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Real-world diesel vehicle emission factors for China





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Rapid and simple spectrophotometric determination of persulfate in water by microwave assisted decolorization of **Methylene Blue**

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ABSTRACT

A rapid and simple method for determination of persulfate in aqueous solution was developed. The method is based on the rapid reaction of persulfate with Methylene Blue (MB) via domestic microwave activation, which can promote the activation of persulfate and decolorize MB quickly. The depletion of MB at 644 nm (the maximum absorption wavelength of MB) is in proportion to the increasing concentration of persulfate in aqueous solution. Linear calibration curve was obtained in the range 0-1.5 mmol/L, with a limit of detection of 0.0028 mmol/L. The reaction time is rapid (within 60 sec), which is much shorter than that used for conventional methods. Compared with existing analytical methods, it need not any additives, especially colorful Fe²⁺, and need not any pretreatment for samples, such as pH adjustment.

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Introduction

In recent years, persulfate has been attracted increasing attention as a stable and powerful oxidant. Persulfate is always applied for degradation of organic contaminants in groundwater and wastewater (Fang and Shang, 2012; Yuan et al., 2013; Ji et al., 2014). Though persulfate has a redox potential of E^0 = 2.01 V (Amin, 1981), activation is necessary for application because of its low reactivity (Yang et al., 2009). A more powerful sulfate radical (SO₄⁻) with $E^0 = 2.6$ V can be produced by activation via thermal method (Huang et al., 2002), UV (Hori et al., 2005), metal ions (especially Fe²⁺) (Liang et al., 2004) and activated carbon (Zhang et al., 2013). Microwave (MW) (Yang et al., 2009) is also an attractive thermal method to activate

persulfate. Yang et al. (2009) discovered that, with MW process, persulfate can oxidize azo dye Acid Orange 7, and the decolorization reached to 100% in 5 min, compared with a few hours without activation.

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Despite that persulfate has a versatile application in many aspects, there are only several methods for determination of it. Wahba et al. (1959) studied the stoichiometric relationship between the redox reaction, then reported the reductometric titration method. Amin (1981) proposed the polarography method which was based on the various changes in current polarography with different concentrations of persulfate. Until lately, Liang et al. (2008) investigated the relationship between the concentrations of persulfate and the absorbance changes of the solution, and developed the

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spectrophotometric method. Among these methods, the most common ones were based on Eq. (1).

$$S_2O_8^{2-} + 2I^- \rightarrow 2SO_4^{2-} + I_2$$
 (1)

The traditional titration method, which was simple and accurate, involved a back-titration by thiosulfate. Together with the slow rate between persulfate and iodide, the reaction time was lasting over 40 min (Huang et al., 2002). Liang et al. (2008) created the rapid spectrophotometric method and reduced the reaction time to 15 min via Fe²⁺ activation. But the process needs pretreatment of sample, such as adjustment of the pH in solution, and needs adding colorful Fe²⁺ into the system, which may disturb the spectrophotometric determination. On the other hand, these determination methods of persulfate always focus on the concentration larger than 1 mmol/L (Wahba et al., 1959; Liang et al., 2008). Considering the numerous applications of persulfate, it is very necessary to develop a sensitive method for determining the lower concentration of persulfate in aqueous solution.

Dyes, which can be decolorized by strong oxidants, were used as indicators (Jurado et al., 2006; Dhaouadi et al., 2006). Zou et al. (2014) reported a spectrophotometric method of peroxymonosulfate by measuring the decolorization extent of Methylene Orange via SO_4^- (Eq. (2)), which was produced by Co²⁺-activated peroxymonosulfate. Ding et al. (2011) proposed a new method which was used to determine the concentration of persulfate by means of decolorization extent of four dyes. In case of the slow reaction rate of persulfate, Ding et al. (2011) combined normal heating with Fe²⁺ activation methods for acceleration. As a result, it can be completed in a few minutes. However, it may lead some obstructions to the scale application. First, as the traditional method, it needs to adjust the pH of sample. What's more, both thermal activation and metal iron activation were used, which made the system complicated and tedious.

$$Dye + SO_4^{-\bullet} \rightarrow Decolorized \ dye + SO_4^{2-}$$
(2)

In this article, we developed a new method to determine persulfate in aqueous solution by measuring the decolorization extent of Methylene Blue (MB) via microwave (MW) process. It was found that a method with more rapid, convenient, and simple procedures was achieved. Under the optimized conditions, a good linear correlation was shown between the decolorization extent and the persulfate concentration. To check the feasibility and accuracy of the proposed method, we also make a comparison between our proposed method and the classic iodometric spectrophotometric methods.

1. Materials and methods

1.1. Reagents and solutions

All the used chemicals were of analytical reagent grade. Sodium persulfate, potassium iodide, sodium hydroxide and hydrochloric acid were purchased from Shanghai Chemical Reagent Company, China. MB was purchased from Tianjin Kermel Chemical Reagent Company, China. Solutions of 1.0 mol/L H₂SO₄ and 1.0 mol/L NaOH were used to adjust pH. All experiments were carried out using double distilled water.

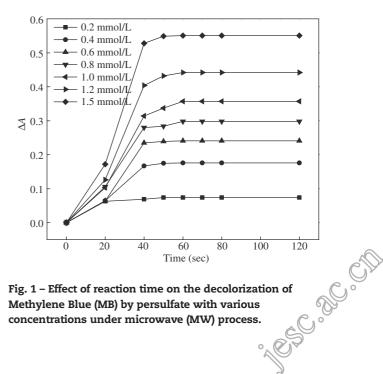
1.2. Apparatus and measurement procedure

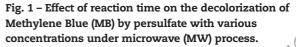
The decolorization experiments were conducted in a series of polypropylene ethylene plastic pipes (10.0 mL) with a total reaction solution volume of 5.0 mL. Sample solutions of persulfate (1.0 mL) and MB (4.0 mL) were mixed. The mixture solution was placed in an oven for a specific time as required. Unless otherwise specified, the initial concentration of MB was 10 mg/L, and the reaction time was 60 sec. MW radiation was provided by a domestic MW oven (900 W). After this process, the pipes were quickly transferred into a water bath for cooling down to room temperature (20 \pm 1°C). It was worth to mention that, during such a short period of time, the loss of the solution caused by volatilization was negligible. The UVvis absorbance of samples was analyzed with a Shimadzu UV1206 spectrophotometer (Shimadzu Corporation, Kyoto, Japan). The degradation of MB was monitored by measuring the maximum absorbance at λ = 664 nm. The decolorization extent was characterized by ΔA , $\Delta A = A_0 - A_t$, where A_0 and A_t were the absorbance of the dye solutions before reaction and after MW process of time t. The changes in pH of the mixed solution of persulfate and MB during the MW process were also investigated. $\triangle pH$ was the difference in pH before and after MW process. To ensure the accuracy and replicability of the collected data, the experiments were operated at least three times under identical conditions.

2. Results and discussion

2.1. Effect of reaction time

MB, one of the basic dyes, was used as the indicator for the determination of persulfate. MW activation, rather than Fe²⁺ activation, was used to promote the reaction. The influence of MW reaction time on the decolorization extent of MB was investigated and the results are shown in Fig. 1. It shows that for various concentrations of persulfate, all the reactions took





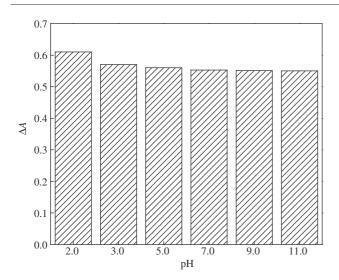


Fig. 2 – Effect of initial pH value on the decolorization of Methylene Blue (MB) by persulfate under microwave (MW) process.

place rapidly particularly in the first 40 sec and reached to the end over 60 sec. The appearance of the plateau was due to the total consumption of added persulfate. This proposed method was more rapid compared with the process of non-activation (40 min) (Huang et al., 2002), the process with Fe²⁺ activation (15 min) (Liang et al., 2008), and the process with normal thermal and Fe^{2+} activation (2–7 min) (Ding et al., 2011).

2.2. Effect of initial pH

As all Fenton-like reactions, pH value is a key parameter for persulfate to react with target substance. In most cases (Do et al., 2013; Jo et al., 2014), it is strict for pH condition in a specific reaction, which makes process complicated and the added acid or alkali may introduce errors. Fig. 2 shows different ΔA against various initial pH values of samples. ΔA was measured as a variable of decolorization between 1.5 mmol/L persulfate sample solution and the control sample without persulfate.

It was obvious that there was no significant change with different initial pH values. In other words, the initial pH had little influence on the decolorization extent. In the previous studies (Liang et al., 2008; Ding et al., 2011), Fe^{2+} was added into the system in order to achieve end points in a shorter time. As a significant feature of Fenton's reaction, pH is a critical parameter of the system. It is very exciting to find that, in our present experiment, the determination process needs no pH adjustment.

2.3. Effect of initial MB concentration

Fig. 3 shows a series of UV-vis absorption spectra corresponding to various initial MB concentrations. It can be clearly seen

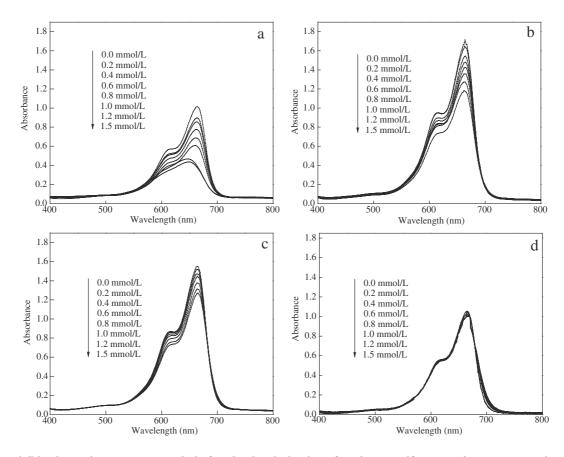


Fig. 3 – UV visible absorption spectra recorded after the decolorization of MB by persulfate at various concentrations, (a) and (b) not diluted, (c) diluted four times, (d) diluted ten times. The initial concentration of MB was (a) 5 mg/L, (b) 10 mg/L, (c) 25 mg/L and (d) 50 mg/L.

Table 1 – Decreases in pH values and peak shifts with various concentrations of Methylene Blue (MB).			
Concentration of MB (mg/L)	∆рН	Blue shift of the peak (nm)	
5	4.5	16	
10	4.1	Negligible	
25	4.1	Negligible	
50	4.0	Negligible	

that with increasing initial MB concentration, gaps between adjacent curves became smaller. When the initial concentration reached up to 25 mg/L, the gaps were too small to be recognized. Moreover, there was no certain relationship between gaps and the various concentrations of persulfate. As the initial concentration increased to 50 mg/L, the curves overlapped a lot and became difficult to be identified. However, the initial MB concentration did not seem the lower the better. When the initial MB concentration was 5 mg/L, the maximum absorbance appeared a blue shift. The data from Table 1 showed the decreases in pH value after reaction with various MB concentrations. The pH variation may be caused by the hydrolysis of SO_4^{-} (Eq. (3)) (Waldemer et al., 2007) and the generated intermediate carboxylic acid (such as formic or oxalic) (Hisaindee et al., 2013). And in strong acid condition, when the concentration of MB was below 5 mg/L, the peak of the maximum absorbance may emerge a blue shift. In addition, with the degradation process of MB, part of the structure may be interrupted. As shown in Fig. 3a, the character peaks of MB generated blue shifts and even disappeared. Therefore, the optimal initial concentration of MB was 10 mg/L.

$$SO_4^{-\bullet} + H_2O \to HO^{\bullet} + SO_4^{2-} + H^+$$
 (3)

2.4. Validation of method

0.6

0.5

The linearity of the proposed method was investigated by recording the ΔA responses to standard persulfate solutions in

0.4 y = 0.00728 + 0.36469x $R^2 = 0.997$ ΔA 0.3 0.2 0.1 0.0 0.20.40.6 0.8 1.01.2 1.4 1.6 Concentration of persulfate (mmol/L)

Fig. 4 – Calibration curve for the determination of persulfate with the MB-MW based spectrophotometric method under optimized reaction conditions.

Table 2 – Recovery of persulfate determined by analyzing three sets of standard samples.		
Concentration	Measured	Recovery
of comple	concontration ^a	(0/)

of sample (mmol/L)	concentration ^a (mmol/L)	(%)	
0.1	0.093 ± 0.002	93.26	
0.3	0.270 ± 0.04	89.75	
0.7	0.719 ± 0.03	103.04	
1.0	0.967 ± 0.012	96.70	
1.2	1.161 ± 0.03	95.92	
1.3	1.291 ± 0.012	99.31	
1.5	1.488 ± 0.001	99.27	
a Data are presented	^a Data are presented as mean , standard deviation		

^a Data are presented as mean ± standard deviation.

the range of 0–1.5 mmol/L. A calibration curve was obtained by adding 1.0 mL persulfate sample into 4.0 mL 10 mg/L MB, then experiencing 60 sec MW process for the mixture system. As shown in Fig. 4, the correlation was linear between ΔA and concentration of persulfate. The remaining MB and the persulfate value also presented a good linear relationship. What's more, correlation coefficient between remaining MB and concentration of persulfate was 0.997, same as that of the calibration curve. It is noticed that the concentration of persulfate shown in Fig. 4 was the original concentration of persulfate sample, not the concentration in the mixed solution.

Since spectrophotometer sensitivity can be accurate to 0.001, the limit of detection (LOD) of the proposed method obtained was 0.0028 mmol/L. Recoveries were measured through determining three sets of standard samples within a range of 0–1.5 mmol/L (Table 2). Good recovery values from 89.75% to 103.04%, a low method detection limit (MDL) of 0.0016 mmol/L, and a relative standard deviation (RSD) of 3.71% based on seven determinations were obtained.

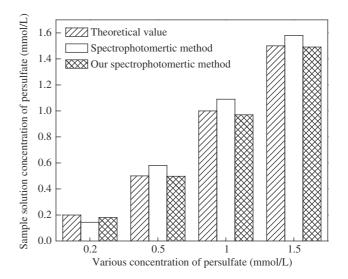


Fig. 5 – Comparisons of persulfate concentrations in the measured sample solutions for the theoretical value, the spectrophotometric method and MB-MW based spectrophotometric method.

2.5. Monitoring aqueous samples using the proposed method

To check the feasibility of the proposed method in practical analysis, the developed method was used to monitor the samples, which were simulated by various known concentrations of persulfate. We made a comparison among the theoretical value, the spectrophotometric method based on iodometric method (Liang et al., 2008) and our method. The results shown in Fig. 5 confirmed that the persulfate concentrations of the proposed method were closer to the theoretical values than those of the iodometric based spectrophotometric method. The average error of the proposed method was 3.43%, while the average error of the spectrophotometric method was 14.58%.

3. Conclusions

The proposed MB-MW based spectrophotometric method for determination of persulfate in water is very rapid, simple and convenient in comparison with other methods. This method process demands no pretreatment of sample, no strict conditions and no valuable instruments. The analytical results reveal its potential use for the detection of low persulfate concentration.

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