO_3$) and sodium phosphates (Na_3PO_4, 12 H_2O) have been manufactured by the company Merck. Sulfuric acid (H_2SO_4) was supplied by the company Fluka.

2.3. Operating mode

A stock solution of RhB (100 mg / L) was prepared in distilled water. Several sets of 500 mL reaction mixture are prepared with the RhB solutions, hydrogen peroxide and ferrous ions. The pH of the reaction mixture is adjusted with sodium hydroxide solution and sulfuric acid solution. One of the following parameters is varied: RhB concentration, Fe^{2+} concentration, H_2O_2 concentration and pH, setting the other parameters. Regarding the influence of inorganic ions, a given concentration is added to the reaction mixture. Then 2 mL of the reaction media is taken every 5 minutes and the absorbance is measured using a HACH DR-6000 UV-visible spectrophotometer. Before each spectrophotometer reading, the collected solution is filtered through filter paper. The exact concentration of RhB remaining in the reaction mixture is determined using a calibration curve. The absorbances of several RhB solutions of known concentration were measured. The values obtained were used to plot the absorbance curve as a function of the RhB concentration. The calibration curve obtained is an equation line:

Absorbance = 0,2765 C (1)

All the experiments were made at ambient temperature of 25°C

3. Results And Discussion

3.1. Influence of H_2O_2 concentration

Figure 2 shows the spectra measured at regular time intervals (5 min) during the RhB oxidation (5 mg / L) in the presence of Fe^{2+} (10^{-3} M), H_2O_2 (10^{-2} M) at pH = 3. This figure shows that the maximum absorbance is obtained at the wavelength of 554 nm. We also note the absorbance decreases during the reaction showing that there is degradation of rhodamine B.

Figure 3 shows the influence of H_2O_2 concentration on RhB discoloration. The concentrations of RhB and Fe^{2+} were respectively set at 5 mg / L and 10^{-3} mol / L. The pH was set at 3 by varying the concentration of hydrogen peroxide. In this figure, we see a rapid change in the degradation rate from 0 to 10 min for all the H_2O_2 concentrations used. Then there is a slow increase in the degradation rate from 10 to 30 min. Beyond 30 min, the degradation rate remains constant. The results of Fig. 3b confirm this result. Thus for the rest of our work, the time used for RhB degradation is 30 min.

Figures 3a and 3b also show that the degradation rate increases when the initial H_2O_2 concentration increases from 0.01 M to 0.03 M. With 0.03 M in H_2O_2 the degradation rate reaches the maximum value (95.94 %). Beyond 0.03 M, the degradation rate of RhB decreases. It goes to 94.74 % for 0.04 M and to 90.60 % for 0.05 M. This shows that 0.03 M constitutes, under the study conditions, the optimal concentration of H_2O_2 necessary for the RhB degradation.

The results obtained can be explained by the fact that increasing the H_2O_2 concentration causes an increase in the amount of $OH^{\bullet}$ and therefore increases the RhB degradation efficiency. However, too high a concentration of H_2O_2 causes trapping of hydroxyl radicals (Eq. 2) due to excess H_2O_2 forming hydroperoxyl radicals ($HO_2^{\bullet}$) and slows down the degradation of the dye (Nidheesh et al. 2013; Behnajady et al. 2008).

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

3.2. Kinetic model of the RhB oxidation

The study of the kinetic model applicable to RhB oxidation by the Fenton process was carried out. The results obtained by applying the 0, 1 and 2 pseudo-order models are shown in Figs. 4a, 4b and 4b.

Table 2
Parameters of the kinetics models

H_2O_2 Concentration	**Pseudo-order 0 kinetic model**		**Pseudo-order 1 kinetic model**		**Pseudo-order 2 kinetic model**	
	R^2	**k_{app} (mn^{-1})**	**R^2**	**k_{app} (mn^{-1})**	**R^2**	**k_{app} ($mol^{-1}.L.min^{-1}$)**
0.1 M	0.862	-0.264	0.959	0.120	0.989	0.075
0.2 M	0.766	-0.277	0.941	0.158	0.972	0.167
0.3 M	0.771	-0.292	0.933	0.206	0.940	0.384
0.4 M	0.668	-0.282	0.857	0.193	0.996	0.354
0.5 M	0.711	-0.268	0.864	0.154	0.970	0.160

Consider the constant production kinetic of hydroxyl radicals and their stable concentration during the process, we have:
For the equation:

$RhB + OH^{\bullet} \rightarrow RhBOH^{\bullet}$ (3)

The kinetic equation is given by;

$$\frac{-d[RB]}{dt} = k\left[OH^{\bullet}\right][RhB]$$

4

Since the concentration of hydroxyl radicals is constant, the oxidation kinetic of the organic compound can be described by an apparent kinetic law of order 1 with respect to the concentration of organic compound (Eq. 5):

Which give:

$$\frac{-d[RB]}{dt} = k_{app}[RhB]$$

5

$$ln\left(\frac{RB}{RB_0}\right) = -k_{app}t$$

6

Where k_{app} is the pseudoconstant of apparent kinetic of order 1, which is in agreement with recent work concerning the reaction of the hydroxyl radical with organic compounds (Chakinala et al. 2008).

Order 2 reaction

$$\frac{-d[RhB]}{dt} = k_{app}[RhB]^2$$

7

$$\frac{1}{[RhB]} = \frac{1}{[RhB]_0} + k_{app}t$$

8

Order 0 reaction

$$\frac{-d[RhB]}{dt} = k_{app}[RhB]^0$$

9

$[RhB]_t = [RhB]_0 - k\ t$ (10)

The curves in Fig. 4 were used to determine the parameters of the 0, 1 and 2 order kinetics models. The values obtained are recorded in Table II. This table shows that the values of the determination coefficients obtained from the order 2 kinetic model are greater than those of the kinetic model of order 0 and 1 regardless of the H_2O_2 concentration. This shows that the kinetic model suitable for our study is the second order kinetic model. Table 2 also shows that the maximum value of the apparent velocity pseudoconstant is maximum when the H_2O_2 concentration is equal to 0.03 M. This confirms that the reaction kinetics are maximum when the H_2O_2 concentration is equal to 0.03 M.

3.3. Influence of Fe^{2+} concentration

The influence of Fe^{2+} concentration on RhB oxidation has been studied. Figure 5 show the results obtained.

Figure 5 show that the RhB degradation rate varies with the Fe^{2+} concentration. These figures show that when going from 5.10^{-4} M to $8.4.10^{-4}$ M the degradation rate increases from 94.43–96.02%. These results are in agreement with the results of the literature which states that the addition of Fe^{2+} improves the RhB destruction rate (Torres et al. 2007; Minero et al. 2005; Dai et al. 2006). Then it is observed that beyond $8.4.10^{-4}$ M, it decreases to reach 85.42% at $1.25.10^{-3}$ M. This shows that the optimum degradation rate of RhB is reached for a Fe^{2+} concentration equal $8.4.10^{-4}$ M. Thus, for optimum degradation of RhB, the $[H_2O_2]$ / $[Fe^{2+}]$ ratio equal to 35.7.

Above $8.4.10^{-4}$ mol / L, Fe^{2+} is engaged in a secondary reaction by consuming hydroxyl radicals, hence the decrease in the RhB degradation. According to Panizza et al. (Panizza et al. 2001) when the Fe^{2+} concentration is very high, side reactions occur (Eqs 11 to 13). These reactions compete with the degradation reaction of organic compounds. This reduces the organic compounds oxidation.

$HO^{\bullet} + Fe^{2+} \rightarrow Fe^{3+} + OH^{-}$ (11)

$Fe^{2+} + H_2O_2 + H^{+} \rightarrow Fe^{3+} + H_2O + HO^{\bullet}$ (12)

$2Fe^{3+} + H_2O_2 \rightarrow 2\ Fe^{2+} + O_2 + 2H^{+}$ (13)

According to Eq. 12, Fe^{2+} react with H_2O_2 to form Fe^{3+}. So if the Fe^{2+} concentration is high, the amount of Fe^{3+} produced will be high. Fe^{3+} reacts with H_2O_2 according to Eq. 13. The sharp decrease in degradation with an excess of Fe^{2+} would also be linked to the decomposition of H_2O_2 by the Fe^{2+} produced.

3.4. Influence of RhB concentration

The amount of material to be degraded is one of the factors that determines the efficiency of the treatment process. Thus, various initial RhB concentrations were studied by fixing the concentration values $[H_2O_2] = 0.03$ M and $[Fe^{2+}] = 8.4.10^{-4}$ M. The results obtained are presented in Fig. 6.

From Fig. 6, the degradation efficiency decreases as the initial RhB concentration increases. This is in agreement with the results of the literature (Wang et al. 2008; Vajnhandl et al. 2007). Also, an almost total degradation (98.52%) is observed after 5 min for the low concentrations of RhB (1.5 mg / L). At high concentrations (10 mg / L), degradation is relatively slow with a rate of 94.27% after 30 min.

The gradual decrease in degradation rates with initial concentration could be explained by competition reactions between the dye molecules and those of the intermediates formed during the Fenton oxidation process (Hu et al. 2008). The dye molecules as well as the intermediate products formed will compete to react with the $HO^{\bullet}$ radicals. The amount of degradation intermediates formed is proportional to the initial concentration of the dye, so the decrease in the RhB degradation efficiency is a direct consequence of the increase in this competitive effect with the initial concentration of RhB (Hu et al. 2008).

3.5. Influence of pH

The media pH is one of the most important parameters influencing the degradation of organic pollutants by advanced oxidation processes. In this work, the RhB degradation was carried out at different pH values (1.5; 2; 3; 4). The results obtained are shown in Fig. 7.

The results of these figures indicate that the degradation rates are higher in a very acidic media (pH between 1.5 and 2) than for pH between 3 and 4. RhB degradation rate is constant at equal pH. 1.5 and 2. A degradation rate of 99.42% was obtained for a pH equal to 1.5 or 2. These latter values therefore constitute the optimum conditions for RhB degradation. Several studies report that pH dramatically influences the transformation of synthetic dyes in aqueous solution, and generally the optimum value is in the range 1 to 3 (Nidheesh et al. 2013; Gad et al. 2009). In the water treatment domain, the pH brings about a modification in the ionization degree of the organic molecule which appears in different forms depending on its ionizable functions. RhB carries a carboxylic acid function having a pKa = 3. If the media is characterized by a pH below this value, the cationic form of RhB (RhB^+) is predominant. Its zwitterionic form is predominant for pH greater than 3.7 (Minero et al. 2008).

The acceleration of degradation kinetics in an acidic media (pH between 1 and 3) is therefore probably due to the protonation of the functional site –COOH, which improves the hydrophobicity of the RhB molecules. These results show that the efficiency of the capture of $HO^•$ radicals by RhB molecules is greater in acidic conditions, due to the strong regeneration of H_2O_2 molecules (Nidheesh et al. 2013).

3.6. Influence of inorganic ions

The influence of the Cl^- concentration on RhB oxidation was studied at pH 2 in the presence of 5 mg / L RhB, 3.10^{-2} M H_2O_2 and $8.4.10^{-4}$ M Fe^{2+}. Fig. 8a shows the results obtained. This figure shows that the addition of chloride reduces the RhB discoloration rate. The reduction in the rate of discoloration increases as the chloride concentration increases. Fig. 8b shows that the RhB discoloration rates are respectively 99.42%, 97.72%, 96.96%, 92.42%, 81.53% and 76.54% for a chloride concentration of 0, $6.49.10^{-4}$, $1.19.10^{-3}$M, $2.91.10^{-3}$M, $7.03.10^{-3}$ M and 10^{-2} M. This shows that the chloride inhibits the degradation reaction of RhB. This inhibition may be due to complexation and radical scavenging (Lu et al. 2005). As shown in equations (14) and (15), chloride react with hydroxyl radicals competing with the RhB oxidation reaction, leading to inhibition of oxidation, and therefore slowing down the oxidation rate (Liao et al. 2001).

$Cl^- + OH^• \leftrightarrow HOCl^{-•} + H_2O$ (14)

$HOCl^{-•} + H^+ \rightarrow Cl^• + H_2O$ (15)

Cl^- can undergo complexing reactions with ferrous and ferric ions, which prevents the reaction causing the formation of hydroxyl radicals product. Complexation reactions are shown in Eqs (16) - (20).

$Fe^{2+} + Cl^- \rightarrow FeCl^+$ (16)

$FeCl^+ + Cl^- \rightarrow FeCl_2$ (17)

$Fe^{3+} + Cl^- \rightarrow FeCl_2$ (18)

$FeCl^{2+} + Cl^- \rightarrow FeCl_2^+$ (19)

$FeCl^{2+} + Cl^- \rightarrow FeCl_3$ (20)

Based on the above reactions, the Fenton reaction was inhibited because ferrous and ferric complexes cannot catalyze hydrogen peroxide to produce hydroxyl radicals as efficiently as when they are free. At the initial stage of the Fenton reaction, the species of iron is the ferrous ion. The reactivity of ferrous ion complexes with chloride are much lower than those of Fe^{2+} (Lu et al. 2005). This results in a reduction in the RhB discoloration rate. The values in Table 1 show that the chloride inhibit the RhB discoloration. The RhB discoloration rate decreases with increasing chloride concentration. This table also confirms that the kinetic model suitable for the oxidation of RhB is the pseudo-order 2 kinetic model.

Table 3
Parameters of the kinetics models

Cl $^{-}$Concentration	**Pseudo-order 1 kinetic model**		**Pseudo-order 1 kinetic model**		**Pseudo-order 2 kinetic model**	
	R^2	**k_{app} (min^{-1})**	**R^2**	**k_{app} (min^{-1})**	**R^2**	**k_{app} ($mol^{-1}.L.min^{-1}$)**
0 M	0.636	- 0.290	0.922	0.206	0.981	1.345
$6.49.10^{-4}$ M	0.623	- 0.287	0.862	0.184	0.944	0.967
$1.19.10^{-3}$ M	0.881	- 0.272	0.945	0.135	0.993	0.343
$2.91.10^{-3}$ M	0.818	- 0.233	0.964	0.105	0.990	0.064
$7.03.10^{-3}$ M	0.819	- 0.197	0.926	0.067	0.984	0.027

The influence of inorganic ions on RhB degradation rate has been investigated. The results obtained are shown in Fig. 9. This figure shows a decrease in the RhB degradation rate in the presence of inorganic ions. It is also noted that for a given ion, the higher its concentration, the more the RhB degradation rate decreases. This figure indicates that the presence of Cl^- has a less negative influence on RhB degradation, compared to nitrate, sulphate and phosphate ions. According to the results obtained, inorganic ions strongly slow down the RhB degradation rate in the following order: Cl^- NO_3^- SO_4^{2-} PO_4^{3-}.

In fact, the Fenton process is very sensitive to inorganic ions present in solution (Lu et al. 1997). These ions can form complexes with Fe (II) (Eqs 21 to 24) thus affecting the distribution and reactivity of free Fe^{2+}.

$Fe^{2+} + Cl^{-} \rightarrow FeCl^{+}$ (21)

$Fe^{2+} + SO_4^{2-} \rightarrow FeSO_4$ (22)

$Fe^{2+} + H_2PO_4^{-} \rightarrow FeH_2PO_4^{+}$ (23)

$Fe^{2+} + NO_3^{-} \rightarrow FeNO_3^{+}$ (24)

The presence of Cl^-, NO_3^-, SO_4^{2-}, PO_4^{3-} leads to competition between organic matter and hydroxyl radicals, which delays the RhB oxidation. Inorganic ions react with hydroxyl radicals to produce inorganic radicals with less powerful oxidizing powers compared to hydroxyl radicals.

The influence of the synergistic effect of the above anions was also examined. The results obtained are illustrated in Fig. 10. Analysis of this figure shows that the presence of several ions (NO_3^-; Cl^-; SO_4^{2-}; PO_4^{3-}) in the reaction media greatly reduces the RhB degradation rate. This reduction in the abatement rate is more accentuated as the ion concentration increases. The results presented in Figs. 8, 9 and 10 clearly indicate that the presence of these ions negatively impacts the degradation of RhB.

Conclusion

This study showed that the mixture of H_2O_2 and Fe^{2+} significantly degrades RhB. This mixture produces hydroxide radicals responsible for the RhB degradation. However, their excess in the media generates ionic competition phenomena which reduce the dye degradation. The study of pH influence on RhB oxidation reveals that for a maximal oxidation of RhB, the pH must be less than or equal to 2. For a pH greater than 2, there is a decrease in RhB oxidation. This is due to side reactions. There are also side reactions that occur if the concentration of Fe^{2+} is high (from 0.001 M). This reduces the amount of oxidized RhB (from 96% to 85.42%). According to the results obtained, for a maximum oxidation of 5 mg / L of RhB, it takes $8.4.10^{-4}$ M Fe^{2+}, 3.10^{-3} M H_2O_2 and a pH equal to 2. It is noted that the RhB oxidation is faster for low concentrations than for high RhB concentrations. This study also showed that the presence of inorganic ions such as NO_3^-, Cl^-, SO_4^{2-} and PO_4^{3-} in the reaction media reduces the RhB degradation rate. During this work, the samples were analyzed by the UV-visible spectrophotometric method which proved to be a simple and fast method.

Declarations

Ethics approval and consent to participate

Not applicable

Consent for publication

Not applicable

Availability of data and materials

Not applicable

Competing interests

The authors declare no competing interests

Funding

Research, materials, writing and publication fees were funded by the authors.

Authors' contributions

Author contribution KO (corresponding author) and AA contributed to the study conception and design. Authors AA and OL supervised the work and instructed all the research. Analyses were performed by KAR, KAE, KKS and KKE. Material preparation and data curation were performed by KO, KKJ and KS and OL. The first draft of the manuscript was written by KO, KAR and KAE and all authors commented on previous versions of the manuscript. All authors read and approved the fnal manuscript.

Acknowledgements

The authors would like to thank the Laboratory of Thermodynamics and Physical Chemistry of the Environment of the University of Nangui Abrogoua for providing access to the facilities.

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Figures

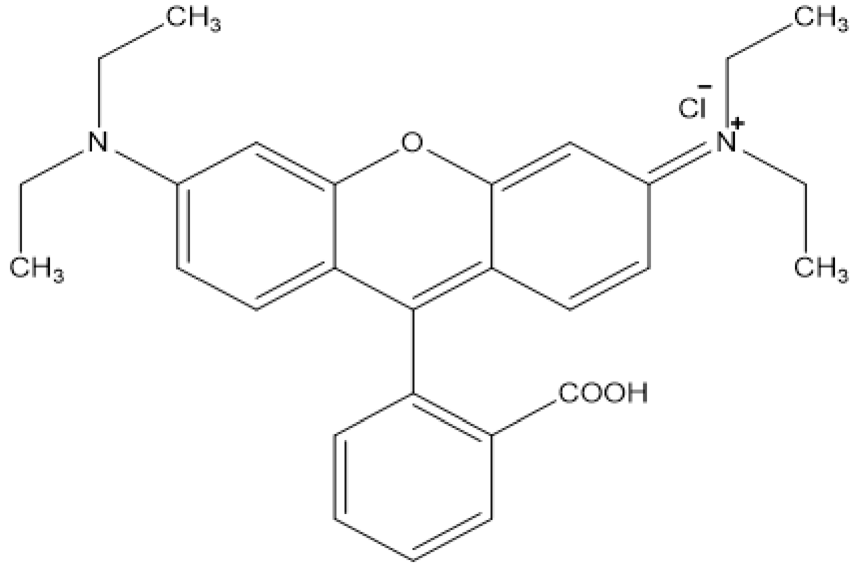

Figure 1

Molecular structure of rhodamine B

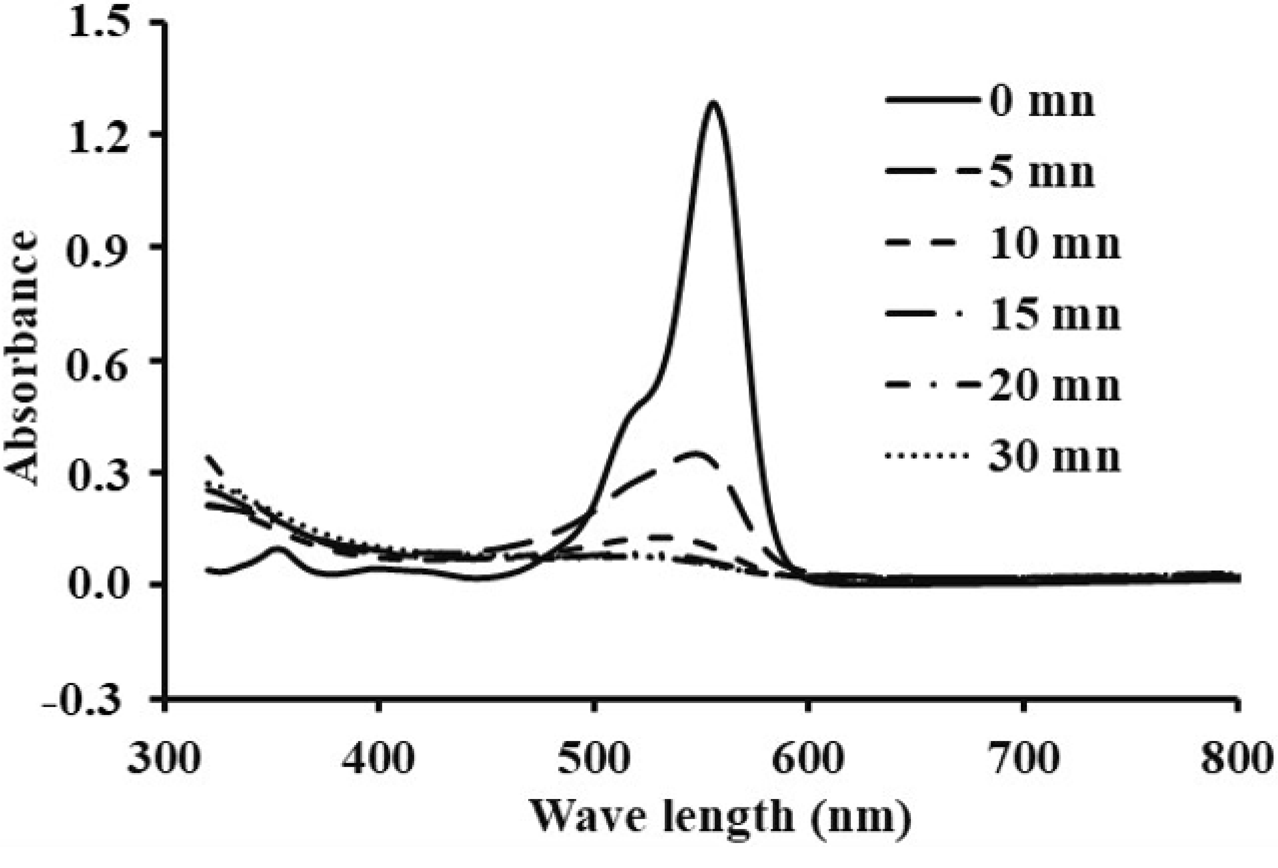

Figure 2

Rhodamine B spectrum (5 mg / L) in the presence of Fe^{2+} (10^{-3} M), H_2O_2 (10^{-2} M), pH = 3

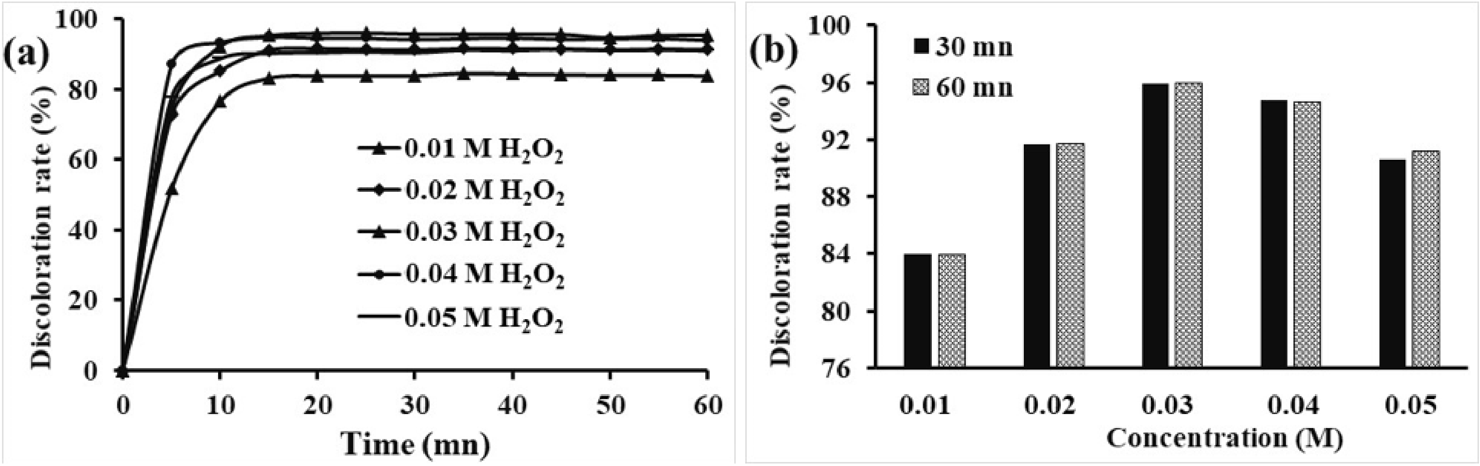

Figure 3

(a) Effect of H_2O_2 concentration on RhB degradation as a function of time; (b) RhB discoloration rate after 30 and 60 minutes; pH = 3; [RhB] = 5 mg / L; $[Fe^{2+}] = 10^{-3}$ M.

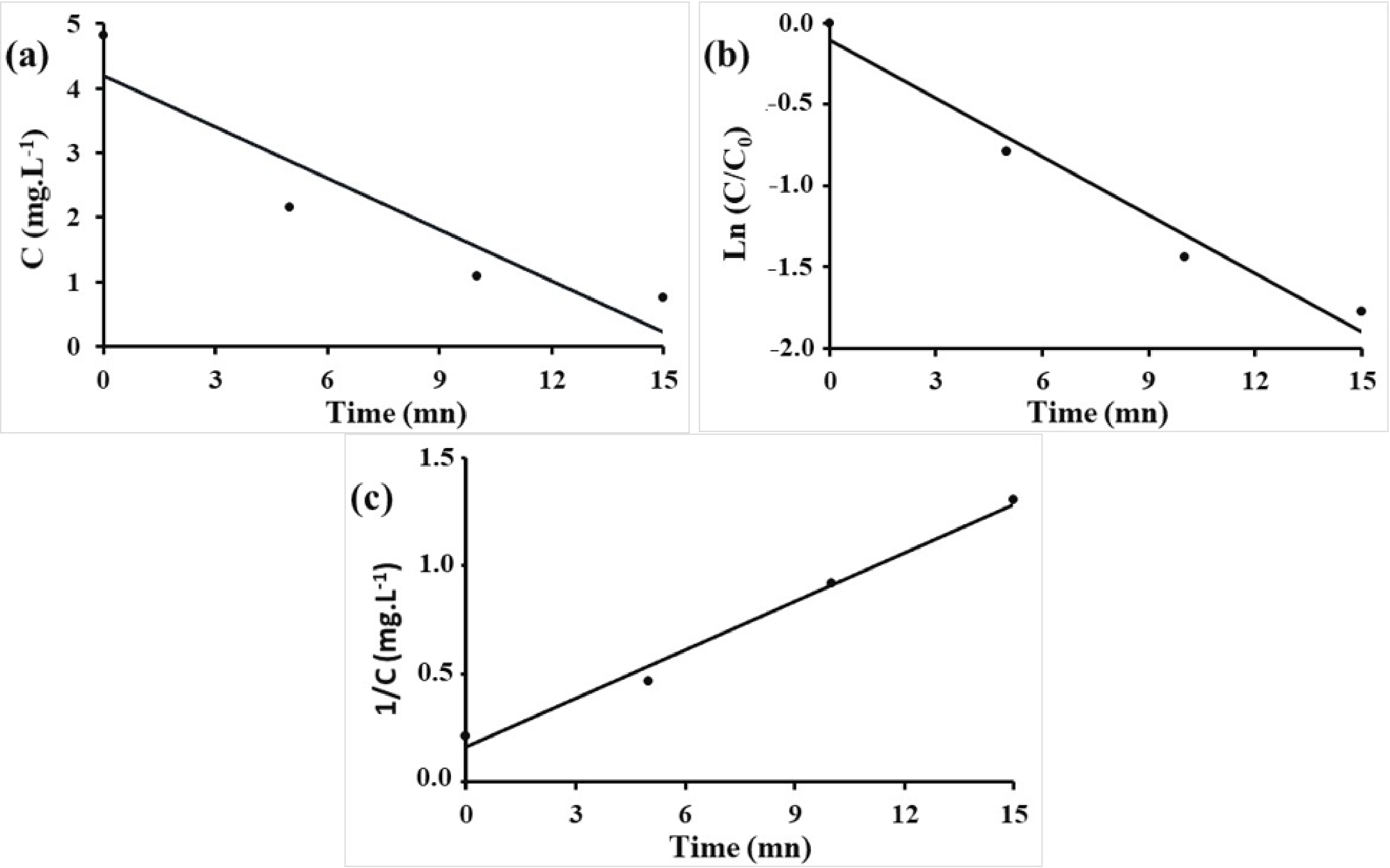

Figure 4

Application of the pseudo-order 0 (a), pseudo-order 1 (b) and pseudo-order 2 (c) models to RhB adsorption; pH = 3; [RhB] = 5 mg / L; $[Fe^{2+}] = 10^{-3}$ M; $[H_2O_2] = 0.1$ M.

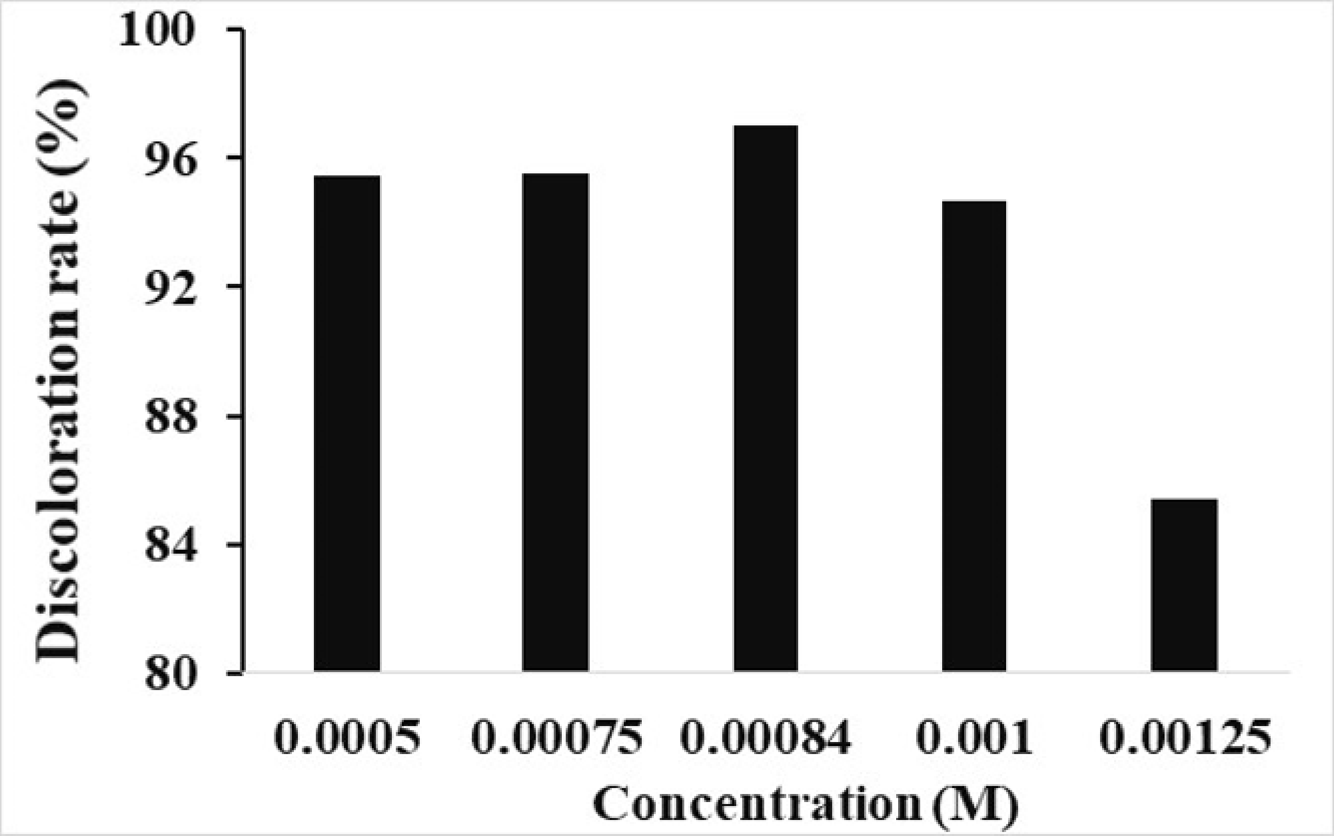

Figure 5

RhB concentration reduction rate for different concentrations of Fe^{2+}; pH = 3; [RB] = 5 mg / L; $[H_2O_2]$ = 3.10^{-2} M.

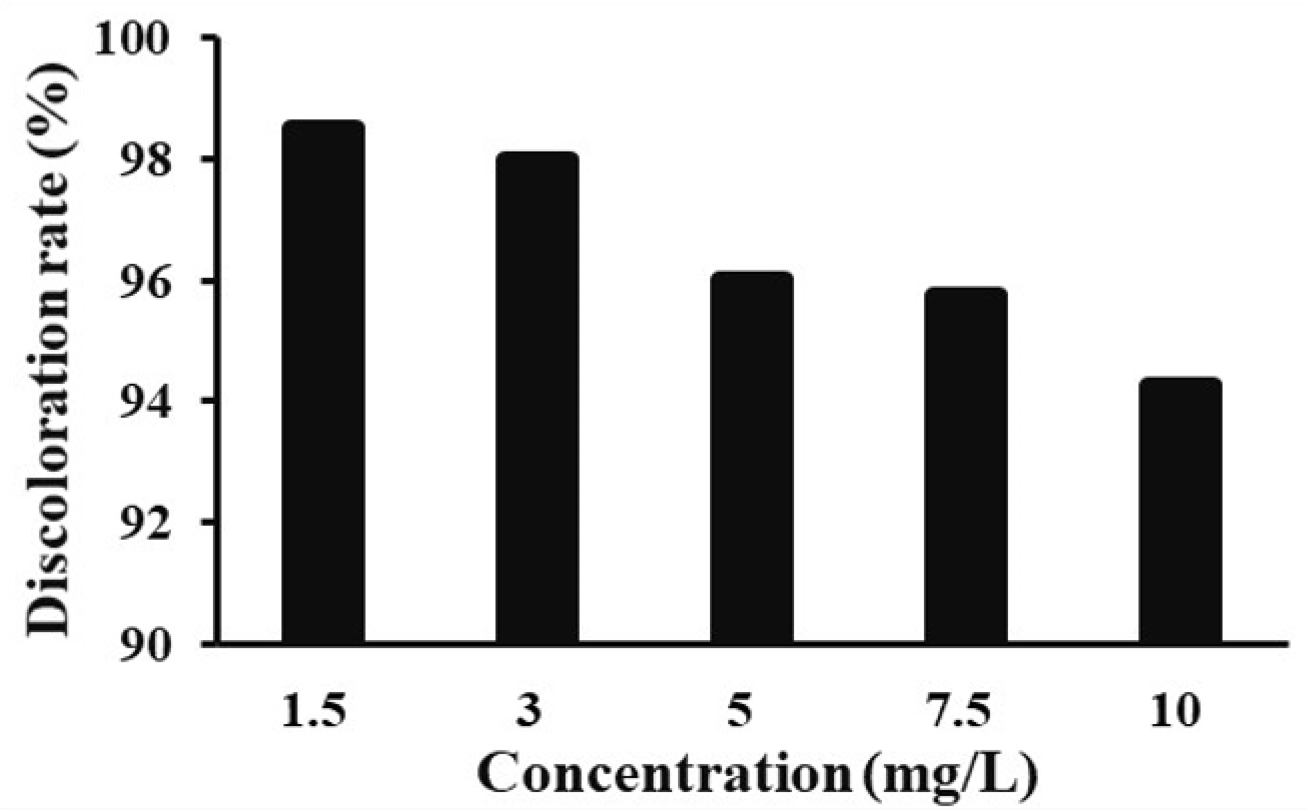

Figure 6

RhB concentration reduction rate for different initial RhB concentration over a period of 20 minutes; pH = 3; [H_2O_2] = 3.10^{-2} M; [Fe^{2+}] = $8.4.10^{-4}$ M

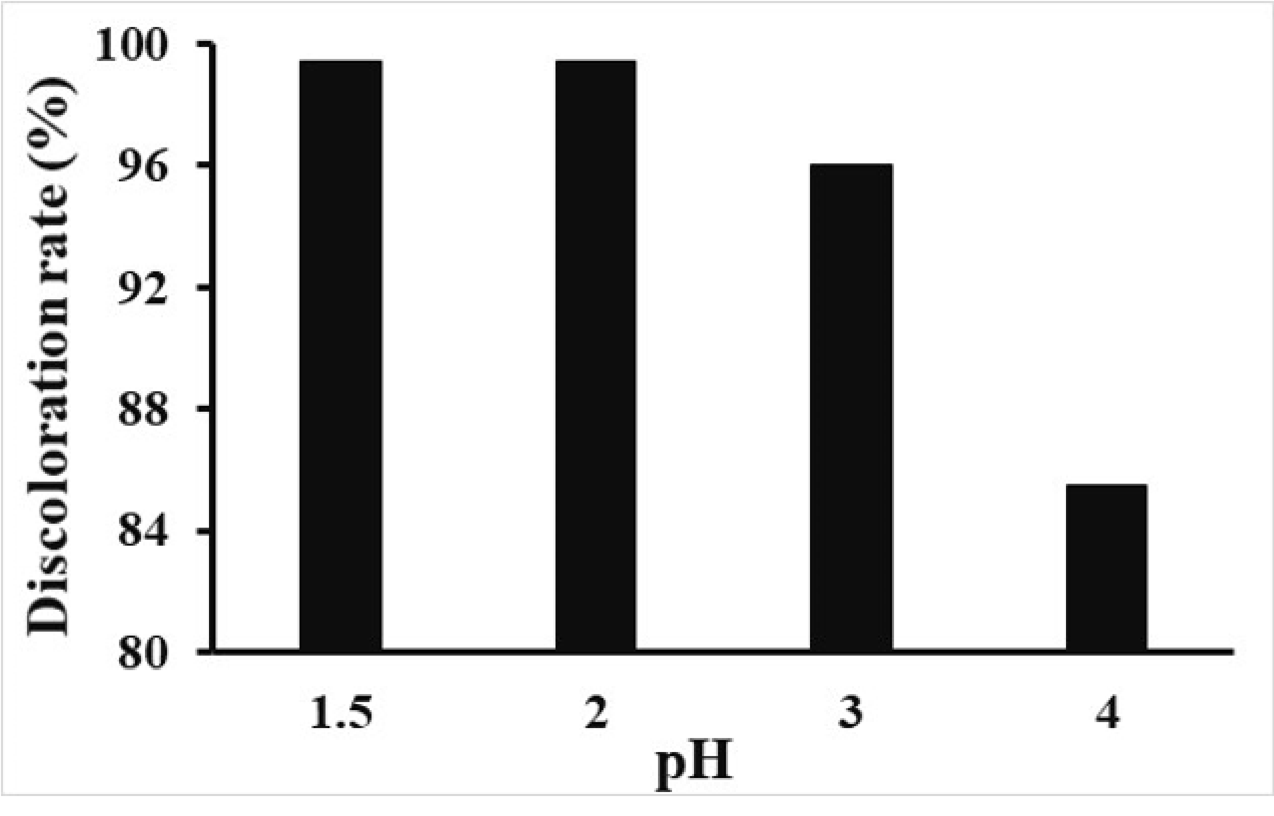

Figure 7

RhB concentration reduction rate for different pHs over a period of 30 minutes; [RhB] = 5 mg / L; $[H_2O_2] = 3.10^{-2}$ M; $[Fe^{2+}] = 8.4.10^{-4}$ M.

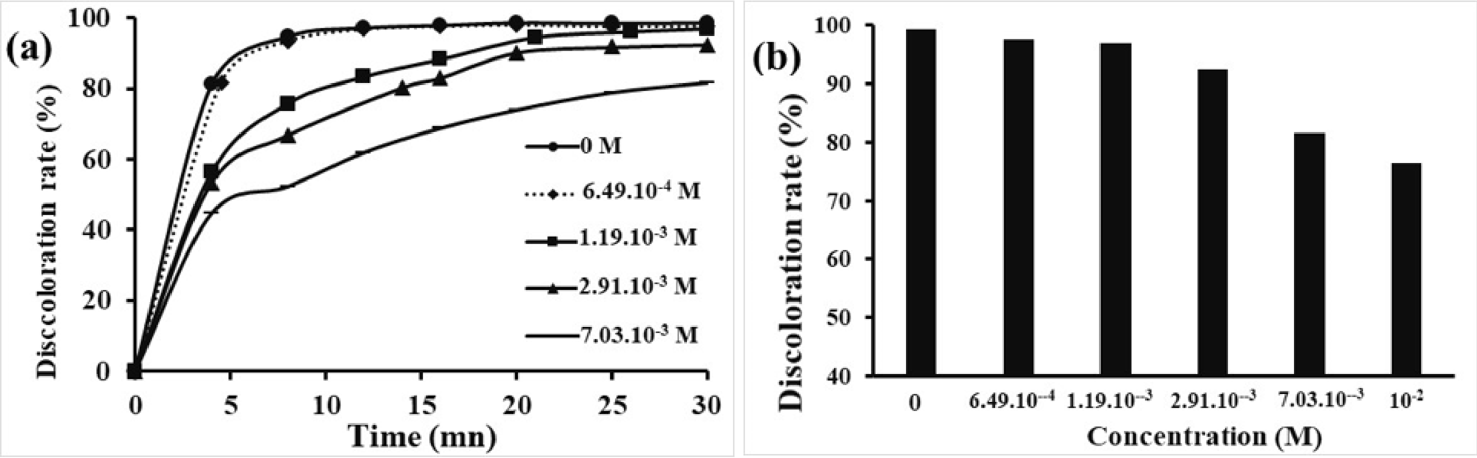

Figure 8

Effect of chloride on RhB discoloration; pH = 2; [RhB] = 5 mg / L; $[H_2O_2] = 3.10^{-2}$ M; $[Fe^{2+}] = 8.4.10^{-4}$ M

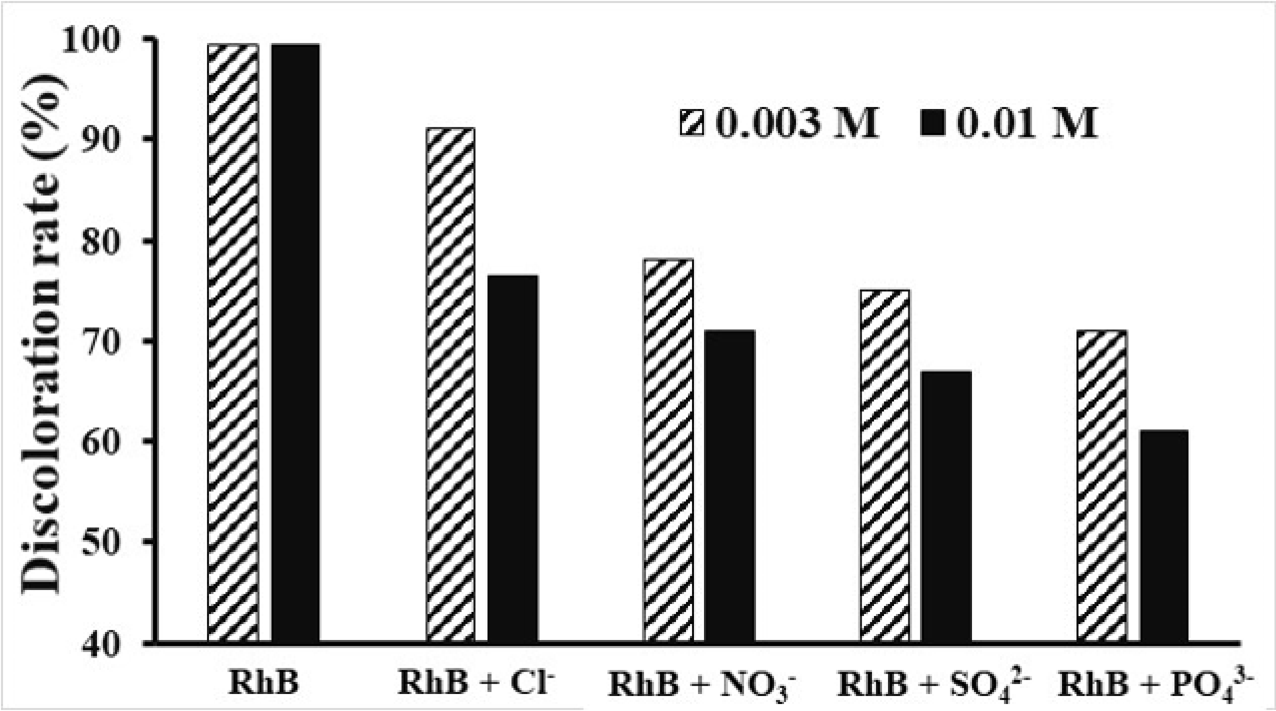

Figure 9

Effect of inorganic ions on RhB degradation; pH = 3, [RhB] = 5 mg / L; $[H_2O_2] = 3.10^{-2}$ M; $[Fe^{2+}] = 8.4.10^{-4}$ M.

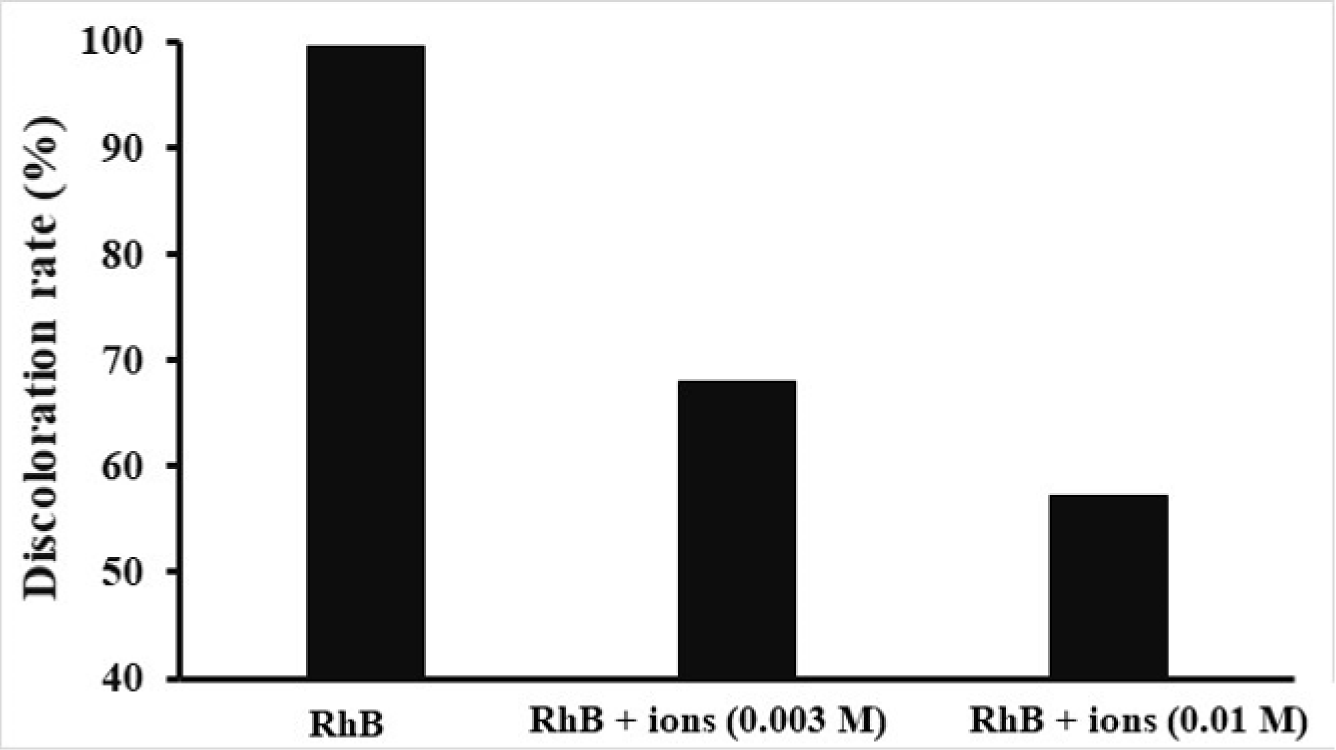

Figure 10

RhB concentration reduction rate in the presence of different inorganic ions (NO_3^- + Cl^- + SO_4^{2-} + PO_4^{3-}) with a concentration of 0.003 M and 0.01 M each; Δt = 30 min, pH = 3; [RhB] = 5 mg / L; [H_2O_2] = 3.10^{-2} M; [Fe^{2+}] = $8.4.10^{-4}$ M.