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Photo-Fenton Degradation of Phenol Red Catalyzed by Inorganic Additives: A Technique for Wastewater Treatment[1]

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Abstract—Oxidation by photo-Fenton like reaction is an economically feasible process for degradation of a variety of hazardous pollutants in wastewater from dyeing and printing industries. In present study, the progress of the reaction has been monitored spectrophotometrically. An effort has been made to observe the effect of various inorganic additives like sodium thiosulfate and potassium bromate. The effect of variation of different parameters such as pH, concentrations of dye, Fe^{3+} ion and additives, amount of H_2O_2, and light intensity on the rate of photodegradation was also observed. A tentative mechanism for the reaction has been proposed.

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The world is facing an ever increasing pace of water pollution with various pollutants like acids, alkalis, detergents, phenols, fungicides etc. which are released from different chemical industries pollute water resources. Dyes used in textile, and other industries are difficult to remove by conventional methods.

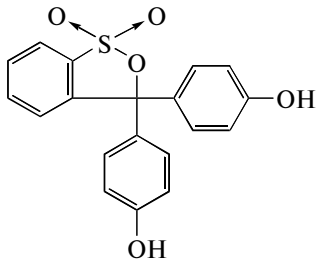
The known technical and economical drawbacks of conventional treatment and biological processes have fuelled the development of new, more effective and economically viable methods for wastewater treatment. In this context, the advanced oxidation processes have enormous potential for becoming viable alternatives for the remediation of polluted water which involve the production of highly reactive hydroxyl radicals and these are of current interest for the destruction of organic pollutants in surface and ground water as well industrial wastewater. The Fenton reagent consisting of H_2O_2 and Fe(II) has been shown to be effective in the degradation of wide spectrum of organic and inorganic pollutants. The mechanism and kinetics of Fenton reaction have been studied by many researchers [1–3]. In the reaction, ferrous ions react with hydrogen peroxide and oxidized to ferric ions with the generation of hydroxyl radicals. These hydroxyl radicals are the active oxidizing species of the degradation of organic compounds. But the main drawback is that this reaction stops after complete consumption of Fe^{2+} ions. The Fe^{2+} ions can be regenerated from Fe^{3+} ions under the present conditions with additional requirement of light. This makes the process cyclic and photodegradation will proceed more smoothly.

A comparative study of degradation of 2-chlorophenol by Fenton and photo-Fenton processes has been done by Kavitha et al. [4]. Decolorization of synthetic dyes by the Fenton reagent and Cu/Pyridine/H_2O_2 system has been investigated by Nerud et al. [5]. Chen et al. [6] studied the photo-Fenton degradation of dye in methanolic solution under both UV and visible irradiation. Matthews [7] reported the photocatalytic oxidation of aromatic compounds generally takes place through the hydroxylation mechanism by $^{\bullet}OH$ radicals. Fernadez et al. [8] studied photoassisted Fenton degradation of non-biodegradable azo dyes (Orange II) in the iron free solution mediated by cation transfer members. The catalytic oxidation of various organic compounds by H_2O_2 has also been reported by many researchers [9–12]. The utility of Fenton reagent for the oxidation of various inorganic compounds has been studied by many groups of researchers [13–15]. A comparative study of Fenton's, photo-Fenton's and other related reagents on the degradation of various types of compounds, e.g. dyes, resorcinol etc has been done [16, 17]. Huang et al. [18] reported the removal of citrate and hypophosphite binary components using Fenton, photo-Fenton and electro-Fenton processes. Methomyl degradation in aqueous solutions by Fenton's reagent and the photo-Fenton system is observed by Tamimi et al. [19]. Optimizing the treatment of landfill leachate by conventional Fenton and photo-Fenton processes has been carried out by Hermosilla et al. [20]. Papic et al. [21] studied the decolourization and mineralization of commercial reactive dyes by using homogeneous and heterogeneous Fenton and UV/Fenton processes, whereas the treatment of wastewater by Fenton and

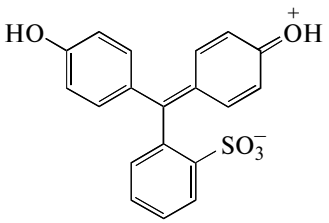
[1] The article is published in the original.

photo-Fenton processes has been investigated by different groups of researchers [22].

Although photo-Fenton reaction is a useful tool for the degradation of dyes as well as organic compounds, the major drawback is its slow rate of degradation. Thus it is important and necessary to find out some better and suitable modifications to increase the efficiency and applications of this reagent. In this context, the rate of photo-Fenton degradation of dye Phenol Red (**A**) is accelerated in the presence of inorganic additives like thiosulfate and bromate ions. Phenol Red can also exist in a zwitter ion form (**B**).



A



B

In the present work, the effect of inorganic additives on the rate of photo-Fenton degradation of Phenol Red was investigated and the main emphasis is given to determine the conditions where these additives show the maximum rate of reaction. An attempt is also made to explain their catalytic behavior.

EXPERIMENTAL DETAILS

All solutions were prepared in doubly distilled water. Irradiation has been carried out with a 200 W tungsten lamp. The light intensity was measured with the help of a solarimeter (CEL, Model SM 201). A water filter has been used to cut off thermal radiations. A digital pH meter (Systronics, Model 335) is used to adjust the pH of the solutions by the addition of previously standardized 0.1 N sulfuric acid and 0.1 N sodium hydroxide solutions. The progress of the photocatalytic reactions was observed by measuring the absorbance at regular time intervals using UV-visible spectrophotometer (Systronics Model 106). Sodium thiosulfate (s.d. Fine), potassium bromate (EM), FeCh (CDH) and Phenol Red (Hi media) have been used as received. Hydrogen peroxide (EM) (concentration 30%, w/v) has been used. An aliquot of 3.0 ml was taken out from the reaction mixture at regular time intervals and the absorbance was measured at $\lambda_{max} = 430$ nm. After measuring the optical density, this solution is further added to reaction mixture, so that the volume of reaction mixture is not changed.

RESULTS AND DISCUSSION

General

It was observed that absorbance of the solution at $\lambda_{max} = 430$ nm decreases with increasing time of exposure, indicating a decrease in concentration of dye Phenol Red. Plots of log of absorbance versus time was linear and follows pseudo-first order kinetics. The rate constant was calculated with the expression

$$k = 2.303 \times \text{slope}.$$

The results are represented in Tables 1 and 2 and by graphs also (Fig. 1). It was observed that reaction is completed in two stages. The first stage has an induction period, may be for the generation of the $^{\cdot}OH$ radicals but during this time a small decrease in absorbance has been observed, which may be attributed as a result of simple photochemical degradation of dye. After this slow stage, a major decrease in absorbance has been observed, which seems to be the second stage of photo-Fenton degradation, where $^{\cdot}OH$ radicals act as oxidizing species for the degradation of dye. All these reactions are shown below.

$$Fe^{3+} + H_2O \xrightarrow[\text{visible}]{h\nu} Fe^{2+} + {}^{\cdot}OH + H^+, \quad \text{(I)}$$

$$Fe^{3+} + H_2O_2 \xrightarrow[\text{visible}]{h\nu} Fe^{2+} + HO_2^{\cdot} + H^+, \quad \text{(II)}$$

$$Fe^{2+} + H_2O_2 \longrightarrow Fe^{3+} + {}^{\cdot}OH + {}^{-}OH, \quad \text{(III)}$$

$${}^{\cdot}OH + H_2O_2 \longrightarrow HO_2^{\cdot} + H_2O, \quad \text{(IV)}$$

$$Fe^{2+} + {}^{\cdot}OH \longrightarrow Fe^{3+} + {}^{-}OH, \quad \text{(V)}$$

$$Fe^{3+} + HO_2^{\cdot} \longrightarrow Fe^{2+} + O_2 + H^+, \quad \text{(VI)}$$

$$\text{Dye} + {}^{\cdot}OH \longrightarrow \text{Colourless degradation products.} \quad \text{(VII)}$$

The effect of various parameters on the rate of degradation is studied by comparing the rate of the second stage of the photo-Fenton reaction. Moreover, when the experimental conditions approach to the optimum conditions, then the time taken by first stage (slow step, induction period) was found to be reduced.

The inorganic additives accelerate the rate of reaction either by reducing the time taken in first stage, i.e., $^{\cdot}OH$ radical generation becomes faster, and/or increasing the rate of second stage.

Table 1. A typical run in Thiosulfate system

Time, min	log of absorbance [arb. units]	
	treated (with additive)	untreated (without additive)
00.0	0.3176	0.3199
05.0	0.3151	0.3197
10.0	0.3148	0.3195
15.0	0.3132	0.3193
20.0	0.3100	0.3186
25.0	0.3042	0.3140
30.0	0.2999	0.3094
35.0	0.2949	0.3072
40.0	0.2844	0.3036
45.0	0.2732	0.2975
50.0	0.2535	0.2946
55.0	0.2660	0.2880
60.0	0.1892	0.2862
65.0	0.1316	0.2734
70.0	0.0481	0.2632
75.0	−0.0360	0.2540
80.0	−0.1993	0.2405
85.0	−0.3392	0.2240
90.0	−0.5004	0.2052
95.0	_	0.1622
100.0	_	0.1152
105.0	_	0.0732
110.0	_	0.0253
115.0	_	−0.0380
120.0	_	−0.0681
125.0	_	−0.1512
130.0	_	−0.2558
	$k_1 = 4.66 \times 10^{-5}$ s^{-1}, $k_2 = 1.21 \times 10^{-3}$ s^{-1}, time taken by first stage 71 min	$k_1 = 2.36 \times 10^{-5}$ s^{-1}, $k_2 = 4.49 \times 10^{-4}$ s^{-1}, time taken by first stage 90 min

Note: [Phenol Red] = 1.33×10^{-4} mol/l, amount of H_2O_2 0.12 ml, [$FeCl_3$] = 3.00×10^{-5} mol/l, [Thiosulfate] = 1.33×10^{-5} mol/l, light intensity 60.0 mW/cm, pH 3.50.

Table 2. A typical run in Bromate system

Time, min	log of absorbance [arb.units]	
	treated (with additive)	untreated (without additive)
0.0	0.2714	0.2650
5.0	0.2645	0.2583
10.0	0.2593	0.2540
15.0	0.2535	0.2484
20.0	0.2464	0.2417
25.0	0.2375	0.2289
30.0	0.2250	0.2172
35.0	0.2089	0.1994
40.0	0.1832	0.1717
45.0	0.1464	0.1296
50.0	0.0831	0.0648
55.0	−0.0561	−0.0625
60.0	−0.4318	−0.3915
65.0	−1.0968	−0.8631
70.0	−1.3468	−1.1986
	$k_1 = 6.74 \times 10^{-5}$ s^{-1}, $k_2 = 3.35 \times 10^{-3}$ s^{-1}, time taken by first stage 52 min.	$k_1 = 6.72 \times 10^{-5}$ s^{-1}, $k_2 = 2.86 \times 10^{-3}$ s^{-1}, time taken by first stage 57 min.

Note. [Phenol Red] = 1.00×10^{-4} mol/l, amount of H_2O_2 0.1 ml, [$FeCl_3$] = 2.33×10^{-5} mol/l, [Bromate] = 5.33×10^{-5} mol/l, light intensity 60.0 mW/cm, pH 3.00.

Effect of pH

The effect of pH on additive catalyzed photochemical degradation was investigated in the pH range 2.5–4.0. The results are reported in Fig. 2.

The rate of photo-Fenton degradation of Phenol Red was maximum at pH 3.50 and 3.00 for thiosulfate and bromate system, respectively. The observations indicate that the rate of photo-Fenton reaction strongly depends on the pH of the system. In the system, the hydroxyl radicals are generated by steps (I) and (III) of the mechanism. These $^{\bullet}OH$ radicals are involved in the photodegradation of the dye through oxidation process. In steps (I), (II), and (VI) of mechanism, Fe^{3+} ions are reduced to Fe^{2+} ions by the reaction with H_2O, H_2O_2, and $HO_2^{\bullet}$, respectively. Regeneration of ferric ions can be occurred due to the reaction with inorganic additives (XI–XIII, see further). At lower pH value (≤2.0) the steps (I) and (II) are comparatively slower in which formation of H^+ ions occurred. As the pH of the medium was increased, the rate of reaction also increases because step (I) and (II) become faster. The increase in the rate continues up to pH 3.50, and 3.00 for thiosulfate and bromate, respectively. Beyond these specific pH values for each additive, the reaction rate again decreases. This may be attributed to the retardation of step (III) in which Fe^{3+} ions an $^{\bullet}OH$ radicals are regenerated with the simultaneous formation of ^{-}OH ions.

Effect of the Amount of Hydrogen Peroxide

The effect of amount of H_2O_2 on the photocatalytic degradation of Phenol Red was also investigated. The results are represented in Fig. 3.

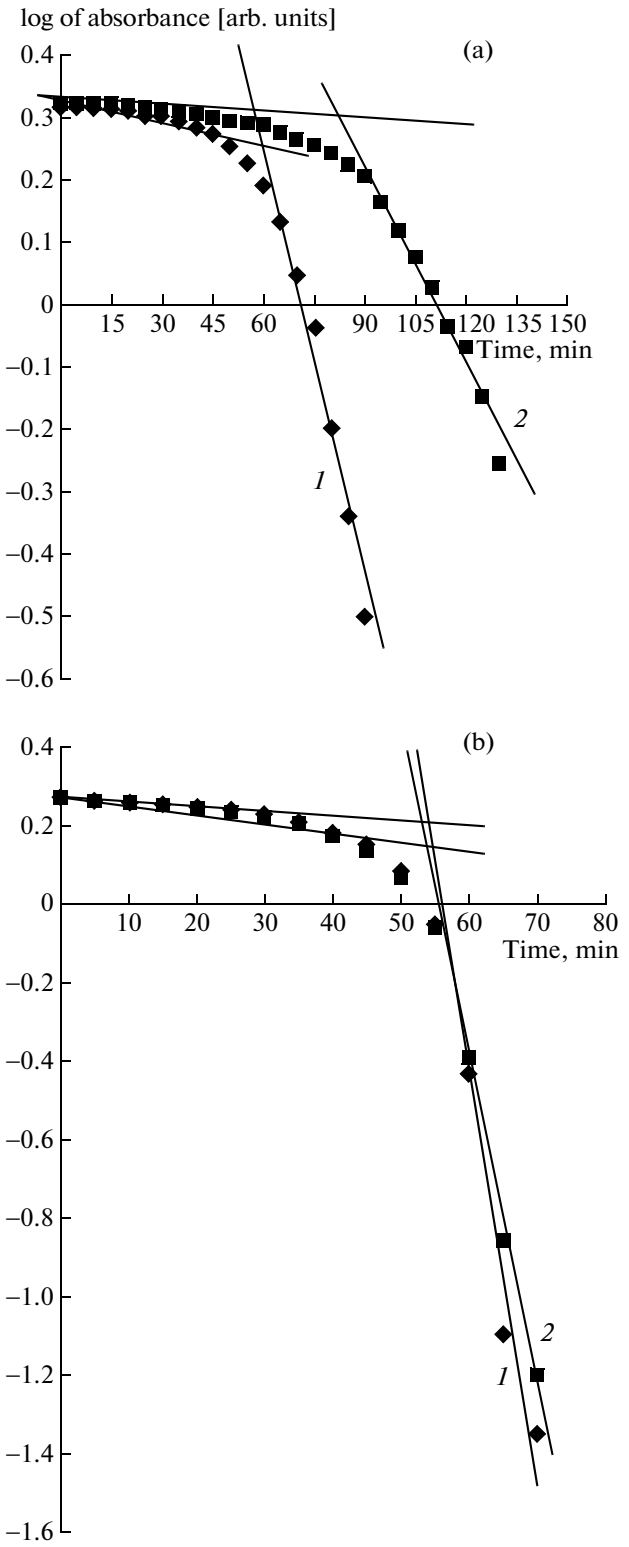


Fig. 1. A typical runs in Thiosulfate (a) and Bromate (b) systems: *1*—with additive, *2*—without additive.

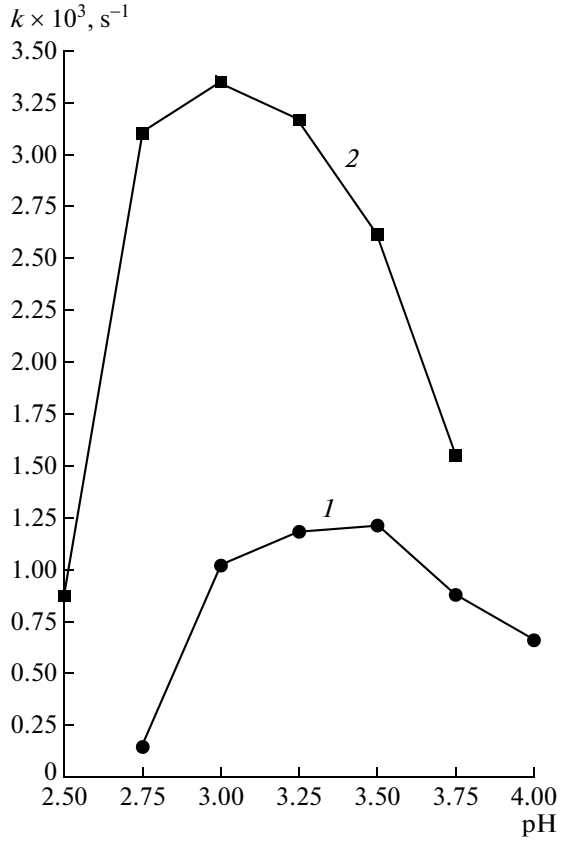


Fig. 2. Effect of pH in Thiosulfate (*1*) and Bromate (*2*) systems. Reaction conditions see in Tables 1 and 2, respectively.

The rates were determined in the range from 0.04 to 0.28 ml and from 0.00 to 0.70 ml for thiosulfate and bromate system, respectively. The photo-Fenton degradation of Phenol Red was maximum at 0.12 and 0.10 ml for thiosulfate and bromate systems, respectively. Further increase in H_2O_2 amount beyond these limits causes retardation of reaction. This may be explained on the basis that as the amount of H_2O_2 was increased, the rate of reaction increases due to acceleration of step (III), in which H_2O_2 reacts with Fe^{2+} ion and generates $^{\bullet}OH$ radicals but a further increase in amount causes the acceleration of step (IV) in which H_2O_2 reacts with $^{\bullet}OH$ radicals and as a consequence, a decrease in the rate of final step (VII) of mechanism has been observed.

Effect of Concentration of Additives

The effect of variation of concentration of additives on the photocatalytic degradation of Phenol Red was also investigated. The results are shown in Fig. 4.

It was observed that rate of reaction increases with the increase in concentration of additives up to 1.33×10^{-5} and 5.33×10^{-5} mol/l for thiosulfate and bromate, respectively. On further increase in concentration of additives beyond this limit, the rate of reaction decreases. In the thiosulfate system, ferrous ions react with hydrogen peroxide and itself oxidized to fer-

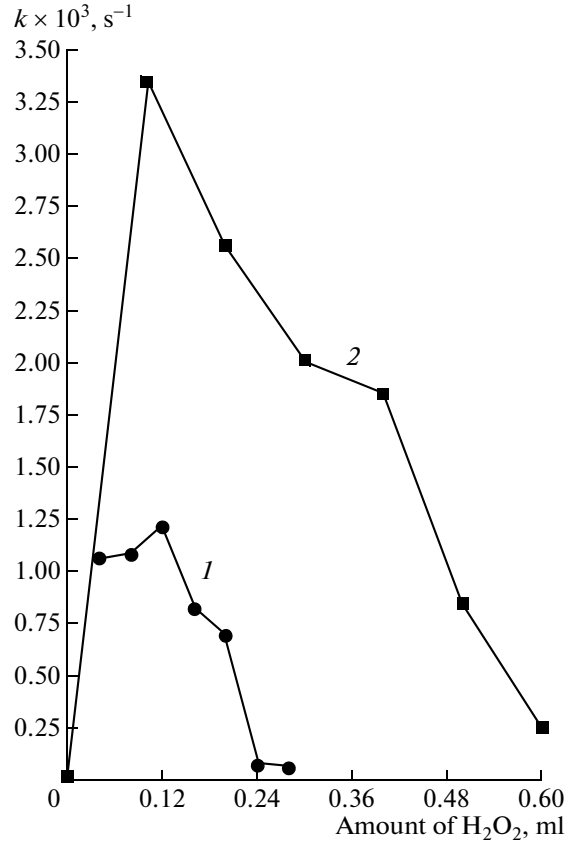


Fig. 3. Effect of amount of hydrogen peroxide in Thiosulfate (*1*) and Bromate (*2*) systems. Reaction conditions see in Tables 1 and 2, respectively.

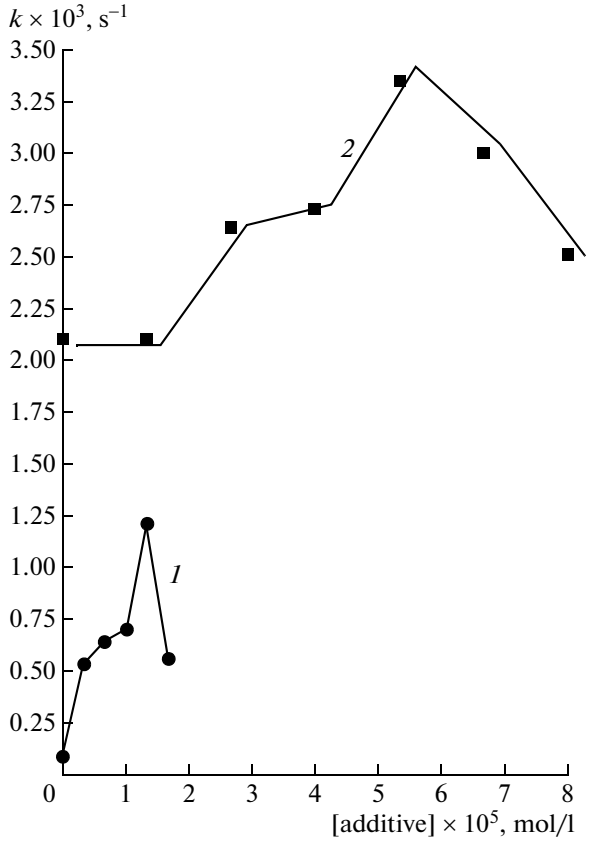


Fig. 4. Effect of additive concentration in Thiosulfate (*1*) and Bromate (*2*) systems. Reaction conditions see in Tables 1 and 2, respectively.

ric ions with the simultaneous formation of hydroxyl radicals. The thiosulfate ions are oxidized to tetrathionate by the reaction with ferric ions, which itself reduce to ferrous ions and by this process, thiosulfate ions provide continuous availability of ferrous ions in the system (step VIII). Further, these thiosulfate ions are again regenerated in the system by the reaction with hydroxyl ions (step IX). Thus, it accelerates the rate of degradation by making the process cyclic. For thiosulfate system:

$$2S_2O_3^{2-} + 2Fe^{3+} \longrightarrow S_4O_6^{2-} + 2Fe^{2+}, \quad \text{(VIII)}$$

$$2S_2O_6^{2-} + 6OH^- \longrightarrow 3S_2O_3^{2-} + 2SO_3^{2-} + 3H_2O, \text{(IX)}$$

$$2S_2O_6^{2-} + H_2O_2 + 2H^+ \longrightarrow S_4O_6^{2-} + 2H_2O. \quad \text{(X)}$$

Bromate ions accelerate the rate of reaction by regenerating the ferric ions in the system. Fe^{+2} ions can react with BrO_3^-, BrO_2, and BrO_2^- ions and oxidized to Fe^{3+} ions (step XI–XIII). Thus, make the process cyclic. For bromate system:

$$Fe^{+2} + BrO_3^- + 2H^+ \longrightarrow Fe^{3+} + BrO_2 + H_2O, \quad \text{(XI)}$$

$$BrO_2 + Fe^{2+} \longrightarrow BrO_2^- + Fe^{3+}, \quad \text{(XII)}$$

$$BrO_2^- + 4Fe^{+2} + 4H^+ \longrightarrow 4Fe^{3+} + Br^- + 2H_2O. \text{(XIII)}$$

The effect of inorganic additives is so important that these can significantly accelerate the Fenton reaction of even at very low concentrations. Moreover, the data show that these additives do not behave only as a catalyst, but also as a reactant. It case of thiosulfate system, after the certain concentration, it may react with H_2O_2 and oxidized directly to tetrathionate instead of giving Fe^{2+} ions in the system (step X) and as a result, the rate of degradation decreases after a maxima has been obtained.

The photo-Fenton degradation rates of Phenol Red with these inorganic additives is in the order Thiosulfate > Bromate.

The efficiency of thiosulfate ions is higher than that of bromate ions, which may be due to the regeneration of thiosulfate ions in the system, while in these conditions, there is no regeneration of bromate ions in the system.

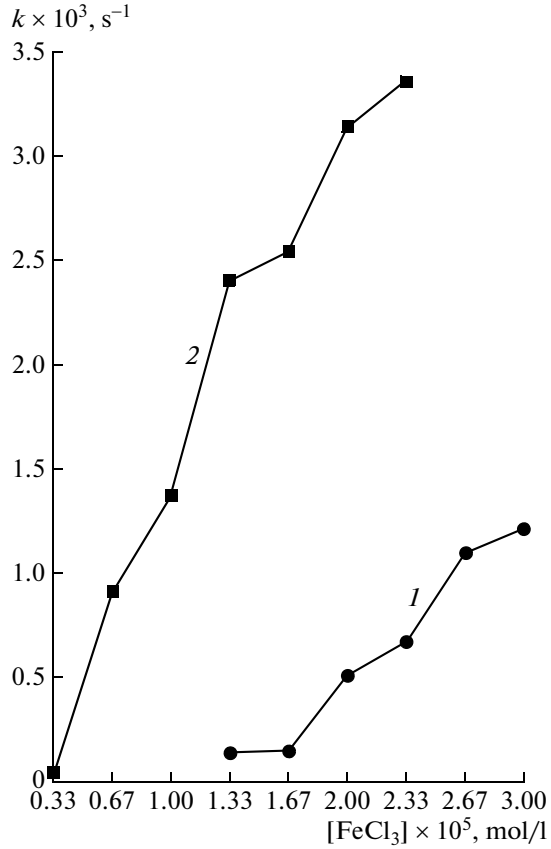


Fig. 5. Effect of ferric chloride concentration in Thiosulfate (*1*) and Bromate (*2*) systems. Reaction conditions see in Tables 1 and 2, respectively.

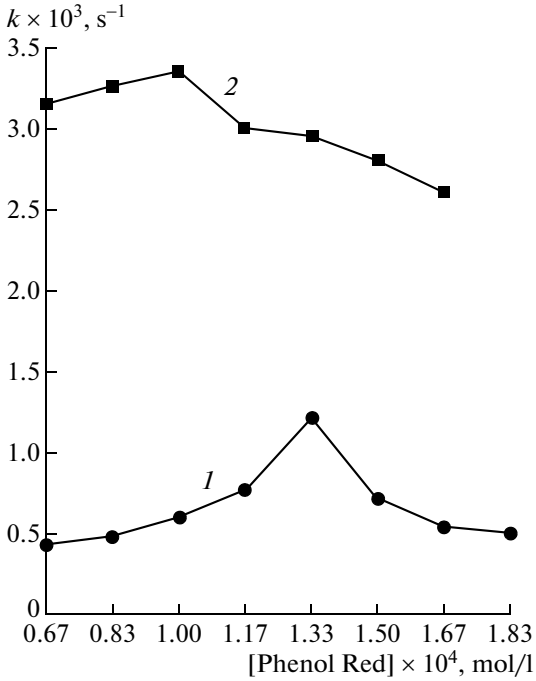


Fig. 6. Effect of Phenol Red concentration in Thiosulfate (*1*) and Bromate (*2*) systems. Reaction conditions see in Tables 1 and 2, respectively.

With both the additives, initial induction period was observed when the experimental conditions were away from optimum. By employing these inorganic additives, this period becomes shorter.

Effect of Ferric Chloride Concentration

The effect of concentration of ferric ions on the rate of photocatalytic degradation of Phenol Red was observed by keeping all other factors identical. The results are summarized in Fig. 5. It is clear from the graph that the rate of photo-Fenton degradation increases on increasing concentration of ferric chloride. The rates were determined for ferric ion concentration up to concentration 3.00×10^{-5} and 2.33×10^{-5} mol/l for thiosulfate and bromate systems, respectively.

It was observed that the rate of reaction increased as the concentration of $FeCl_3$ increased. Beyond this concentration, the rates were extremely high and it was not possible to record the observations correctly due to experimental limitations. This increasing trend may be explained on the basis that on increasing the concentration of ferric chloride, the concentration of hydroxyl radicals and Fe^{2+} ions increases in the system (steps I and II), which further enhanced the generation of $^{\bullet}OH$ radicals (step III) and as a consequence, the rate of photochemical degradation also increases.

Effect of Phenol Red Concentration

The effect of Phenol Red concentration on the rate of its photo-Fenton degradation was observed and the results are shown in Fig. 6.

The rate of photo-Fenton degradation was found to increase with increasing concentration of Phenol Red up to 1.33×10^{-4} and 1.00×10^{-4} mol/l for thiosulfate and bromate system, respectively. Further increase in concentration beyond these limits results in decrease in the rate of degradation. This may be explained on the basis that on increasing the concentration of Phenol Red, the reaction rate increases, since more molecules of dye were available for degradation. But further increase in concentration causes retardation of reaction due to increase in number of collisions between dye molecules themselves, whereas probability of collisions between dye and $^{\bullet}OH$ radicals will decrease. As a consequence, retardation in rate of reaction was observed. Unsuitable steric orientation is also one of the factor for a decrease in the rate of reaction. Further, at higher concentrations, dye molecules themselves can act as a filter for incident light which causes decrease in the rate of reaction.

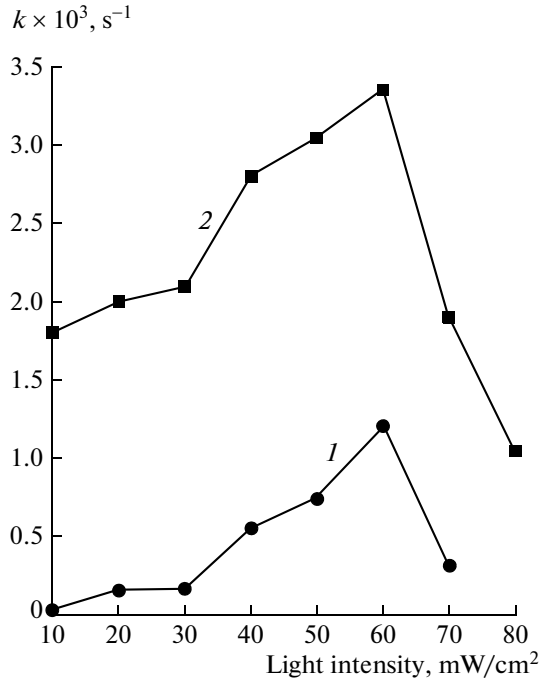


Fig. 7. Effect of light intensity in Thiosulfate (*1*) and Bromate (*2*) systems. Reaction conditions see in Tables 1 and 2, respectively.

Effect of Light Intensity

The effect of light intensity on the photo-Fenton degradation of Phenol Red was investigated. The results are represented in Fig. 7.

The data indicate that as the light intensity was increased, the rate of reaction also increases and maximum rate of reaction have been found at 60.0 mW/cm in both systems. Because with the increased light intensity, the number of photons increases, which accelerate the step (I) and (II) and as a result, the rate of reaction is increased. Further increase in the intensity results in a decrease in the rate of reaction, may be due to thermal side reactions.

MECHANISM OF PHENOL RED DEGRADATION

On the basis of the experimental observations and corroborating existing literature, a tentative mechanism has been proposed for the degradation of Phenol Red by photo-Fenton's reagent in presence of inorganic additives, which has been represented by equations (I)–(VII).

The aqueous solution of ferric ions on exposure to light generates a proton and $^{\bullet}OH$ radicals and it is reduced to ferrous state. These ferrous ions further react with H_2O_2 producing hydroxyl ions and hydroxyl radicals and oxidized back to ferric ions. These hydroxyl radicals are the most probable active species in the degradation of dye.

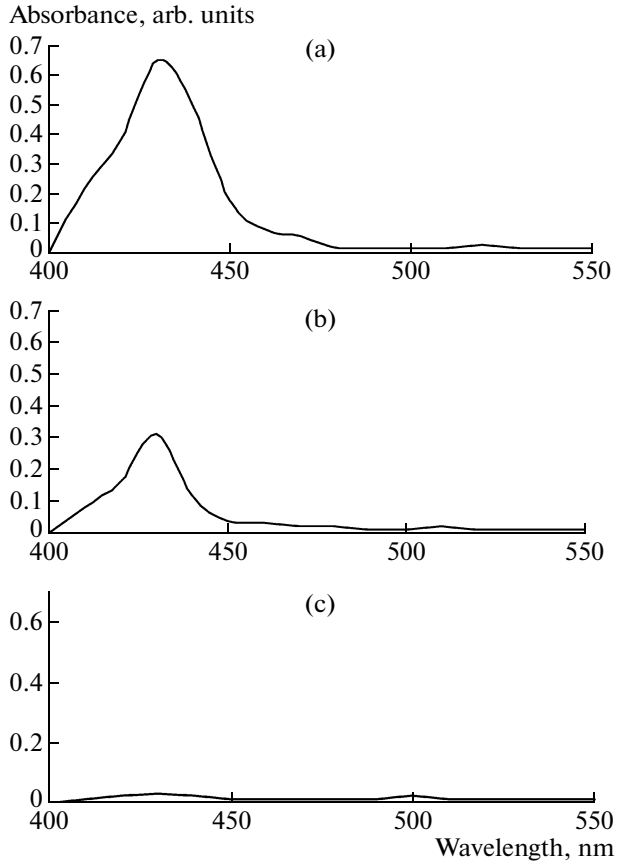


Fig. 8. Absorption spectra of reaction mixture: (a) before experiment, (b) in the middle of experiment, (c) at the end of experiment.

Inorganic compounds can play an important role in catalyzing the Fenton reaction. These have a significant role in the continuous availability of ferrous and ferric ions in the system, which accelerate the reaction even at low concentration of ferric ions, thus, make the process less hazardous to aquatic ecosystem. All the reactions have been previously explained in the section of effect of additive concentration (stages VIII–XIII).

The hydroxyl radicals degrade the dye Phenol Red. The participation of $^{\bullet}OH$ radicals as an active oxidizing species was confirmed by using hydroxyl radical scavengers, e.g., isopropanol, where the rate of photodegradation was drastically reduced. The UV-Vis spectra of reaction mixture shown in Fig. 8 demonstrate the decrease in the concentration of Phenol Red with the reaction time.

Further, this method has more advantages over other methods. It does not add to pollution any further. The active oxidizing species, i.e., the hydroxyl radicals, will dimerize to give hydrogen peroxide, which may degrade ultimately to water and oxygen. In this reaction, very low concentration of chemicals is used and the process is cyclic in nature, it can be con-

sidered as one of the environmentally friendly methods for the treatment of waste water.

Thus, the rate of photo-Fenton degradation of Phenol Red is enhanced by inorganic additives. The increasing order of the rate with different additives is as follows: Thiosulfate > Bromate.

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