

Application of full factorial design for methylene blue dye removal using heat-activated persulfate oxidation



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ABSTRACT

In this study, a thermally activated persulfate oxidation process was investigated to treat aqueous methylene blue (MB) dye. Effects of temperature (55–80 °C), initial pH (2.3–9.3), and potassium persulfate concentration (500–2000 mg/L) on MB color removal were examined. Results showed that acidic and neutral pH values were more favorable for MB oxidation than basic pH values. Based on experimental results, increasing the temperature from 55 to 80 (°C), and oxidant dose from 500 to 2000 (mg/L) enhanced the color removal efficiency (CR%) from 36% to 99.5% and from 55% to 98%, respectively. Kinetic study indicated that MB dye removal was a pseudo first-order reaction with respect to the various concentrations of MB and persulfate. Activation energy for the hot persulfate oxidation of MB was obtained as 112.3 (kJ/mol). To analyze the experimental data, an experimental design was applied by considering four operating parameters, including initial dye concentration, oxidant dosage, temperature and reaction time as input variables. A model was proposed for the statistical analysis of color removal prediction. Model predictions were found to be in good agreement ($R^2 = 0.96$, adjusted $R^2 = 95.6\%$) with experimental data.

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1. Introduction

Certain amounts of aromatic components are wasted during a process which contains a wide range of non-biodegradable contaminants that cause environmental problems [1]. One of the substances with this feature which is considerably applicable in various industries is MB. It has widespread applications such as medical, cosmetic usage and textile industrial. However, it has a cariogenic effect on humans and animals [2,3]. In addition, its chloride salt is listed in the Canadian Workplace Hazardous Materials Information System (WHMIS) ingredient disclosure list [2].

Different technological processes such as chemical methods (ozonation, electrocoagulation [4], photo-degradation [5], electrochemical reduction, and oxidation), physical adsorption [3,6] and biodegradation [7,8] have been developed for dye removal.

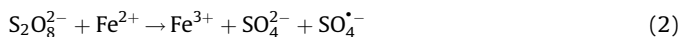
Advanced oxidation processes (AOPs) based on radical generation are alternative techniques for the destruction of aromatic organics in wastewater and effluents. AOPs commonly involve Fenton reagent, UV/TiO₂, UV/H₂O₂, O₃/H₂O₂, or a combination of them. The generated radicals in the AOPs are a strong oxidant with the capability of complete oxidation of most organic compounds into carbon dioxide, water, and mineral acids or salts [9,10]. Another AOP technique, which is widely used, is persulfate oxidation. Persulfate is the newest oxidant source used for in situ chemical oxidation [11]. Relative stability of persulfate in the subsurface, strong oxidation potential, widespread reactivity, high aqueous solubility, fairly low cost, and benign end products make persulfate oxidation a promising choice among AOPs for treating aromatic organic compounds [12,13]. However, this technique suffers from several drawbacks such as needing activation, increasing salt content in the effluent and slow reaction in the peroxydisulfate anion state [12,14].

Persulfate with great oxidation potential ($E^0 = 2.05$ V) is generally activated under heat, photo or metal-catalyzed conditions. High reactive species, sulfate radicals ($\text{SO}_4^{\cdot-}$; $E^0 = 2.5$ – 3.1 V) and hydroxyl radicals (HO^{\cdot} ; $E^0 = 2.7$ V) are generated from the decomposition of persulfate ions in the aqueous phase under

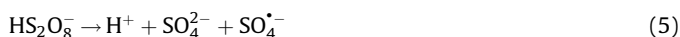
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thermal or photolysis conditions [14,15]. These radicals are generated in water through the following reaction (Eqs. (1)–(3)).



Under acidic conditions, breakdown of persulfate into sulfate free radicals can be further acid catalyzed through Eqs. (4) and (5) [16]. Sulfate anion radical reacts with hydroxide anion, or water, and forms hydroxyl radical according to Eqs. (6) and (7) [16,17].



The previous study, conducted by Ghauch *et al.* on heat-activated persulfate oxidation [18] introduced this method as a viable option for complete dye removal and mineralization potentials. Several studies have shown that different factors like initial persulfate, pollutant concentration, pH, and temperature have an influence on persulfate decomposition rate [19,20]. Since the effect of a parameter may not be similar in different processes, considering the interaction among other operational conditions and the pollutants' molecular structure is essential. To have a precise study on the effect of each operating parameter and its interaction on depended variable(s), statistical technique can be helpful [21,22].

Factorial design is a statistical technique that is practical in planning experiments where several factors are controlled and their effects on each other are investigated at two or more levels [23]. General full factorial design is appropriate where dealing with multiple levels of several factors [24]. The capability of General full factorial design in the modeling of complex systems makes it a more practical method, than other traditional techniques for modeling a multivariable system, which usually define one factor at a time [25].

In the present study, decolorization of MB as an environmental pollutant was conducted by heat-activated persulfate oxidation. Systematic investigations that indicate the behavior of the process, main variable, interaction effects, and their relative importance, have been rarely studied. General full factorial design was applied to examine factors affecting treatment efficiency, including process temperature (T), initial MB concentration (C_{MB}), reaction time (t), and persulfate oxidant dosage (C_{ox}). Activation energy and kinetic parameter were calculated from the experimental data. Finally, a model was proposed for the performance of CR%.

Table 1
Characteristics of methylene blue dye.

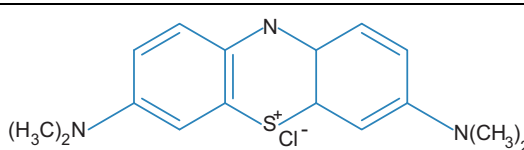
Chemical class	Chemical structure	Molecular formula	C.I. number	λ_{max} (nm)	M_w
Thiazine dye		$\text{C}_{16}\text{H}_{18}\text{N}_3\text{SCl}$	52,015	668	319.8

Table 2
Characteristics of real water.

Characteristics	Value
Calcium hardness (mg/L CaCO_3)	101
Magnesium hardness (mg/L CaCO_3)	6
TDS (mg/L)	140
pH	7.3

2. Materials and methods

2.1. Chemicals and reagents

The dye, MB (C.I. Number = 52,015) (Table 1), ammonium persulfate (APS) and potassium persulfate (KPS) were provided in analytical grade (Merck Co., Germany). KPS was used as a main oxidant in all experimental runs, and APS form was used just for comparing of the two oxidants capability in dye removal. Sodium hydroxide and hydrochloric acid were used to adjust pH (Merck Co., Germany). All solutions were prepared in real water used in the finishing and textile factories in our region (Tabriz, Iran). The characteristics of the real water are presented in Table 2.

2.2. Analysis methods

In order to measure the dye concentration during the experiment, at first, wavelength of maximum absorbance in the range of visible light was determined by UV-Vis spectrophotometer (Model 6715, JENWAY Co. England). Then, calibration curve was obtained using the solution with the known concentration of the dye that showed a good fitness between absorbance and dye concentration ($R^2 = 1$). Decolorization efficiency was calculated by Eq. (8).

$$\% \text{Decolorization} = \left(1 - \frac{[\text{MB}]_t}{[\text{MB}]_0} \right) \times 100 \quad (8)$$

where $[\text{MB}]_0$ and $[\text{MB}]_t$ represent mg/L concentration of MB at times 0 and t , respectively. The pH was measured using a pH meter (Mettler-Toledo, Switzerland).

2.3. Experimental procedures

The reaction of persulfate oxidation of MB was carried out in a 250 mL Erlenmeyer flask as completely mixed batch reactor systems. The initial pH of the MB solution was adjusted with concentrated NaOH (10 wt.%) and HCl (1.0 M) solutions to the desired value before starting the experiment. A magnetic stirrer equipped with an electronic contact thermometer (Fanavaran Sahand Azar, HMS300) was used to ensure constant temperature and perfect mixing at a stable rate during all experiments. In order to control the conformity of solution temperature, another thermometer was fixed in the solution. To determine the dye removal efficiency of persulfate oxidation process, samples were withdrawn at the specific times, and immediately were analyzed.

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