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Decoloration of three azo dyes in water by photocatalysis of Fe (III)–oxalate complexes/H_2O_2 in the presence of inorganic salts

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Abstract

Coloration of textiles is improved by inorganic salts, which increase pollution load on dyehouse effluent in general. In this paper, four inorganic salts including sodium chloride, sodium sulphate, sodium nitrate and potassium chloride were used for investigating their effects on photocatalytic decoloration of three azo dyes, Mordant Black PV, Reactive Blue MS and Direct Sky Blue 5B in water based on Fe (III)–oxalate complexes/H_2O_2 system in a specially designed photoreactor. Also the photocatalytic degradation and mineralization of the dyes were examined by UV–vis spectrum and TOC measurements. The results indicate that photocatalytic decoloration of the dyes is significantly limited in the presence of salts. Increasing concentration of inorganic salts in water can give rise to reduced decoloration efficiency and kinetic reaction constants of the dyes. Sodium chloride and potassium chloride exhibit stronger inhabiting power on photocatalytic decoloration and degradation than sodium sulphate and sodium nitrate under the same reaction condition.

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Keywords: Azo dyes; Inorganic salts; Decoloration; Fe (III)–oxalate complexes

1. Introduction

Textile dyeing is a significant consumer of water and producer of contaminated aqueous waste streams. The dyeing processes require the addition of inorganic salts and discharges from the dyeing baths and subsequent washing procedure contain both salts and residual dyes. Inorganic salts have different functions in exhaustion or padding dyeing of textile products according to dyes and fibers used. In general, they can serve as exhausting and retarding agents. For example, they are used as exhausting agents or promoters in the coloration of cotton fabrics with direct dyes and reactive dyes. Because reactive dyes have a lower affinity than direct dyestuffs, more inorganic salt is required when using reactive dyes in order to improve the affinity of the dyes and accelerate absorption through ‘‘salting out’’ effect [1]. On the other hand, inorganic salts are employed as retarding or leveling agents in the application of acid dyes on wool and basic dyes on acrylics. Both sodium chloride and sodium sulfate are the very commonly used inorganic salts in dyeing of textiles. The addition of these inorganic salts in the dye bath increases the pollution load on the effluent generated as well as has an effect on various effluent treatment processes. Additionally, commercial azo dyes used in textile coloration are mixtures of dyes including other additives such as intermediates produced during dye synthesis and inorganic salts such as sodium chloride and sodium sulfate [2].

The photochemistry of Fe (III)–oxalate complexes had been studied since the 1950s. They are photochemically active under sunlight because these species exhibit strong ligand-to-metal charge absorption bands in the near-UV and visible region. Fe (III)–oxalate complexes can generate Fe (II) ions when irradiated and the Fe (II) ions can in turn participate

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in the Fenton reaction and generate additional OH radical. Thus, the photolysis of ferrioxalate in the presence of hydrogen peroxide provides a continuous source of Fenton' reagent. The hydroxyl radical is a powerful oxidant and starts a cascade of oxidation reactions that can ultimately lead to total mineralization of organic pollutants [3,4]. However, much attention has been paid on Fe (III)−oxalate complexes until 1990s because the ferric ions and oxalate ions are the two major species in natural water and the photolysis of Fe (III)−oxalate complexes was an important pathway for the H_2O_2 in atmospheric water [5]. Also, Fe (III)−oxalate complexes system has higher efficiency than other ferric species systems in photooxidation and degradation of organic compounds. In the past years, Fe (III)−oxalate complexes were used as a kind of catalyst or promoter in the photodegradation of textile dyes in water [6−8] and are expected to play an important role in degradation and mineralization of textile dyes in the days to come.

The survey of literature revealed that there are a few studies on the effect of inorganic salts on the decoloration of textile dyes by using advanced oxidation technology. Gahr and co-authors [9] made a brief mention of the effect of salt during ozone decoloration of reactive dye effluent and reported that at higher pH levels, the presence of salt lowers the decomposition of reactive dyes. Muthukumar and Selvakumar [10] found that salt content in the effluent increases the decoloration time of the acid dye effluents and among the salts, sodium chloride requires more time than sodium sulphate. Another work also deals with the interaction between chloride ions and OH radicals in photocatalytic treatment of reactive dye based on TiO_2. It was indicated that chloride ions could react with OH radicals and reduce the amount of OH radicals available for the oxidation reaction [2]. Research on the effect of dye assisting chemicals, such as sodium chloride on photochemical oxidation of reactive azo dye with UV−H_2O_2 process was conducted by Muruganandham and Swaminathan [11]. It is concluded that addition of sodium chloride does not affect the removal rate significantly. Sokmen and Ozkan [12] reported that the catalytic activity of TiO_2 was significantly retarded in the presence of common inorganic anions in the photocatalytic degradation of textile dye Astrazone Orange G. But it is noticed that most of these work were undertook by ozone decoloration or photocatalytic treatment based on TiO_2, very few studies focused on the decoloration of textile dyes through photocatalytic oxidation of ferric oxalate complexes/H_2O_2 system in the presence of inorganic salts.

In this work, photocatalytic decoloration of three azo dyes, Mordant Black PV, Reactive Blue MS and Direct Sky Blue 5B, in water was carried out by using Fe (III)−oxalate complexes/H_2O_2 system in a specially designed reactor under a high pressure mercury lamp in the presence of inorganic salts. The effects of sodium chloride, potassium chloride, sodium sulfate and sodium nitrate on decoloration of the dyes were studied. Moreover, the photocatalytic degradation of the three dyes was examined and compared by UV−vis spectrum. In addition, mineralization of the dyes in the presence of sodium chloride was investigated through TOC measurements.

2. Experimental

2.1. Reagents and chemicals

Three original dyes Mordant Black PV, Reactive Blue MS and Direct Sky Blue 5B were provided by Tianjin Sanhuan

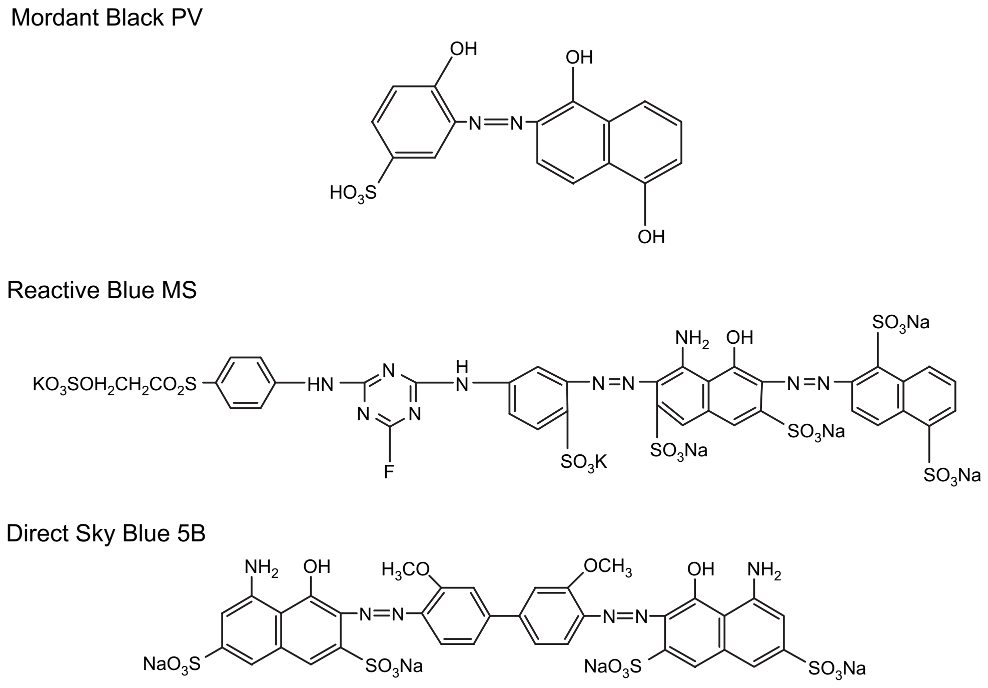
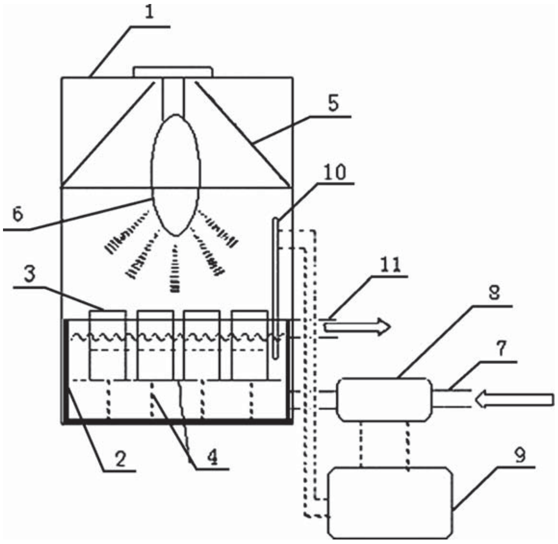


Fig. 1. Chemical structure of the three azo dyes.



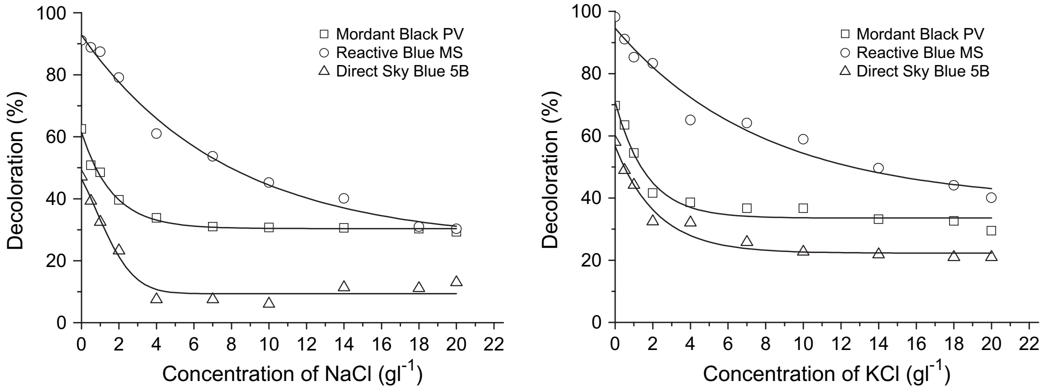
1.chamber, 2.water bath, 3.glass vessel, 4.support, 5.lamp-chimney, 6.mercury lamp, 7.water in, 8 electromagnetic valve, 9.relay, 10.thermometer, 11.water out

Fig. 2. The schematic diagram of photoreaction system.

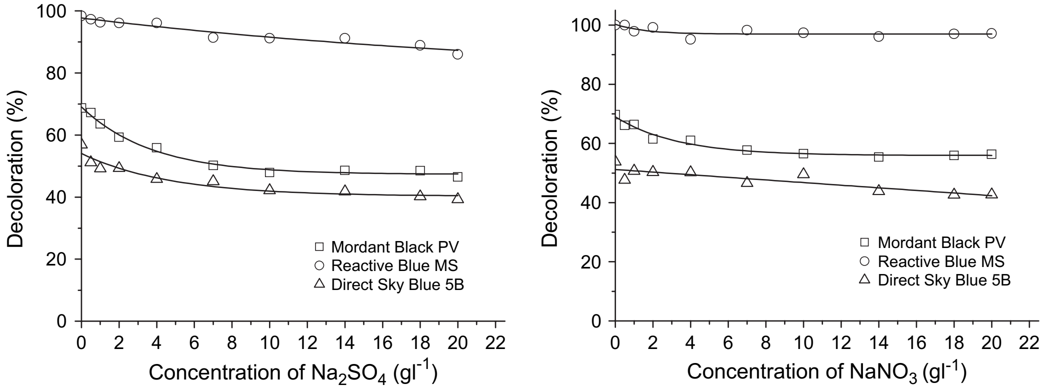
Chemical Company, China and used in this experiment without further purification. Analytical grade ferric sulfate and oxalic acid were the sources of Fe (III) ions and oxalate, respectively. Hydrogen peroxide (30% w/w) was of analytical grade and sodium chloride, sodium sulfate, sodium nitrate and potassium chloride were of reagent grade. These chemicals were obtained from Tianjin Chemical Reagent Company, China. Double distilled and deionized water was used throughout the study. The chemical structures of the three dyes are shown in Fig. 1.

2.2. Photoreactor and light source

The photoreaction system was specially designed in this experiment and consists mainly of chamber, lamp, electromagnetic valve, relay, and water bath. A 400 W high pressure mercury lamp (Osram Foshan Illuminance Company, China) was used for photoreaction. The chamber was made of polished aluminum because it is highly reflective in the UV and visible light range. Ten open Pyrex vessels of 150 ml capacity were used as reaction receivers in water bath. An electromagnetic valve was operated by relay and used to control temperature in water bath. Light intensity inside photoreaction system was measured as 10.1 mW cm^{-2} (400–1000 nm), 0.93 mW cm^{-2} (365 nm) and 30 500 lx using FZ-A radiometer, UV-A radiometer (BNU Light and Electronic Instrumental Factory, China) and TES 1332 digital light meter (TES Electrical Electronic Corp. Taiwan), respectively. The schematic diagram of photoreaction system is presented in Fig. 2.



(a) NaCl (b) KCl



(c) Na_2SO_4 (d) $NaNO_3$

Fig. 3. Decoloration of the three azo dyes at different concentrations of inorganic salts.

2.3. Procedure

Stock dye solutions were prepared by dissolving 5.0 g of each dye in 1 l of boiling deionized water. Test solutions were made by diluting the stocks to typical effluent dye concentration (50 mg l^{-1}) and followed by addition of ferric sulfate, oxalic acid and hydrogen peroxide. Care should be taken that it contains 50 mg l^{-1} dyes, 0.2 mmol l^{-1} Fe (III), 0.6 mmol l^{-1} oxalate ions and 1.0 mmol l^{-1} hydrogen peroxide at pH 3.5 ± 0.1. Test solutions (100 ml) were then placed into reaction vessels and exposed to irradiation of lamp in photoreaction system. At different time intervals, 1–2 ml of the sample was withdrawn and diluted suitably, and the absorbance of which was measured at the $\lambda_{\max}$ of the dye solutions. Concentration of dyes was determined and UV–vis spectrum from 190 to 800 nm was recorded with a UV-2401 spectrophotometer (Shimadzu Company, Japan). The pH of the solution was measured by using DHS-25C digital pH meter (Shanghai Jingmi Instrumental Company, China). The temperature in reaction vessel was kept at 25 ± 1 °C. The decoloration percentage of the dyes was expressed as:

$$\text{Decoloration } (\%) = \frac{C_0 - C}{C_0} 100\%$$

where C_0 is the initial concentration of the dye (mg l^{-1}) and C is the residual concentration of the dye (mg l^{-1}). Total Organic Carbon (TOC) was measured with a 1020A TOC analyzer (OI Corporation, USA).

3. Results and discussion

3.1. Concentration of inorganic salts

Fig. 3 shows that decoloration of the three dyes, especially Reactive Blue MS declined significantly with increasing concentration of sodium or potassium chloride from 0.5 g l^{-1} to 20 g l^{-1}. This is owing to the hydroxyl radical scavenging effect of chloride ion. Similar retardation by the addition of sodium chloride has been reported in ozonation [10], UV–H_2O_2 process [11] and UV–TiO_2 process [12]. Another possible explanation is that addition of salts such as sodium chloride is believed to increase the degree of aggregation of ionic dyes in water, which is sometimes called the "common ion effect" [1]. Aggregation of various classes of dyes is a very well known phenomenon. Generally, the dissolving situation of dyes in water depends mainly on both their aggregation and their ionization. Aggregation and ionization equilibrium of dye molecules in water are described in Fig. 4.

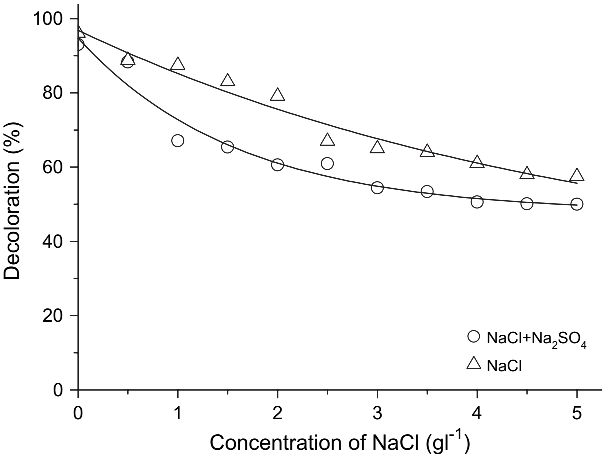


Fig. 5. Effect of mixed salts on decoloration of Reactive Blue MS.

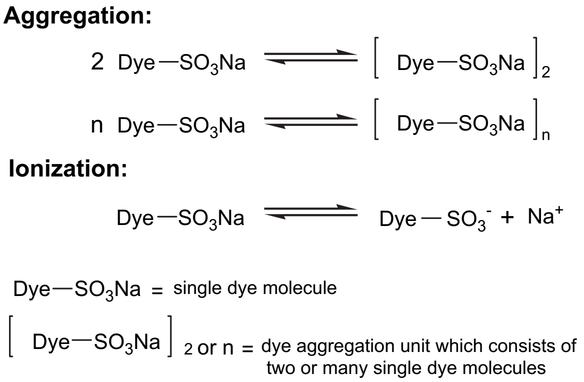


Fig. 4. Schematic model of aggregation and ionization equilibrium of dye molecules in water.

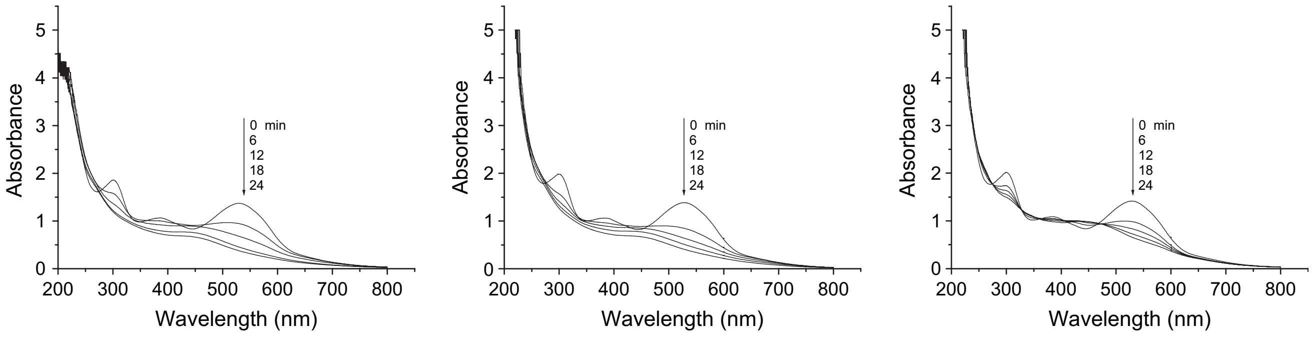
Aggregation equilibrium of dye molecules in water is affected particularly by concentration of dyes and inorganic salts. It have been concluded that in the presence of salt, the π-electron repulsion between two ionic dye molecules is small compared to the affinity of the hydrophobic association. Addition of sodium chloride can reduce the electrostatic repulsion forces between dye anions, which will lead to the increase in aggregation tendency of the dye species [13]. It is believed that aggregation enhanced by inorganic salts can limit significantly solubilization and ionization of dyes, particularly direct dyes in water and lower capability of dye molecules reacted with hydroxyl radicals.

However, increasing concentration of sodium sulfate, in particular, from 4 g l^{-1} to 20 g l^{-1} lead to limited decrease in decoloration of the three dyes in water. This may be due to the balance of two counteracting reasons. It is obvious that the "common ion effect" of salts mentioned above is able to inhabit the removal of dyes. On the other hand, the

Table 1
Decolorization rate constants for the dyes in the presence of inorganic salts

Dyes	Salts	$k \times 10^{-2}$ ($\min^{-1}$)	R	N
Mordant Black PV	NaCl	2.76	0.9658	10
	Na_2SO_4	4.67	0.9861	10
	Control	6.10	0.9912	10
Reactive Blue MS	NaCl	4.48	0.9918	10
	Na_2SO_4	8.68	0.9778	10
	Control	18.17	0.9999	10
Direct Sky Blue 5B	NaCl	1.10	0.9770	10
	Na_2SO_4	3.94	0.9999	10
	Control	4.66	0.9999	10

Concentration of salts in aqueous solution: 60 mmol l^{-1}.



(a) Control (b) Na_2SO_4 (c) NaCl

Fig. 6. The changes in UV–vis spectrum of Mordant Black PV.

reaction between sulfate ions and hydroxyl radical can produce sulphate radicals and peroxydisulphate ions, which show the higher oxidation power [11]. Moreover, it has been stated that the sulphate radical has a unique nature of attacking the dye molecules at various positions resulting in rapid fragmentation of the molecules [14], so as to improve the decoloration efficiency of dyes in aqueous solution.

Sodium nitrate exhibits similar trend in its effect on decoloration to sodium sulfate owing to the balance of 'common ion effect' of salts and nitrate-induced photooxidation of dyes in water [15]. In addition, decoloration values of dyes except Reactive Blue MS decrease slowly and keep steady when concentration of inorganic salts in water is more than 8 g l^{-1}. This implied that excess dosage of salts in dyeing solution could not give rise to strong retardant influence on removing dyes from water in this work.

3.2. Mixed salts

In order to examine the combined influence of different salts on decoloration of azo dyes in water, Reactive Blue MS was selected and decolorized in the presence of a mixture of sodium chloride and sodium sulfate. It is noticed that the total concentration of mixed salts in solution was kept constant at 5 g l^{-1} and the concentration of either sodium chloride or sodium sulfate in solution varied from 0 g l^{-1} to 5 g l^{-1}. For comparison, control experiment in which sodium sulfate was absent in solution was carried out, experimental results of which are presented in Fig. 5.

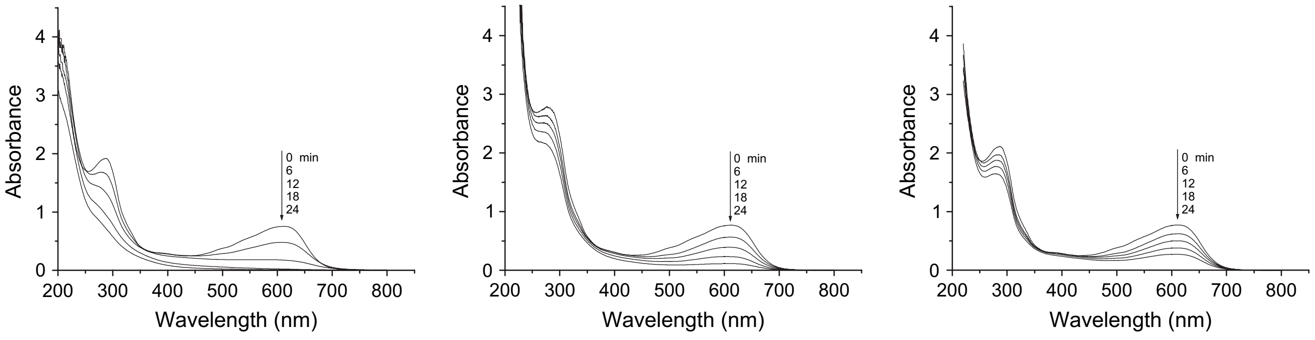
It is obvious from Fig. 5 that decoloration levels of Reactive Blue MS decreased gradually with increasing concentration of sodium chloride from 0 g l^{-1} to 5 g l^{-1} in the presence of mixed salts or only sodium chloride. Also decoloration percentage in the presence of mixed salts is lower than that in the presence of only sodium chloride at the same concentration. This result demonstrates that the combination of sodium chloride with sodium sulfate has a more powerful retardant influence on decoloration of Reactive Blue MS than sodium chloride, especially, when these salts are well proportioned. This may be owing to the enhanced common ion effect of the combined salts.

3.3. Decoloration kinetics

Photocatalytic decoloration of azo dyes in water based on Fe (III)–oxalate complexes/H_2O_2 can be well described by using pseudo-first order kinetics with respect to their concentration:

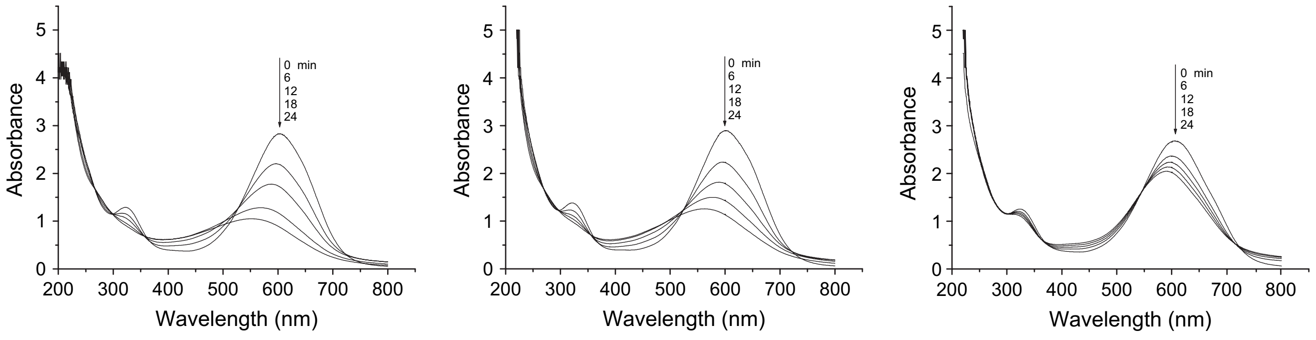
$$\frac{-\mathrm{d}C}{\mathrm{d}t} = kt \qquad (1)$$

where k is the pseudo-first order rate constant, t is the irradiation time, and C is the dye concentration at a given time.



(a) Control (b) Na_2SO_4 (c) NaCl

Fig. 7. The changes in UV–vis spectrum of Reactive Blue MS.



(a) Control (b) Na_2SO_4 (c) NaCl

Fig. 8. The changes in UV–vis spectrum of Direct Sky Blue 5B.

Eq. (1) can be solved by integration between the limits ($t_0 = 0$, C_0) and (t, C) [16]. The integrated expression is:

$$\ln\left(\frac{C_0}{C}\right) = kt \tag{2}$$

The pseudo-first order rate constants of decoloration after 20 min of reaction time were calculated from linear regression $\ln(C_0/C)$ versus time plots with all regression coefficients greater than 0.95, and listed in Table 1.

It can be seen from Table 1 that the pseudo-first order rate constants of decoloration were reduced remarkably in the presence of inorganic salts, particularly, sodium chloride. This agrees well with experimental results given in Fig. 3.

3.4. UV–vis spectrum

The obvious changes of photocatalytic decoloration and degradation of the three dyes using UV–vis absorbance spectra are shown in Figs. 6–8.

In general, the absorbance at 400–800 nm corresponds to the n → π* transition of the azo and hydrazone form, which is due to the color of azo dyes and it is used to monitor the decoloration. The absorbance at 200–400 nm was attributed to the n → π* transition of benzene and naphthalene ring, represents the aromatic content of azo dyes and its decreasing is due to the degradation of aromatic part of the dye [11,17]. The characteristic absorbance of the three dyes is summarized in Table 2.

Figs. 6–8 show that both the absorbance at 200–400 nm and the absorbance at 400–800 nm of the dyes decrease gradually with prolonging exposure time due to increasing decoloration and degradation of the dyes in water. Moreover, additions of salts particularly sodium chloride can enhance absorbance levels strongly at the two bands mentioned above at the same exposure time compared with controls because of the limiting effect of salts on decoloration and degradation. In addition, it is observed clearly from Table 2 that with respect to the absorption ratios of the three dyes at 200–400 and 400–800 nm, sodium chloride is higher than sodium sulfate when exposure time is 12 min or 24 min. This demonstrates that decoloration and degradation of the three dyes are reduced remarkably by sodium chloride. Also, Table 2 indicates that the absorption ratios of Reactive Blue MS at 287 and 611 nm in the presence of sodium sulfate are almost the same as that in the presence of sodium chloride and much

Table 2
The UV–vis characteristic absorbance of the three dyes in the presence of inorganic salts

Exposure time (min)	0		12		24	
Absorption bands (nm)	200–400	400–800	200–400	400–800	200–400	400–800
Black PV	301, 392	529	295, 392	529	295, 392	529[a]
Control	1.00, 1.00	1.00	0.73, 0.86	0.50	0.64, 0.67	0.28[b]
NaCl	1.00, 1.00	1.00	0.81, 0.95	0.59	0.74, 0.90	0.47
Na_2SO_4	1.00, 1.00	1.00	0.71, 0.84	0.46	0.70, 0.70	0.29
Blue MS	287	611	287	611	287	611[a]
Control	1.00	1.00	0.69	0.24	0.38	0.01[b]
NaCl	1.00	1.00	0.88	0.65	0.77	0.13
Na_2SO_4	1.00	1.00	0.86	0.51	0.72	0.14
Sky Blue 5B	323	605	323	605	323	605[a]
Control	1.00	1.00	0.82	0.60	0.71	0.30[b]
NaCl	1.00	1.00	0.94	0.83	0.89	0.74
Na_2SO_4	1.00	1.00	0.80	0.61	0.71	0.38

[a] The characteristic absorption wavelengths of the dyes.

[b] The absorption ratios were calculated by A (residual absorption value at the different exposure time)/A_0 (the initial absorption value of the dyes).

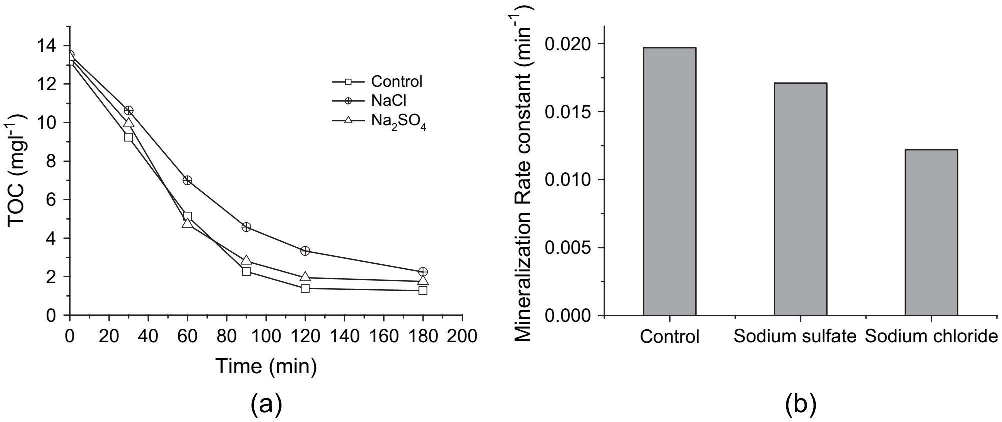


Fig. 9. Influence of inorganic salts on mineralization of Reactive Blue MS.

higher than that in the control experiment; and in contrast, the absorption ratios of Mordant Black PV and Direct Sky Blue 5B at their characteristic absorption wavelengths in the presence of sodium sulfate are almost the same as that in control experiment and much lower than that in the presence of sodium chloride when exposure time is 12 min or 24 min. This implies that sodium sulfate exhibits powerful retardant effect on decoloration and degradation of Reactive Blue MS. On the other hand, it is noted in Fig. 8 and Table 2 that there is a little degradation of Direct Sky Blue 5B in the presence of sodium chloride within 24 min of irradiation because of its higher absorption ratios at 323 nm. These results may be attribute to differences among the three dyes in terms of molecular weight and constitutes such as sulfonic groups.

3.5. Mineralization

The hydroxyl radical produced from photolysis of Fe (III)–oxalate complexes/H_2O_2 system is a powerful oxidant and can ultimately cause mineralization of textile dyes. In this work, mineralization of Reactive Blue MS was conducted in the presence of sodium chloride and sodium sulfate, respectively. For comparison, control experiment in which inorganic salts were absent in solution was carried out, experimental results of which are given in Fig. 9.

Fig. 9 shows the TOC reduction and mineralization rate constant of Reactive Blue MS aqueous solution by photocatalytic oxidation with or without salts. It is found that TOC reduction and mineralization rate constant were effected dramatically by the kind of salt, and sodium chloride presented serious limitation to mineralization compared with sodium sulfate at the same concentration of salts under irradiation, which is very consistent with the decoloration and degradation reduced by sodium chloride mentioned above.

4. Conclusions

Decoloration of the three azo dyes in water by photocatalysis of Fe (III)–oxalate complexes/H_2O_2 was undertaken in the presence of inorganic salts, sodium chloride, sodium sulfate, sodium nitrate and potassium chloride. Photocatalytic decoloration of these dyes was affected dramatically by sodium chloride and potassium chloride due to the hydroxyl radical scavenging effect of chloride ion and enhanced aggregation and limited ionization of dye molecules in water. Moreover, a higher concentration of sodium chloride and potassium chloride can enhance significantly their retardant effect on photocatalytic decoloration of the dyes. In addition, sodium sulfate and sodium nitrate have slight influence on photocatalytic decoloration of the dyes under the same conditions compared with sodium chloride and potassium chloride. This may be a balance of enhanced aggregation of dye molecules caused by inorganic salts and improved photooxidation of dyes induced by sulfate or nitrate. Also, photocatalytic decoloration of azo dyes in water can be described by using pseudo-first order kinetics in the case of inorganic salts, but reaction rate constants were decreased. UV–vis spectrum indicates that the degradation of aromatic parts of dyes was reduced in the presence of inorganic salts, particularly, sodium chloride. Finally, the mineralization of dyes was influenced remarkably by sodium chloride with respect to TOC reduction and mineralization rate constants.

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