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# Spectrophotometric determination of persulfate anion via oxidative depolarization of methyl orange induced by ferrous ions

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ABSTRACT

A rapid, simple, and sensitive spectrophotometric method was developed for the determination of persulfate in this study, based on the oxidation decolorization of methyl orange (MO) with sulfate radicals and hydroxyl radicals generated by the conjunction of persulfate with ferrous ions. The depletion of MO at its maximum absorption wavelength (507 nm) is in proportion to the concentration of persulfate in water. There was a good linear relationship (above 99%) between the depletion of MO and the concentration of persulfate in the ranges of 0.5–100 μmol $L^{-1}$ with a detection limit of 0.17 μmol $L^{-1}$ under the optimized conditions. The proposed MO method for the determination of persulfate in water was effective to tolerate the interferences of common coexisting foreign species in aqueous solutions. The results were as satisfactory as that obtained by the classical iodometric method used in practical water samples. Additionally, the proposed MO method is rather inexpensive to measure the concentration of persulfate by using standard laboratory instruments (spectrophotometer).

*Keywords:* Spectrophotometric determination; Oxidative decolorization; Methyl orange; Persulfate; Ferrous ions

## 1. Introduction

Persulfate is a form of important oxidant currently being used for chemical oxidation of organic pollutants in contaminated groundwater, wastewater, and soils [1–3]. Owing to the strong oxidation capacity [4], persulfate has been widely used to degrade ethanol [5], sulfur dioxide [6], and oximes [7]. What's more, the stronger reactive oxidants (sulfate radical and/or hydroxyl radical) can be generated by the activation of persulfate with transition metals and/or UV [8,9]. The activation of persulfate has been widely used to degrade the refractory organic pollutants of atrazine [10], chlorophenols [11,12], and dyes [13,14] in polluted waters and soils. However, the degradation of the refractory organic pollutants is affected by the concentration of persulfate. Thus, it is necessary to determine the concentration of persulfate during the remediation treatments of polluted waters and soils. A few methods have been developed to determine the concentration of persulfate in water, including

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chemiluminescence method [15] and iodometric method [16].

The chemiluminescence method for the determination of persulfate was developed [15], which was based on the chemiluminescent reaction of luminol with persulfate in alkaline solutions. Although the chemiluminescence method for the determination of persulfate is highly sensitive, the determination of chemiluminescence requires rather specialized kinds of instruments, which are not available in conventional analytical laboratories. The iodometric method for the determination of persulfate was proposed by Wahba and co–workers [16], which is based on the redox reaction of persulfate with KI. Although the iodometric method is sensitive and accurate, it involves back-titration steps after the redox reaction with persulfate, which is rather tedious and time-consuming to perform.

To our knowledge, little is reported for a rapid, simple, and sensitive method for the determination of persulfate in water up to now. Therefore, it is of considerable importance to develop a suitable method to detect the concentration of persulfate in water. Recently, we are interested in oxidation decolorization of dye wastewater with $Fe^{2+}$/persulfate process. In the present work, it was found that methyl orange (MO) can be remarkably decolorized in $Fe^{2+}$/persulfate process, and the decolorization degree of MO at its maximum absorption wavelength (507 nm) is proportion to the concentration of persulfate. In addition, it should be noted that MO has been used as a good indicator for the determination of $CCl_4$ [17], $H_2O_2$ [18], and peroxymonosulfate [19] in water due to its high molar absorption coefficient. Therefore, in this work, a rapid, simple, and sensitive spectrophotometric method was developed to detect the concentration of persulfate in water based on the oxidation decolorization of MO with sulfate radicals and hydroxyl radicals generated by the conjunction of persulfate with ferrous ions.

## 2. Materials and methods

### 2.1. Chemicals and solutions

Potassium persulfate, 4–chlorophenol, 2,4–dichlorophenol, benzoic acid, and oxalic acid were of ACS reagent grade and supplied by Sigma–Aldrich, Inc. Suwannee River natural organic matter (SRNOM) was purchased from the International Humic Substances Society. Methyl orange (MO), methanol, formic acid, sodium acetate, nitrilotriacetic acid (NTA), sodium sulfate, potassium nitrate, potassium chloride, potassium bromate, barium nitrate, manganese sulfate, ferric chloride, ferrous sulfate heptahydrate, perchloric acid, sodium hydroxide, potassium iodide, starch, and sodium thiosulfate were of analytical reagent grade and purchased from Sinopharm Chemical Reagent Co., Ltd. All the chemicals were of analytical reagent grade and used without further purification. Solutions of 1.0 mol $L^{-1}$ $HClO_4$ and 1.0 mol $L^{-1}$ NaOH were used to adjust the pH value of the test solutions. All solutions were prepared with ultrapure water produced from a Milli–Q Biocel ultrapure water system.

### 2.2. Analytical methods

The absorption spectra from 200 to 800 nm and the absorbance at 507 nm were carried out on an UV–vis spectrophotometer (Varian Carry 300) using 1 cm quartz UV cell. PH measurements were carried out with an Ultrabasic 7 pH meter (Denver Instrument). The stock solutions of persulfate were firstly standardized with the iodometric titration method [16] and then prepared as needed. Dissolved organic carbon (DOC) was measured with a Multi N/C 3100 analyzer (Jena, Germany). All measurements were repeated at least three times, and the average values along with one standard deviation (±SD) were provided in this study.

### 2.3. Experimental procedure

The experiments for the oxidation decolorization of MO were performed in 25 mL glass bottles at 25 ± 2˚C. Unless specified elsewhere, the general procedure for the determination of persulfate with the proposed MO method was set as follows: 0.390 mL of MO stock solution (1.283 mmol $L^{-1}$) and 8.91 mL of ultrapure water were added to a 25 mL glass bottle. Then, 1.0 mL of $FeSO_4$ solution was added immediately after the addition of 0.1 mL of sample containing persulfate. After 2.5 min, 3.7 mL of reaction solution was transferred into 1 cm photometric cell to read the absorbance at 507 nm. A blank solution was prepared by using the sample without the addition of persulfate and ferrous ions.

To check the feasibility of the proposed MO method for the determination of persulfate in water practical samples, a lake water sample, a groundwater sample, a river water sample, and a tap water sample were obtained from Mopanshan Lake, Jiangbei groundwater, Songhua river and drinking water supply system in HIT campus, respectively. Their main quality indexes were proposed in our published paper [19]. To compare the proposed MO method with the classical iodometric method reported by Wahba et al. [16], each sample containing 1 mmol $L^{-1}$ persulfate was measured at the same time by both methods. For the determination of persulfate in practical water samples with the iodometric method, 5 mL of sample was added into 30 mL of acetate buffer (pH 3) containing KI. After 45 min, reaction solution was titrated with 5 mmol $L^{-1}$ sodium thiosulfate using starch indicator.

## 3. Results and discussion

### 3.1. *Decolorization of MO in the presence of persulfate activated by ferrous ions*

In this study, the generated sulfate radicals and hydroxy radicals by the activation of persulfate with ferrous ions were used in the decolorization of MO in water. As shown in Fig. 1, the azo dye of MO was obviously decolored in $Fe^{2+}$/persulfate process. Meanwhile, it was observed that the decolorization extent of MO at (the decrease of the absorbance at its maximum absorption wavelength) was proportional to the concentration of persulfate in water. Therefore, the linear correlation between the concentration of persulfate and the decolorization degree of MO at 507 nm allows us to develop a rapid, simple, and sensitive spectrophotometric method for the determination of persulfate in water.

### 3.2. *Effects of operation parameters on the determination of persulfate*

To develop the rapid, simple, and sensitive spectrophotometric method for the determination of persulfate in water, it is of importance to investigate the effects of operation parameters, including reaction time, initial solution pH, initial MO concentration, and initial $Fe^{2+}$ concentration.

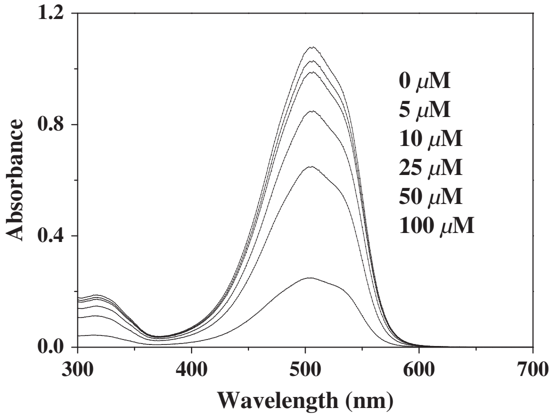

Fig. 1. UV–visible absorption spectra of MO after oxidation decolorization with different concentrations of persulfate in $Fe^{2+}$/persulfate process. Conditions: $[MO]_0 = 25$ μmol $L^{-1}$, $[Fe^{2+}]_0 = 0.2$ mmol $L^{-1}$, $[\text{persulfate}]_0 = 0–100$ μmol $L^{-1}$, $pH_0$ 2.5, reaction time ($t$) = 2.5 min, and 25 ± 2˚C.

#### 3.2.1. *Effect of reaction time*

Fig. 2(a) shows the influence of reaction time on the decolorization extent of MO in $Fe^{2+}$/persulfate process. The decolorization extent of MO at 507 nm is represented by the absorbance change $\Delta A$ ($\Delta A = A_0 - A_t$, where $A_0$ and $A_t$ are the absorbance of the MO solution at 507 nm before and after the reaction). For the given persulfate concentration in water, $\Delta A$ approaches an almost constant value after 2.0 min. The occurrence of the plateau for the $\Delta A$ value is due to either the complete degradation of the added persulfate or the complete decolorization of the added MO at longer reaction time. Therefore, reaction time of $t = 2.5$ min is selected as the optimized operation time for the determination of persulfate in the further experiments.

#### 3.2.2. *Effect of initial solution pH*

Solution pH is an important factor influencing the degradation of MO in $Fe^{2+}$/persulfate process. As shown in Fig. 2(b), the influence of solution pH on the decolorization of MO was investigated. The value of $\Delta A$ reached a maximum at pH 2.5 and decreased as the solution pH increase from 2.5 to 4.0. The decreasing in the value of $\Delta A$ at higher pH values is partly due to the precipitation of $Fe^{3+}$, which is unfavorable to the recycle of $Fe^{2+}/Fe^{3+}$. Thus, pH 2.5 is selected as the optimized initial solution pH for the determination of persulfate in water in further experiments.

#### 3.2.3. *Effect of MO concentration*

Fig. 3(a) shows the influence of initial MO concentration on the determination of persulfate in water. For the given concentration of persulfate, $\Delta A$ increases with the increase of MO concentration initially, and then approaches an almost constant value when initial MO concentration reaches a certain value depended on the concentration of persulfate. At lower initial MO concentration, almost all of the MO molecules will be degraded and the MO solution will be completely decolorized when the persulfate level is high enough. In contrast, at higher initial MO concentration, only a very small fraction of the MO molecules may be degraded and the decolorization degree of MO is very small when the persulfate level is not high enough.

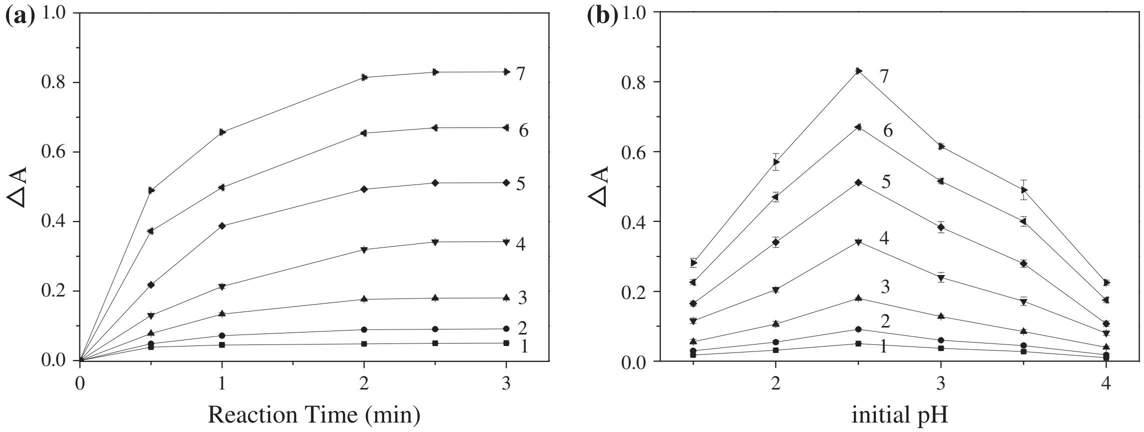

Fig. 2. Effect of reaction time (a) and initial solution pH (b) on the decolorization of MO at 507 nm for initial persulfate concentrations of (1) $5.0 \times 10^{-6}$, (2) $1.0 \times 10^{-5}$, (3) $2.0 \times 10^{-5}$, (4) $4.0 \times 10^{-5}$, (5) $6.0 \times 10^{-5}$, (6) $8.0 \times 10^{-5}$, and (7) $1.0 \times 10^{-4}$ mol $L^{-1}$ in $Fe^{2+}$/persulfate process. Conditions: $[MO]_0 = 25$ μmol $L^{-1}$, $[Fe^{2+}]_0 = 0.2$ mmol $L^{-1}$, $pH_0$ 2.5 or $t = 2.5$ min and 25 ± 2°C.

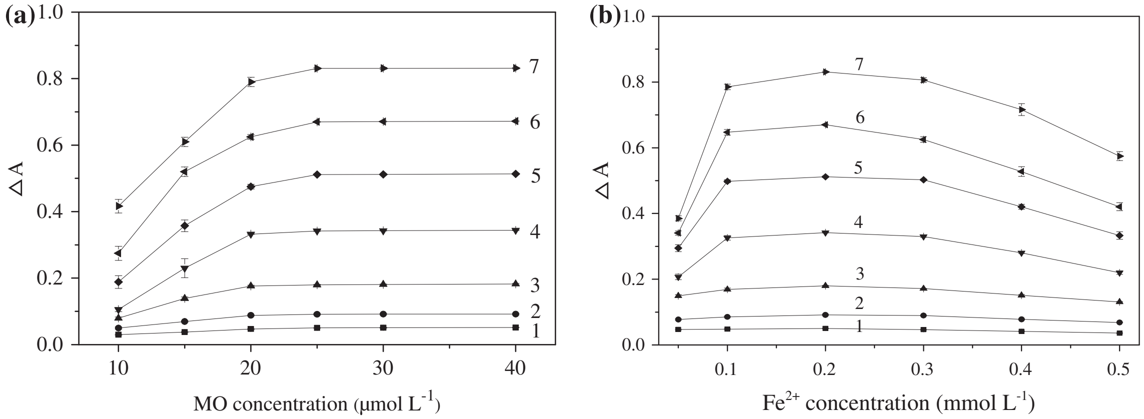

Fig. 3. Effect of initial MO concentration (a) and initial $Fe^{2+}$ concentration (b) on the decolorization of MO at 507 nm for initial persulfate concentrations of (1) $5.0 \times 10^{-6}$, (2) $1.0 \times 10^{-5}$, (3) $2.0 \times 10^{-5}$, (4) $4.0 \times 10^{-5}$, (5) $6.0 \times 10^{-5}$, (6) $8.0 \times 10^{-5}$, and (7) $1.0 \times 10^{-4}$ mol $L^{-1}$ in $Fe^{2+}$/persulfate process. Conditions: $[Fe^{2+}]_0 = 0.2$ mmol $L^{-1}$ or $[MO]_0 = 25$ μmol $L^{-1}$, $pH_0$ 2.5, $t = 2.5$ min, and 25 ± 2°C.

Therefore, initial MO concentration should match with the concentration of persulfate when the proposed MO method is applied for the determination of persulfate in water. Since the concentration of persulfate was no more than 100 μmol $L^{-1}$ in this study, initial MO concentration of 25 μmol $L^{-1}$ was selected as the optimized initial MO concentration for the determination of persulfate in the further experiments.

### 3.2.4. Effect of initial $Fe^{2+}$ concentration

Since the sulfate radicals and hydroxyl radicals were generated by the activation of persulfate with ferrous ions, it is of considerable importance to optimize the initial concentration of $Fe^{2+}$ for the establishment of the analytical method. Fig. 3(b) shows the influence of initial $Fe^{2+}$ concentration on the decolorization degree of MO in $Fe^{2+}$/persulfate process. For each given persulfate concentration, the depletion of MO increases with the increase of initial $Fe^{2+}$ concentration initially, and then approaches an almost constant value when initial $Fe^{2+}$ concentration reaches a certain value which depends on the persulfate concentration. Since persulfate concentration was no more than 100 μmol $L^{-1}$ in this study, the initial $Fe^{2+}$ concentration of 0.2 mmol $L^{-1}$ was selected as the optimized initial $Fe^{2+}$ concentration for the determination of persulfate in our further experiments.

### 3.3. Calibration curves for persulfate determination

The calibration curve for the determination of persulfate in ultrapure water was obtained by optimizing the reaction parameters ($[MO]_0 = 25$ μmol $L^{-1}$, $[Fe^{2+}]_0 = 0.2$ mmol $L^{-1}$, $pH_0$ 2.5, and $t = 2.5$ min) as shown in Fig. 4. A good linear correlation was obtained between persulfate concentration in water and $\Delta A$ under the optimized reaction conditions. The correlation is linear in the ranges of 0.2–100 μmol $L^{-1}$ for the determination of persulfate with the regression equation of $\Delta A = 0.00841 C_{persulfate} + 0.00118$. The correlation coefficient for the determination of persulfate is more than 0.99. Due to the high molar absorption coefficient of MO, our proposed MO spectrophotometric method is highly sensitive for the determination of persulfate in water with the detection limit of as low as 0.17 μmol $L^{-1}$ ($DL = 3\sigma/k = 0.17$ μmol $L^{-1}$, where $\sigma$ is the standard deviation of blank sample and $k$ is the slope of the regression equation). Meanwhile, the effect of stirring rate on the sensitivity of proposed MO spectrophotometric method was checked and found to be negligible at very slow and vigorous mixing condition (data not shown).Additionally, an investigation was carried out to study effect of typical transition metal ions in practical samples on the determination of persulfate. The relative error for the determination of 40 μmol $L^{-1}$ persulfate was no higher than 5% with the existence of $FeSO_4$ (0–75 μmol $L^{-1}$), $FeCl_3$ (0–100 μmol $L^{-1}$), $MnSO_4$ (0–150 μmol $L^{-1}$), and $CuSO_4$ (0–150 μmol $L^{-1}$) (data not shown), which means that our proposed method is well effective to tolerate the interferences of typical transition metal ions in water.

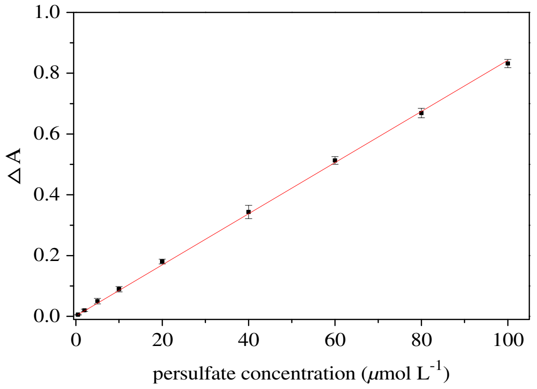

Fig. 4. Standard curve for the determination of persulfate in ultrapure water with the MO method at 507 nm under optimized reaction conditions. Conditions: $[MO]_0 = 25$ $\mu mol^{-1}$ L, $[Fe^{2+}]_0 = 0.2$ $mmol^{-1}$ L, $pH_0$ 2.5, $t = 2.5$ min and 25 ± 2℃.

In addition, the effects of common coexisting foreign species in practical water samples on persulfate determination under the optimized reaction conditions were investigated. The relative error for the determination of 50 μmol $L^{-1}$ persulfate was below 5% at the presence of $Na_2SO_4$ (0–5 mmol $L^{-1}$), $KNO_3$ (0–5 mmol $L^{-1}$), KCl (0–5 mmol $L^{-1}$), $KBrO_3$ (0–5 mmol $L^{-1}$), $Ba(NO_3)_2$ (0–5 mmol $L^{-1}$), $MnSO_4$ (0–0.05 mmol $L^{-1}$), and $FeCl_3$ (0–0.02 mmol $L^{-1}$), methanol (0–0.2 mmol $L^{-1}$), formic acid (0–0.1 mmol $L^{-1}$), sodium acetate (0–0.1 mmol $L^{-1}$), oxalic acid (0–0.01 mmol $L^{-1}$), NTA (0–0.01 mmol $L^{-1}$), 4–chlorophenol (0–0.01 mmol $L^{-1}$), 2,4–dichlorophenol (0–0.01 mmol $L^{-1}$), benzoic acid (0–0.01 mmol $L^{-1}$), SRNOM (0–0.2 mg $L^{-1}$) (data not shown). The results indicate that proposed MO spectrophotometric method is quite effective to tolerate the interferences of common coexisting foreign species in water.

### 3.4. Practical applications

To check the feasibility of our proposed MO spectrophotometric method in practical analysis, persulfate concentration was monitored in four different types of practical samples (A lake water sample, a groundwater sample, a river water sample, and a tap water sample were obtained from Mopanshan Lake, Jiangbei groundwater, Songhua river and drinking water supply system in our campus, respectively). To compare the proposed MO spectrophotometric method with the classical iodometric method reported by Wahba et al. [16], each water sample containing 1 mmol $L^{-1}$ persulfate was measured by both methods. As shown in Table 1, there were no obvious differences between proposed MO spectrophotometric method and the classical iodometric method (a relative error of less than 5% between the two methods). Additionally, the $F$–test with 95% confidence level was also carried out to compare the proposed MO method and the classical iodometric method. As shown in Table 1, the $F$–values of four practical water samples (1.24, 0.87, 0.34, and 0.71 for the lake water sample, the groundwater sample, the river water sample, and the tap water sample, respectively) were much lower than the tabulated critical value ($F_{(5,5)} = 5.05$) at the 95% confidence level. The results indicate that there are no significant differences between the two methods for persulfate determination. Therefore, our proposed MO spectrophotometric method in this study is an accurate and credible method for persulfate determination in practical water samples.

Table 1
Determination of persulfate in practical water samples with our proposed MO spectrophotometric method and the classical iodometric method ($n = 6$)

| | Persulfate concentration (mmol $L^{-1}$) | | | |
|---|---|---|---|---|
| Samples | The MO method | The iodometric method | Relative error (%) | $F$ values in $F$–test[a] |
| Mopanshan lake | 1.064 ± 0.025 | 1.025 ± 0.021 | 3.80 | 1.24 |
| Jiangbei groundwater | 0.965 ± 0.022 | 0.998 ± 0.028 | –3.31 | 0.87 |
| Songhua river | 1.024 ± 0.021 | 1.031 ± 0.036 | –0.68 | 0.34 |
| Drinking water | 1.022 ± 0.024 | 1.036 ± 0.033 | –1.35 | 0.71 |

[a] For comparison, $F_{(5,5)} = 5.05$

## 4. Conclusions

Based on the oxidation decolorization of MO with sulfate radicals and hydroxyl radicals generated by the conjunction of persulfate with ferrous ions, a rapid, simple, and sensitive spectrophotometric method for the determination of persulfate in water was proposed in this study. Under the optimized conditions ($[MO]_0 = 25$ μmol $L^{-1}$, $[Fe^{2+}]_0 = 0.2$ mmol $L^{-1}$, $pH_0$ 2.5, and $t = 2.5$ min), a good linear relationship is found between the depletion of MO and the concentration of persulfate in the ranges of 0.5−100 μmol $L^{-1}$. The main advantages and features of the proposed MO spectrophotometric method for the determination of persulfate are as follows:

1. The oxidation degradation of MO with sulfate radicals and hydroxyl radicals generated by the conjunction of persulfate with ferrous ions is rather fast. Only 2.5 min is enough for the determination of persulfate in water with our proposed MO method. The proposed MO method is highly sensitive for the determination of persulfate in water due to the high molar absorption coefficient at 507 nm of MO.
2. The proposed MO method is rather simple and does not require any difficult or time-consuming operations. The proposed MO method is rather inexpensive to detect the concentration of persulfate in water using standard laboratory instruments (spectrophotometer). The proposed MO method is accurate enough for the determination of persulfate in water because the results are as satisfactory as that obtained by the classical iodometric method.

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