Optimization of the operating parameters using RSM for the Fenton oxidation process and adsorption on vegetal carbon of MO solutions

Adriana Saldaña-Robles a, Ricardo Guerra-Sánchez a, Manuel I. Maldonado-Rubio b, Juan M. Peralta-Hernández a,*

a Centro de Innovación Aplicada en Tecnologías Competitivas (CIATEC), Departamento de Investigación Ambiental, Omega-201, Fraccionamiento Industrial Delta, 37545 León, Guanajuato, Mexico
b Plataforma Solar de Almería (CIEMAT), Carretera Senés, Km 4, 04200 Tabernas (Almería), Spain

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A B S T R A C T

This study focused on the application of RSM on the Fenton process and the adsorption of vegetal carbon (VC) to obtain the optimal conditions for the minimization of the colored synthetic wastewater. Methyl orange (MO) with an azo dye was used as the model organic compound. Fenton processes were investigated to establish the optimal conditions. The Fe2+/H2O2 ratio was studied to establish the major MO degradation when 100 and 200 mg/L of MO were treated. For the adsorption process, to determine the optimal conditions, the principal variables studied were the vegetal carbon mass dosage, degradation time and dye concentration.

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1. Introduction

Large quantities of dye-containing wastewater are commonly discharged into local water treatment facilities during the manufacturing and processing of textile industry products [1]. The effluents of the textile industry are heavily colored due to high levels of dissolved organic matter [2], and these compounds must be treated before being discharged into the environment.

Several treatment technologies, including biological, physical and chemical processes, have been used for the removal of azo dyes from water. It has been reported that a number of biological and physicochemical methods have low efficiencies for dye removal [3–7]. Nonetheless, the most promising physicochemical alternatives include the so-called advanced oxidation processes (AOPs). These processes are based on the production of free hydroxyl radicals (•OH) as a potentially strong oxidant for organic compounds [8–10]. The most commonly used AOP is Fenton’s reaction [11–13].

Fenton’s reaction is an advanced oxidation process in which •OH species are generated from hydrogen peroxide (H2O2) and a catalytic mixture of Fe(II)/Fe(III) [14]. In Fenton’s reaction, ferrous ions react with H2O2 to produce the •OH radicals, as follows:

\[
Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + •OH + HO^-
\]  

(1)

The Fe3+ can then be reduced by reaction with the excessive H2O2 to reform ferrous ions and more radicals. The latter step in the second process is slower [15] than reaction (1) and allows Fe3+ regeneration, giving way to a catalytic mechanism, as shown below:

\[
Fe^{3+} + H_2O_2 \leftrightarrow FeOOH^{2+} + H^+
\]  

(2)

\[
FeOOH^{2+} \rightarrow HO_2^- + Fe^{2+}
\]  

(3)

\[
Fe^{3+} + HO_2^- \rightarrow Fe^{2+} + O_2 + H^+
\]  

(4)

A number of reported studies have demonstrated that the removal of organic compounds from water by Fenton’s reagent is highly efficient and inexpensive [16] compared with other treatments, such as membrane-based methods or the coagulation–floculation processes. In addition, the byproducts of AOPs either are CO2 and H2O when complete oxidation takes place or are byproducts that could be efficiently treated by biological processes or removed by adsorption treatment.

Dyes have a great variety of organic structures characterized by the presence of unsaturated groups that act as chromophores and...
provide the dye its color, such as –C≡C–, –N=N– and –C≡N–, and functional groups responsible for the dye’s fixation to fibers, such as –NH₂, –OH, –COOH and –SO₃H [17–19]. In this sense, methyl orange (MO), the chemical structure of which is shown in Fig. 1, is an azo dye belonging to the acid dye family.

Porous material has such properties as ion-exchange, catalysis and separation [20,21] and has been used both as an adsorbent and as a catalyst in various adsorbents for the removal of dyes. The adsorption capacity of porous material is largely controlled by the amount of available surface area [22,23]. Carbon materials have been attractive due to their potential number of micropores and consequently high surface areas [24].

In this study, the removal of MO dye from water has been investigated using both the Fenton process and an adsorbent, highly porous VC. Both processes were studied, and the decolorization efficiency was compared.

2. Methodology

2.1. Chemicals

MO dye was purchased from Sigma–Aldrich and was used to prepare a stock solution of 1000 mg/L. Appropriate dilutions were prepared to obtain solutions with concentrations of 100, 150 and 200 mg/L. Hydrogen peroxide (H₂O₂) 30% (w/v) and ferrous sulfate heptahydrate (FeSO₄·7H₂O) 99.5%, both from Merck, were used for the Fenton oxidation. For all experiments, deionized water was used to prepare the required solutions. Commercial VC was used as a porous material. The VC was purchased in a local commercial center.

2.2. Dye concentration

To determine the MO dye concentration in a solution, UV–vis spectroscopy was employed using a spectrum in the range of 200–600 nm. Two main bands were detected at 508 and 260 nm. The 508-nm wavelength band was selected to measure the MO dye concentration in solution.

2.3. Fenton oxidation

To degrade MO dye from aqueous solution, free hydroxyl radicals were obtained in situ by Fenton’s reagent at room temperature and pressure. Solutions of 1000 mL of MO dye with concentrations of 100, 150 or 200 mg/L were continuously stirred at 170 rpm, and defined quantities of H₂O₂ (8, 14 or 20 mM) and Fe²⁺ solution (0.1, 0.2 or 0.3 mM) were added to the dye solution, which was chosen according to the technical literature [25]. Aliquots were withdrawn from the solution, before and during the experiment, to determine the initial and the remaining concentration of MO dye in solution.

2.4. Adsorption experiments

The effect of the VC mass dosage and the initial dye concentration on the adsorption capacity of the MO dye was evaluated by VC batch adsorption experiments. The experiments were conducted at room temperature in an open beaker with a 300-mL capacity at 170 rpm, and suspensions of 200 mL of solutions with different initial dye concentrations (100, 150 or 200 mg/L) were stirred with different mass dosages (0.5, 0.75 and 1 g.) Aliquots were withdrawn from the solution before and throughout the experiment. The initial and final (at equilibrium) dye concentrations were spectrophotometrically measured at 508 nm. All experiments were conducted in duplicate.

2.5. Response surface methodology

Response surface methodology (RSM) was applied for the experiments. RSM is an experimental methodology that allows the optimal conditions of a process to be found when the experimental region is delimited by the experimentation range of each factor [26]. RSM is essentially a particular set of mathematical and statistical tools for designing experiments, building models, evaluating the effects of the operating conditions, and most importantly, for researching the optimal values of factors to predict target responses [27–29].

RSM allows for a considerable reduction in the number of experiments and easy interpretation by making it possible to study a large number of factors and detect the possible interactions between them [30]. Using this experimental design methodology, it was possible to design and optimize the decolorization efficiency of MO using both the Fenton process and adsorption by VC and to construct a prediction model for the response.

2.5.1. Central Composite Design (CCD) by the Fenton process

A Central Composite Design (CCD) was used in the RSM, and the codec factors and corresponding levels for the MO decolorization efficiency with by the Fenton process are shown in Table 1. The data were evaluated by analysis of variance (ANOVA) using Design Expert Version 6.0.1 (StatEase, USA). The design was performed with 52 experiments: 16 factorial points (2 replicates), 4 central points and 8 axial points (2 replicates) and a 99% confidence interval.

2.5.2. Central Composite Design (CCD) by the adsorption process

A CCD was used in the RSM, and the codec factors and corresponding levels for the MO decolorization efficiency with VC are shown in Table 2. The data were evaluated by ANOVA using Design Expert Version 6.0.1 (StatEase, USA). The design was performed with 30 experiments: 8 factorial points (2 replicates), 2 central points and 6 axial points (2 replicates) and a 99% confidence interval.

3. Results and discussion

3.1. Decolorization efficiency of MO by the Fenton process

The Fe²⁺ and H₂O₂ concentrations are the key factors in improving the efficiency of the Fenton process. However, both

\begin{equation}
\text{Table 1}
\end{equation}

<table>
<thead>
<tr>
<th>Experimental design of MO decolorization efficiency by Fenton process.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Independent variable</td>
</tr>
<tr>
<td>-----------------------</td>
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<tr>
<td></td>
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<tr>
<td>FeSO₄(mM)</td>
</tr>
<tr>
<td>H₂O₂(mM)</td>
</tr>
<tr>
<td>MO(mg/L)</td>
</tr>
<tr>
<td>t(min)</td>
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</tbody>
</table>

\begin{equation}
\text{Table 2}
\end{equation}

<table>
<thead>
<tr>
<th>Experimental design of MO decolorization efficiency by VC.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Independent variable</td>
</tr>
<tr>
<td>----------------------</td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td>VC(g)</td>
</tr>
<tr>
<td>MO(mg/L)</td>
</tr>
<tr>
<td>t(min)</td>
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</table>
H₂O₂ and Fe²⁺ can also react with *OH and can therefore inhibit oxidation reactions if either of them is not at the optimal concentration. The optimal concentrations of H₂O₂ and Fe²⁺ could be affected by the matrix of the treated water sample and by the expected degree of wastewater mineralization. The decolorization efficiency (\( \eta \)) is defined as

\[
\eta (%) = \left( \frac{C_0 - C}{C_0} \right) \times 100, \tag{5}
\]

where \( C_0 \) and \( C \) are the MO concentrations at the initial time and a given time \( t \), respectively [5]. The decolorization efficiency experiments were conducted by the Fenton process proposed in Sections 2.3 and 2.5. Fig. 2 depicts the influence of Fe²⁺ and H₂O₂ on the decolorization efficiency for MO.

As seen in Fig. 2a, the decolorization efficiency rate decreased as H₂O₂ was added to the solution. A maximum decolorization efficiency was reached with an H₂O₂ concentration of 8 mM. The enhancement in the oxidation power is noticeable in Fig. 2b, suggesting that the process was improved as the Fe²⁺ concentration was increased from 0.1 to 0.3 mM (Fig. 2b). An MO decolorization efficiency of 70% was achieved after 22 min of reaction. In Fig. 2c and d, the same behavior was observed as in Fig. 2a and b. The MO decolorization efficiencies for Fig. 2c and d were 5% and 45%, respectively.

3.2. Decolorization efficiency of MO by adsorption

The decolorization efficiency (\( \eta \)) is defined in Eq. (5). Decolorization efficiency experiments were conducted using the adsorption process proposed in Sections 2.4 and 2.5. Fig. 3 depicts the influence of VC on the decolorization efficiency for MO. As seen in Fig. 3a, the decolorization efficiency rate increased as VC was added to the solution, reaching a maximum decolorization efficiency of 72% with a VC mass dosage of 1 g. The adsorption process improved as the VC mass dosage increased from 0.5 to 1.0 g (Fig. 3b). An MO decolorization efficiency of 78% was achieved after 400 min of reaction.

3.3. Experimental design and optimization of operation conditions in the decolorization efficiency of MO

3.3.1. Fenton process

In light of the experimental design results (Table 3), a cubic regression model that established the correlation between the decolorization of MO and the independent factors was determined.
to be

\[ \eta(\%) = 2.81 + 6.48A + 0.85B - 12.31C - 2.28D - 3.63AB \\
+ 2.55AC + 4.87AD + 0.15BC - 3.52BD - 3.76CD \\
+ 3.86A^2 + 1.56B^2 + 7.5C^2 + 9.6D^2 - 3.37ABD \\
- 1.58ACD - 3.73A^2B + 4.79A^2C + 8.14A^2D \\
+ 9.56AB^2 \]

(6)

In Eq. (6), a positive effect of a factor means that the response is improved when the factor level increases, and a negative effect of the factor means that the response is not improved when the factor level increases.

Table 4 presents the ANOVA results for the model, indicating that the model was significant for MO decolorization efficiency with a $R^2$ value of 0.99. For this model, $P > F$ is less than 0.0001, indicating that the term was significant in this model. $A$:FeSO$_4$, $C$:MO and $D$:t were significant factors, and $AB$, $AC$, $AD$, $BD$, $CD$, $A^2$, $B^2$, $C^2$, and $D^2$ were significant interactions.
C\textsuperscript{2}, D\textsuperscript{2}, ABD, ACD, A\textsuperscript{2}B, A\textsuperscript{2}C, A\textsuperscript{2}D, AB\textsuperscript{2} AD\textsuperscript{2} were significant model interactions. The seventh column of this table shows the percent contribution (PC\%) of each of the individual terms in the model; the principal factors that showed a greater contribution were C, AD, AB\textsuperscript{2}, AD\textsuperscript{2}, C\textsuperscript{2} and A. The standardized Pareto chart, which demonstrates the importance of the factors and their interactions and agrees with the PC\%, is shown in Fig. 4.

The lack of fit, which is significant in the present model, does not invalidate the model because the $R^2$ is greater than 0.9 and the standard error is relatively small, showing a good fit with the experiments. This test fails when a systematic component of the unexplained variance is large compared to the pure error component. This phenomenon is observed when there is a poor fit of the model to the data, but it can also be observed when the model adequately represents the data, but the precision of the measurements is so high that the pure error is small. To test the model, the optimal point has been reproduced by experimentation. This reproduction has been performed by selecting a point from the space of the optimal solution.

3.3.1.1. Response analysis of MO decolorization efficiency. The decolorization efficiency improved when the factor (Fe\textsuperscript{2+} concentration) increased from 0.1 to 0.3 mM (Fig. 5) while holding the other factors constant at 8.9 mM of H\textsubscript{2}O\textsubscript{2} and 115 mg/L of MO with a time of 27 min.

![Fig. 3. Adsorption of MO in the presence of different VC concentrations (a) 0.5 g (×) 1.0 g under different MO concentrations: (a) $C_0 = 100$ mg/L and (b) $C_0 = 200$ mg/L.](image)

![Fig. 4. Pareto chart standardized for Fenton process, (+) and (−) on the Y axis is the effect positive and negative respectively in the model.](image)

![Fig. 5. One factor plot of Fe\textsuperscript{2+} for MO decolorization efficiency.](image)
A study of the response surface and contour plots provided an easy method for optimization of the efficiency of the Fenton process. The response surface plot for MO decolorization efficiency is presented in Fig. 6a, in which the MO concentration was established at 115 mg/L, and the time was established at 27 min. The response surface of the decolorization efficiency increased with the increasing Fe²⁺ concentration from 0.1 to 0.3 mM and decreased as the H₂O₂ concentration increased from 8 to 20 mM. In the scope of the results, the maximum value of the decolorization efficiency of MO was 91% at 0.2851 mM of Fe²⁺ and 8.9 mM of H₂O₂ (Fig. 6b).

In the perturbation plot, Fig. 7 shows the effect when all factors at the optimal experimental conditions in the design space are compared. The perturbation plot assists in the comparison of the effects of all of the factors and a particular point in the design space; when the factor curvature is sharper, the factor effect is more important to the response. The plot was obtained for 115 mg/L of MO and 27 min. Fig. 7 shows that the response of MO decolorization efficiency was most sensitive to the Fe²⁺ concentration followed by the H₂O₂ and MO concentrations and, finally, the degradation time.

<table>
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<tr>
<th>Experimental number</th>
<th>Code level of factors</th>
<th>Observed percentage of removals</th>
<th>Average</th>
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</table>
3.3.1.2. Verification study. To confirm the capability of the model, one assay was randomly selected from the space of optimal conditions given by the model to obtain a maximum MO decolorization efficiency result. Fig. 8 shows the experimental results under the optimal conditions, which have been compared with the decolorization efficiency by the Fenton process without RSM analysis. As seen in Fig. 8, the MO decolorization efficiency was close to 84.95% compared with 70.28% for the Fenton process without RSM analysis.

The evolution of MO by the Fenton process under non-optimized conditions (t = 20 min, 0.3 mM Fe\(^{2+}\), 8 mM H\(_2\)O\(_2\), 100 mg/L MO) and optimized conditions (t = 20 min, 0.2851 mM Fe\(^{2+}\), 8.9 mM H\(_2\)O\(_2\) and 115 mg/L MO) is shown in Fig. 9. The concentration of MO by the Fenton process under non-optimized conditions (t = 20 min, 0.3 mM Fe\(^{2+}\), 8 mM H\(_2\)O\(_2\), 100 mg/L MO) and optimized conditions (t = 20 min, 0.2851 mM Fe\(^{2+}\), 8.9 mM H\(_2\)O\(_2\) and 115 mg/L MO) is shown in Fig. 9. The concentration of MO by the Fenton process under non-optimized

<table>
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<tr>
<th>Variation source</th>
<th>Sum of square</th>
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<th>F-value</th>
<th>P-value</th>
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<tr>
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**Table 6**
ANOVA results of the cubic model of MO decolorization using EFP.

Fig. 8. Degradation of MO under optimum conditions by CCD design.

Fig. 9. Degradation of MO by Fenton under non-optimized conditions (o) and optimized conditions (c).

Fig. 10. Pareto chart standardized for adsorption process, (+) and (−) on the Y axis is the effect positive and negative respectively in the model.
conditions (a) slowly decreased and had a smaller MO degradation efficiency than the Fenton process under optimal conditions (b). The results indicate the suitability of the model. The RSM was successfully applied to optimize the Fenton process of MO decolorization.

3.3.2. Adsorption process
In light of the experimental design results (Table 5), a cubic regression model, which establishes the correlation between the MO decolorization efficiency and the independent factors, was found to be

\[ \eta(\%) = 63.80 + 13.32A + 8.93B + 32.79C - 2.81AB - 3.89AC + 1.71A^2 - 7.45C^2 + 3.31ABC - 23.69A^2C \]  

(7)

A positive effect of a factor means that the response is improved when the factor level increases and a negative effect of the factor means that the response is not improved when the factor level increases.

The results for the model are presented in the ANOVA of Table 6, where the model’s F-value of 30.16 implies that the model was significant for MO decolorization efficiency with an \( R^2 \) of 0.93. For this model, \( P > F \) is less than 0.0001, indicating that the term was significant. A, B, C and \( A^2C \) are the most significant terms in the present model, which is in agreement with the PC% of each of the individual terms in the model. Fig. 10 shows the Pareto chart, which presents the standardized effect for each factor and interaction. VC, t and MO are the terms that show a greater contribution. The lack of fit is significant; however, this finding does not invalidate the model because the \( R^2 \) is greater than 0.9 and the standard error is relatively small, indicating a good fit with the experiments. This test can fail when the precision of the measurements is sufficiently high that the pure error is small in comparison with the lack of fit error. To test the model, the optimal point was reproduced by experimentation. This reproduction was achieved by selecting a point from the space of the optimal solution.

3.3.2.1. Response analysis of MO decolorization efficiency. The decolorization efficiency improved when the factor VC increased from 0.5 to 0.85 g, and the decolorization efficiency decreased when this factor increased from 0.87 to 1 g (Fig. 11) while holding the other factors constant at 118 mg/L MO and 1719 min.

![Fig. 11. One factor plot of VC for MO decolorization efficiency.](image)

A study of the response surface and the contour plots provided an easy method for the optimization of the adsorption process efficiency. The response surface plot for MO decolorization efficiency is shown in Fig. 12a, in which the MO concentration was established at 118 mg/L MO. The semispherical response surface of the decolorization efficiency increased with an increasing VC concentration from 0.5 to 1 g and increased as the adsorption time increased from 10 to 1800 min of treatment. In the scope of the results, the maximum value of the decolorization percentage of MO was 83% at 0.77 g VC and 1719 min (Fig. 12b).

The perturbation plot, Fig. 13, presents the effect when all of the factors are compared at the optimal experimental conditions in the design space. The perturbation plot provides additional
information on the comparison of the effects of all the factors at a particular point in the design space; when the factor curvature is sharper, the factor effect is more important to the response. The plot was obtained for 118 mg/L of MO and 1719 min of treatment. Fig. 13 demonstrates that the response of MO decolorization was most sensitive to the adsorption time followed by the VC biomass dosage and, finally, by the MO concentration.

3.3.2.2. Verification study. To confirm the capability of the model, one essay was randomly selected from the space of optimal conditions given by the model to obtain a maximum MO decolorization efficiency result. Fig. 14 presents the experimental results under the optimal conditions, which have been compared with the MO decolorization efficiency of the adsorption process without RSM analysis. Fig. 14 shows an MO decolorization efficiency close to 79.17% compared with the 72.19% efficiency for the adsorption process without RSM analysis.

The behavior of MO by the adsorption process under non-optimized conditions (t = 1800 min, 1 g VC, 100 mg/L MO) and optimized conditions (t = 1719 min, 0.77 g VC and 118 mg/L MO) is presented in Fig. 15. As observed, the MO decolorization efficiency by the adsorption process under non-optimized conditions (○) slowly decreased and absorbed a lower MO concentration than the adsorption process under the optimal conditions (□).

The results indicate the suitability of the model. The RSM was successfully applied to optimize the adsorption process of MO.

4. Conclusions

The Fenton process and adsorption treatment for MO degradation in a model wastewater was investigated on a laboratory scale. The study demonstrated that the response surface methodology and CCD statistical experimental design could provide statistically significant results for Fenton and adsorption degradation of MO and could determine the optimal conditions for enhancing the overall MO degradation efficiency. Independent variables and their interactions were found to be effective in decolorization by both processes.

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