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Efficient removal of methyl orange by electrogenerated ferrate(VI)

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Abstract. The degradation of methyl orange (MO) by ferrate(VI) was examined to demonstrate the potential of this oxidant for the treatment of azo dye in water. The effects of solution pH, ferrate(VI) dosages, treatment time, temperature, and constituents were studied. The results suggested that the suitable conditions for MO removal were pH 3, the molar ratio of Fe(VI) to MO greater than 5.1:1.0, temperature range of 20 to 50 °C. The oxidation reaction MO by Fe(VI) occurred primarily within the first 3 minutes. The MO removal achieved 94.9 and 99.3 % after 3 minutes at ferrate(VI) dosages of 5.1:1.0 and 8.5:1.0, respectively. Anions (Cl^- and NO_3^-) and cations (Mg^{2+} and Fe^{3+}) had no influence on the MO removal. However, cation Cu^{2+} in water reduced the MO removal efficiency by ferrate(VI). The MO destruction was determined by UV-vis spectra and LC-MS chromatograms of MO solutions before and after adding ferrate(VI).

Keywords: methyl orange, ferrate(VI), azo dye, oxidation, electrosynthesis.

Classification numbers: 3.7.3, 3.4.2, 3.2.1.

1. INTRODUCTION

The textile, printing, and dyeing industries consume a huge amount of water and also discharge large amounts of wastewater, accounting for 70 % of global wastewater [1, 2]. Effluent containing dyes is a serious pollutant of the environment and affects human health and aquatic biota due to their toxic, nonbiodegradable, and carcinogenic characteristics [3, 4]. Globally, around 10,000 tons of synthetic dyes are used in textile industries each year, and azo dyes are the most extensively used dyes in the textile industry, estimating more than 60 %. Ineffective textile dyeing processes release 15-50 % of the azo dyes that are not bonded to fibers and textiles into the produced wastewater [5]. Methyl orange (MO), an azo dye with a stable azo functional group (-N=N-) and two aromatic rings, has high chromaticity, toxicity, carcinogenic

and mutagenic effects, and low biodegradability. It poses major dangers and has harmful impacts on living organisms [6, 7]. As a result, the MO removal from industrial effluent before releasing it into the environment has become an important problem. A variety of treatment approaches have been used for the decolorization of MO dye, including adsorption, flocculation, electro-coagulation, membrane filtration, advanced oxidation processes (AOPs), and biological treatments [7-10]. Among these, the chemical oxidation process is always regarded as an essential and significant process in water and wastewater treatment, even when other treatments have been proven to be somewhat effective. Many chemical oxidants, such as ozone, chlorine dioxide, chloramines, and permanganate, have been studied for dye degradation in wastewater. However, these oxidants can produce a variety of byproducts, some of which have been shown to be toxic to aquatic creatures and people [11-13].

Ferrate(VI), a compound of iron with the oxidation state +6, has reduction potentials of 2.20 V in acidic and 0.72 V in alkaline media. Ferrate is known as a multifunctional compound, including an oxidant, a disinfectant, and a coagulant [14]. Thus, it has been considered for a number of applications, especially in water and wastewater treatment [15, 16]. During the oxidation process of pollutants by ferrate(VI), Fe(III) ion or ferric hydroxide produced can act as a coagulant, which removes microorganisms and inorganic pollutants such as arsenic(V), cadmium(II), and copper(II) from water [17-19]. Therefore, ferrate(VI), an environmentally friendly chemical, is one of the most promising options for water and wastewater treatment.

Various approaches have been used to synthesize the ferrate(VI), including wet chemical, thermal, and electrochemical methods [14, 20-22]. The electrochemical method is a simple, low-cost, and green process; in addition, it can generate ferrate online and be applied directly on-site for water and wastewater treatment [23, 24].

In the electrochemical synthesis, Fe(VI) was commonly generated by anodic dissolution of iron or iron alloys in strongly alkaline solvents. Many factors such as electrolysis temperature, electrolyte type and concentrations, current density, electrolysis time, and anode materials affecting ferrate synthesis efficiency have been studied [25, 26]. Our previous research used mild steel, grey cast iron, and ductile iron, three low-cost and widely available anode materials, to synthesize ferrate(VI). The results revealed that ductile iron with high silicon content and high carbon content in the graphite form of spheres was the optimum anode material for the electrogeneration of ferrate (VI). The optimal conditions for ferrate(VI) electrogeneration using ductile iron anode were a current density of 40 mA/cm^2 in 14 M NaOH solution, and electrolysis temperature of 30 – 40 °C [27]. The passive layer formed on the ductile iron anode after different electrolysis times affected ferrate electrosynthesis. With the electrolysis time greater than 6 h, the thicker passive layer consisting of a mixture of iron oxides (Fe_2O_3 and Fe_3O_4) decreased considerably ferrate generation [28]. In this paper, we investigate the degradation of MO by ferrate(VI) synthesized by the electrochemical method and identify the suitable conditions for MO removal, including pH, the molar ratios of Fe(VI) to MO, temperature, and treatment time. The influence of constituents on MO removal by ferrate(VI) was also studied. Furthermore, the MO destruction mechanism was determined by UV-vis spectra and LC-MS chromatograms of MO solutions before and after adding ferrate(VI).

2. MATERIALS AND METHODS

2.1. Materials

All chemicals used in this study were purchased from Sigma-Aldrich. Ductile iron with a weight composition of 93.70 % Fe, 3.65 % C, 2.33 % Si, 0.01 % P, 0.01 % S, and 0.30 % Mn from Hanoi Mechanical Limited Company (Viet Nam) was used as a sacrificial anode material for ferrate electrochemical synthesis.

2.2. Electrosynthesis of ferrate(VI)

Ferrate(VI) was synthesized by galvanostatic polarization at a current density of 40 mA/cm^2 in a three-electrode cell on the electrochemical workstation system Zahner IM6 (Zahner-Elektrik, Germany). A three-electrode cell contained the ductile iron electrode as the working electrode, the titanium electrode as the counter electrode, and the Ag/AgCl electrode (saturated KCl solution) as the reference electrode. Details for the ferrate synthesis conditions were given in our previous study [27, 28]. The prepared ferrate concentration was measured by UV-vis method using Hach DR6000 UV-vis spectrophotometer (USA).

2.3. Removal of MO by obtained ferrate

The degradation of MO was conducted by adding generated ferrate(VI) solution into MO solution, and the pH of the mixed solutions was adjusted from 1 to 9 by H_2SO_4 and NaOH solutions. The initial MO concentration was 50 mg/L at a pH of 7, and the ferrate(VI) to MO molar ratio varied from 1.7:1.0 to 8.5:1.0. The mixed solution was stirred for 3 minutes, and after that, the remaining MO was quantified by UV-vis measurements on Hach DR6000 UV-vis spectrophotometer (USA) at wavelengths of 509 nm. The calibration curves obtained through experiments utilizing standard solutions were used to determine the concentrations of MO. The MO removal efficiency (R(%)) was calculated following Eq. (1).

$$R = \frac{C_0 - C_t}{C_0} x100 \quad (1)$$

where C_0 is the initial MO concentration (mg/L) and C_t is the residual MO concentration (mg/L) after a time (t) of MO treatment.

To investigate the effect of temperature on MO removal, the experiments were carried out by varying the temperature from 10 to 50 °C. The influence of environmental factors on MO removal was studied by mixing initial MO solutions with individual inorganic anions (Cl^- and NO_3^-) by adding NaCl and $NaNO_3$, and cations (Cu^{2+}, Fe^{3+}, and Mg^{2+}) by adding $CuSO_4$, $Fe_2(SO_4)_3$ and $MgSO_4$.

Hach DR6000 UV-vis spectrophotometer (USA) and Liquid chromatography-mass spectrometry (LC-MS) (X500R QTOF, SCIEX, USA) were used to analyze MO solution before and after treatment with ferrate(VI).

MO was also analyzed for mineralization based on total organic carbon (TOC) and chemical oxygen demand (COD) removal (%). The TOC analysis of MO solutions before and after 60 minutes of treatment with ferrate was done using a TOC analyzer (TOC-VCPN, Shimadzu, Japan). The TOC removal efficiency (%) was calculated following Eq. (2).

$$TOC\ removal = \frac{TOC_b - TOC_a}{TOC_b} \quad (2)$$

where TOC_b and TOC_a are the total organic carbon concentrations (mg/L) of MO before and after treatment with ferrate, respectively.

The COD of MO solution before and after treatment with ferrate was analyzed by colorimetric method. The COD removal efficiency (%) was calculated following Eq. (3).

$$COD\ removal = \frac{COD_b - COD_a}{TCOD_b} \quad (3)$$

where COD_b and COD_a are the chemical oxygen demand concentrations (mg/L) of MO before and after treatment with ferrate, respectively.

3. RESULTS AND DISCUSSION

3.1. Effect of solution pH on MO degradation by ferrate(VI)

The pH of the mixed solution is an important factor in MO degradation by ferrate(VI). The MO has a pKa of 3.46 and has two chemical structures depending on pH. In acidic media with a pH below 3.4, the MO has quinoid structure with a corresponding red color. When pH value is greater than 3.4, the MO has azo structure with a corresponding yellow color. The effects of pH on MO removal were conducted in the pH range 1-9. The results given in Figure 1 show that the removal of MO increases with increasing pH from 1 to 3, after which it reduces with increasing pH from 3 to 9 at the ferrate(VI) to MO molar ratio of 3.4:1.0. The suitable condition of pH was 3, and the removal efficiency of MO was 92 %. This result can be explained by the oxidizing power of the active species found in ferrate(VI) solution. The forms that exist in aqueous solution of ferrate(VI) solution are $H_3FeO_4^+$, H_2FeO_4, $HFeO_4^-$, and FeO_4^{2-}, depending on pH, following Eqs. (4)-(6) [29].

$$H_3FeO_4^+ \leftrightarrow H^+ + H_2\,FeO_4 \qquad pK_1 = 1.6 \quad (4)$$

$$H_2FeO_4 \leftrightarrow H^+ + HFeO_4^- \qquad pK_2 = 3.5 \quad (5)$$

$$HFeO_4^- \leftrightarrow H^+ + FeO_4^{2-} \qquad pK_3 = 7.3 \quad (6)$$

From the dissociation constants pK_1, pK_2, and pK_3, it can be seen that $H_3FeO_4^+$ is dominant at pH 1, while H_2FeO_4 dominates at pH 2 and 3, the $HFeO_4^-$ predominates at pH 4 - 6, and FeO_4^{2-} dominates at pH 8 and 9. The $H_3FeO_4^+$ is very unstable compared to other forms, and the oxidation power of the three active species is found to rise in the order of $FeO_4^{2-} < HFeO_4^- < H_2FeO_4$, [30]. Furthermore, in acidic media pH 2, the H_2FeO_4 is less stable than pH 3 environment. Besides, although ferrate is less stable in acidic than alkaline environments, the reaction between ferrate(VI) and MO dominates the self-decomposition reaction of ferrate(VI) in water, resulting in a greater elimination of MO. Thus, the pH 3 was suitable for MO degradation, and this result is consistent with previous research [28, 30].

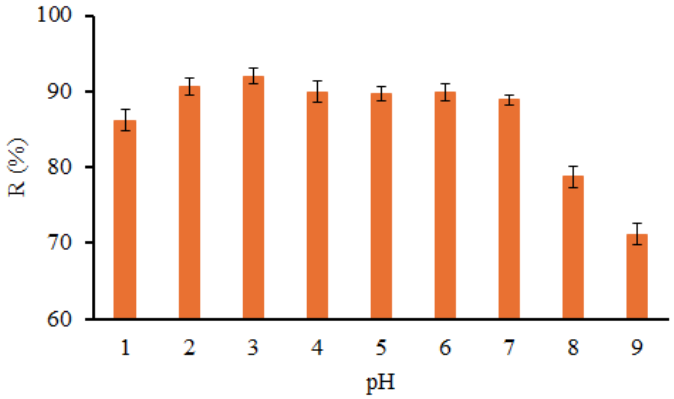


Figure 1. Removal efficiency of MO at different pH. Experimental condition: [MO] = 50 mg/L, molar ratio of Fe(VI):MO of 3.4:1.0, T of 20 °C, treatment time of 60 min.

3.2. Effect of ferrate(VI) dosages on the MO degradation

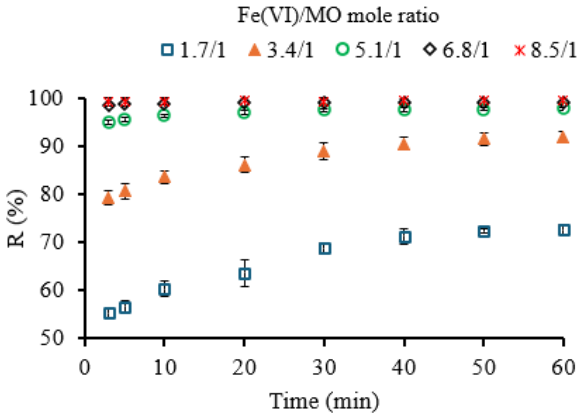


Figure 2. Removal efficiency of MO at different molar ratios of ferrate(VI) to MO over time. Experimental condition: [MO] = 50 mg/L, pH 3, T of 20 °C.

The degradation of MO by ferrate(VI) was studied at different molar ratios of ferrate(VI) to MO over time at pH 3. The result given in Figure 2 shows that MO degraded primarily within the first 3 minutes at all these molar ratios. The MO removal achieved 94.9 to 99.3 % after 3 minutes at higher ferrate(VI) dosages (5.1:1.0, 6.8:1.0, and 8.5:1.0), while utilizing a molar ratio of 3.4:1.0 and 1.7:1.0 resulted in 79.3 % and 55.2 %, respectively. Following that, with extending the reaction time from 3 to 60 minutes the MO removal increased slowly from 0.1 to 4.0 % for higher ferrate(VI) dosages, but increased from 13.0 to 17.5 % with lower ferrate(VI) dosages. Especially, after 40 to 60 minutes, the MO treatment efficiency was almost unchanged with all ferrate dosages. The MO degradation increased consistently as the molar ratios of Fe(VI) to MO rose. This was explained by an increase in the amount of Fe(VI) available for oxidizing MO. At molar ratio of Fe(VI) to MO greater than 5.1:1.0, the ferrate could be totally oxidized MO, and removal achieved of 99 % after 60 minutes.

3.3. Effect of temperature on MO degradation

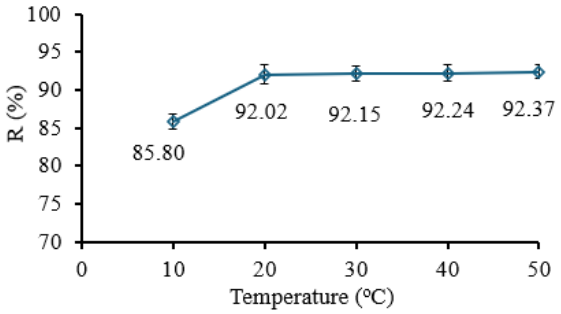


Figure 3. Effect of temperature on the MO degradation. Experimental condition: [MO] = 50 mg/L, molar ratio of Fe(VI): MO = 3.4:1.0, pH 3, treatment time of 60 min.

Figure 3 shows the influence of temperature on the removal of MO. The degradation efficiency was the lowest at 85.80 % at 10 °C. As the temperature increased from 20 to 50 °C, the removal changed negligibly, about 92 %. Thus, the suitable temperature range for MO treatment with ferrate(VI) was from 20 to 50 °C.

3.4. Effect of constituents on MO degradation

Natural water contains inorganic ions, which may affect the removal of organic contaminants. This work studies the impact of two anions (NO_3^-, and Cl^-) and three cations (Cu^{2+}, Mg^{2+}, and Fe^{3+}) on the MO degradation, and the results are given in Figure 4. Ferrate(VI) was synthesized by anodic dissolution of ductile in 14 M NaOH solution [27, 28]. Therefore, ferrate solution contains Na^+ cation, and the blank sample also contains Na^+. To evaluate the influence of Cl^- and NO_3^-anions, we used NaCl and $NaNO_3$ salts to add to the solution. The amount of Na^+ ion increased by a maximum of 10 mM compared to the blank sample, however, according to previous research [31], the Na^+ ions did not affect the organic matter treatment process. Thus, the experimental results here were influenced by anions Cl^- and NO_3^-. Figure 4(a) shows that no obvious effect on MO removal was found after addition of NO_3^- and Cl^- anions. The MO removal efficiency varied slightly from 97.7 to 98.5 % in the presence and absence of these anions, with anion concentrations ranging from 1 to 10 mM. This result is consistent with some previous studies [31, 32]. Similarly, the influence of cations Cu^{2+}, Mg^{2+} and Fe^{3+} was investigated by adding salts $CuSO_4$, $MgSO_4$ and $Fe_2(SO_4)_3$. The blank sample also contained anion SO_4^{2-} because H_2SO_4 was used to adjust pH, and SO_4^{2-} did not affect on the oxidation process of ferrate [31]. Therefore, the obtained results were due to the influence of additional cations. The results given in Figure 4(b) present that Mg^{2+} and Fe^{3+} cations had a negligible effect, but the Cu^{2+} cation decreased the MO removal. Increasing Cu^{2+} cation concentration from 1 to 10 mM, the degradation dropped from 96.4 to 95.4 %, as compared to the 97.8 % removal in the blank sample. This finding can be explained by the tendency of transition metal ions to form complexes with organic molecules, reducing the organic degradation efficiency of ferrate(VI) [31].

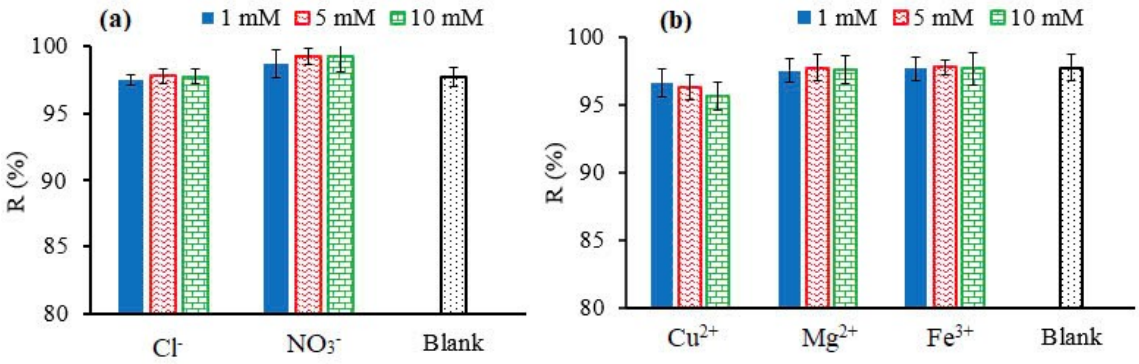


Figure 4. Effect of anions (a) and cations (b) on the MO degradation. Experimental condition: [MO] = 50 mg/L, molar ratio of Fe(VI):MO = 5.1:1.0, pH 3, T of 20 °C.

3.5. MO destruction by ferrate

Organic molecules are oxidized with ferrate(VI) via one- or two-electron transfer, hydrogen abstraction, or oxygen transfer [33, 34]. The attack of Fe(VI) on the azo bond is the first step in MO degradation. Following that, the aromatic ring is hydroxylated to provide phenolic metabolites. The amino group can either be converted to hydroxylamine. The breakdown products of MO can be further degraded to H_2O, CO_2, and inorganic ions such as ammonium, nitrate, and sulfate [34].

To investigate destruction mechanism of MO by ferrate (VI), MO solutions before and after adding ferrate(VI) at different times were analyzed by UV-vis spectra and LC-MS chromatograms. UV-vis spectrum of the initial MO solution in Figure 5 shows a strong absorption peak at 509 nm assigned to the azo bond (-N=N-) and two peak at 280 nm and 320 nm attributed to the aromatic ring [35-37]. As treatment time increased from 5 to 60 minutes, the intensity of all the peaks reduced, indicating the breakage of the azo bond and the destruction of the aromatic ring. After treatment time of 60 minutes, all absorption peaks were faint, suggesting that most of the MO and intermediate organic molecules had been destroyed and removed.

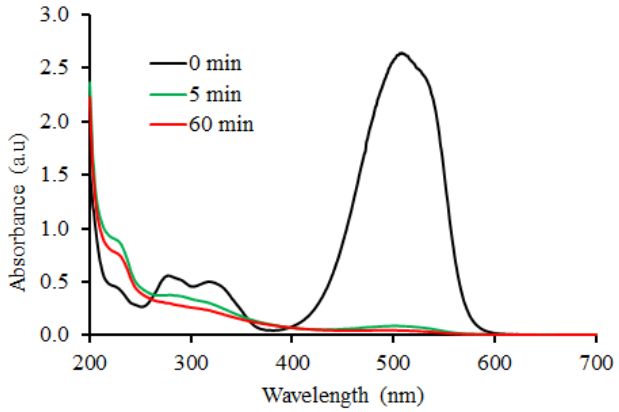


Figure 5. UV-vis spectra of MO solution after different treatment time.

The products produced during the oxidation of MO solution by ferrate(VI) were determined by LC-MS analysis. Figure 6 shows the LC-MS chromatograms of MO solutions

before and after 60 minutes of treatment. The spectrum of the initial MO solution contains a significant signal at m/z = 304, which indicates the MO characteristic (Figure 6(a)). After treatment time of 60 minutes, new signals form at m/z = 202, 172, 166, and 136, and the intensity of these signals is hundreds of times lower than that of initial MO (Figure 6(b)), corresponding to the proposed by-products listed in Table 1.

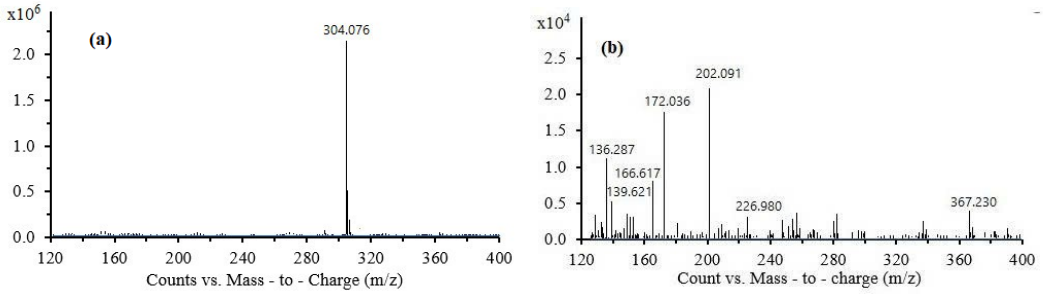
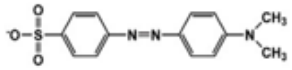
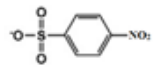
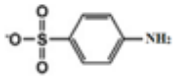
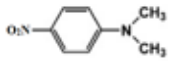
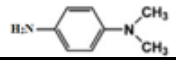


Figure 6. LC-MS chromatograms of MO solutions before and after treatment by ferrate(VI).

Table 1. The proposed by-products of MO degradation by ferrate(VI).

Molecular weight	Structure
304	
202	
172	
166	
136	

The evaluation of MO mineralization is measured by the reduction in total organic carbon (TOC) and chemical oxygen demand (COD). For this purpose, the TOC and COD tests of the initial MO solution and the MO solution after 60 minutes of treatment with ferrate were performed. The TOC decreased from 23 to 10 mg/L, which corresponded to 57 % TOC removal. Similarly, the COD removal was 67 %. As a result, the intermediates of the MO oxidation process would partially breakdown and mineralize into CO_2 and H_2O, as previously reported [34, 36].

3.6. Comparison of MO degradation by ferrate(VI) with other methods

In order to remove MO from waste water, many treatment methods have been used, such as adsorption, chemical coagulation, photocatalysis, electrocatalytic oxidation, advanced oxidation processes (AOPs), and biological methods [6-10, 38-40]. However, the adsorption and chemical coagulation convert dyes from the liquid to the solid phase, so it causes secondary pollution. The biological method is not suitable for MO treatment due to its low

biodegradability. Other methods provide operational challenges and high capital expenses. The various literature reports on the degradation of MO are shown in Table 2. The comparative study reveals that ferrate(VI) exhibits better removal efficiency in a short time. Furthermore, the ferrate is a green oxidant, easily synthesized, and low-cost, making ferrate(VI) a viable choice for removing MO in textile dyeing effluent.

Table 2. Comparing treatment methods for the MO degradation.

Treatment system	Treatment method	Degradation time (min)	Removal efficiency (%)	Ref.
$O_3/K_2S_2O_8$	Oxidation	50	98.59	[10]
H_2O_2/Fe^{2+} - UV	Fenton reaction	15	97.8	[9]
$[FemIL@SiO_2@Mag]_2MoO_4$ - UV	Photocatalysis	30	99.00	[4]
Fe_3O_4/TiO_2 - UV	Photocatalysis	60	90.3	[38]
MoS_2/Fe_3O_4 - UV	Photocatalysis	100	79.53	[39]
PbO_2 -TiO_2	Electrocatalytic oxidation	50	99.31	[40]
Ti/SnO_2 -Sb_2O_3 /PbO_2 -TiO_2	Electrocatalytic oxidation	240	95.5	[8]
Reduced – TiO_2	Photoelectro-catalytic process	30	98.4	[36]
Ferrate(VI)	Oxidation	3	99.3	This study

4. CONCLUSIONS

The results of the MO treatment demonstrated that ferrate(VI) can efficiently remove MO from water. The removal efficiency was strongly dependent on pH value, and the suitable pH was 3. Increasing the ferrate(VI) dosages improved the degradation performance, and the MO removal was most effective with the ferrate (VI) dosage greater than 5.1:1.0. The MO oxidation reaction of ferrate(VI) occurred mainly in the first 3 minutes. Removal efficiency achieved 99.3 % after 3 minutes at the ferrate(VI) dosage of 8.5:1.0. The presence of cation Cu^{2+} in water decreased the efficiency of MO removal by ferrate(VI). The MO destruction was investigated by UV-vis spectra and LC-MS chromatograms of MO solutions before and after treatment by ferrate(VI).

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CRediT authorship contribution statement. Nguyen Thi Van Anh: Formal analysis, Investigation, Methodology. Phan Thi Binh: Formal analysis, Supervision. Tran Huu Quang, Nguyen The Duyen, Phan Hoang Yen: Formal analysis. Mai Thi Thanh Thuy: Formal analysis, Methodology, Project administration, Writing – review & editing.

Declaration of competing interest. The authors state there is no conflict of interest.

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