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Investigating the Capability of MCM-41 Nanoparticle for COD Removal From Iraqi Petroleum Refinery Wastewater

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Abstract. Nanoparticles (NPs) can be used as adsorbents in the removal of different pollutants from oil wastewater discharged from petroleum refineries. This paper aims to investigate the ability of NP type Mobil Composition of Matter No. 41 (MCM-41) to eliminate chemical oxygen demand (COD) from oily wastewater produced from an oil refinery plant located in Baghdad, Iraq. The work was carried out under the effect of the dosage of NPs (0.02–0.10 g), reaction time (5–120 min), and mixing speed (100–250 rpm) using a batch-scale reactor. The experiments were designed to belong to the RSM-Central composite design statistical method. Minitab statistical program was performed to analyze the results and find the mathematical correlations and optimum values of the operational variables. Results reveal the treatability of this type of NPs to minimize COD concentration in discharged oily wastewater. The removal efficiency of COD reaches 99.58% at optimum values of 0.06 g, 64.24 min, and 174.24 rpm for NP dosage, reaction time, and stirring speed, respectively.

***Keywords*:** Oily wastewater; COD; Nanotechnology; RSM-CCD design; Optimization; Adsorption models

INTRODUCTION

Large amounts of wastewater are discharged from petroleum refineries and petrochemical activities every year because large quantities of water are consumed, but the consumption value is high [1, 2]. These wastewaters affect the environment, whether the aquatic systems or soil [3]. The contaminants included in oily wastewater differ in their

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kinds, concentration, and effect intensity depending on the oil field sites; therefore, oily wastewater is classified as a complex crisis [4, 5].

Wastewater has several pollutants with different pollution levels such as organic compounds, including oil content, chemical oxygen demand (COD), biochemical oxygen demand, and total organic carbon, and inorganic compounds, including heavy metals.

Several conventional treatment methods have been used to eliminate different pollutants from oily wastewater, such as coagulation/flocculation [7], adsorption [8], flotation [9], membrane technologies [10, 11], and electrochemical technologies [12, 13]. However, most of these methods have drawbacks such as low cost, non-considerable efficiency, and the formation of secondary pollutants [14].

Nanotechnology has been performed to eliminate pollutants from wastewater discharged from different industrial activities by using different types of nanomaterial, which have several specifications such as chemical and mechanical properties that allow the effective removal of different pollutants from organic wastewater [15]. Adsorption is primarily used by these materials to remove pollutants, where the high surface-to-volume ratio provides these materials high adsorption efficacy [16–18].

Nanomaterials are used for several applications, such as carbon nanotubes (single and multi-walls), $BaFe_2O_4$ nanoparticles (NPs), and metal oxides [19–22]. The type of MCM-41 nanoparticles has a hexagonal and ordered mesoporous silica material with uniform mesopores. They have a high surface-to-volume ratio, high magnetic saturation and mechanical resistance, a significant controlling ability of the pore dimension, and potential acidity [15].

This work aimed to investigate the ability of Mobil Composition of Matter No. 41 (MCM-41) nanoparticles (MCM-41 NPs) to remove COD pollutants from real oily wastewater discharged from an oil refinery. Experiment design and result analysis were performed using the response surface methodology-central composite design (RSM-CCD) statistical method and a statistical software program (Minitab). Moreover, the adsorption isotherms and kinetics of the treatment process have been studied.

MATERIALS AND METHODS

CHEMICALS AND ANALYTICAL ANALYSIS

Real oily wastewater was obtained from a refinery crude oil plant located in Al-Dora city/Baghdad, Iraq. The collected samples were preserved in a polypropylene container to be treated by a batch reactor. The COD concentration presented in this wastewater was 257 ppm. Other chemicals used for preparing MCM-41 nanoparticles included ethanol (purity 99%), sodium hydroxide (NaOH), cetyltrimethyl ammonium bromide (purity 99%), and tetraethyl orthosilicate (purity 98%), which were all manufactured by Sigma Aldrich. MCM-41 nanoparticles were prepared in accordance with the scientific method found in the literature [23, 24]. At the end of each experiment, the analytical determination of COD concentration in treated solutions was estimated using an oil analyzer (Ocma-350-Horiba LTD, Japan). The characterization of MCM-41 was also performed on the adsorbent using X-ray diffraction, scanning electron microscopy, and Fourier transform infrared spectroscopy in a previous study [15].

The COD removal efficiency was estimated using Eq. (1):

$$Y_{CODR} = \frac{C_i - C_t}{C_i} \times 100\% \quad (1)$$

Where Y_{CODR} is the percentage of COD removal; Ci and Ct are the initial and final concentrations of COD, respectively (mg COD/L).

In addition, the amount of COD pollutant adsorbed per gram of MCM-41 (mg/g), that is, the adsorption capacity (qe), was measured using Eq. 2 [25]:

$$q_e = \frac{(C_i - C_t)\,V}{M} \quad (2)$$

Where q_e is the adsorption capacity at equilibrium (mg/g); V is the sample volume (Liter), and M (g) is the amount of MCM-41 used.

APPARATUS

Batch treatment experiments were conducted using a 250 mL glass reactor placed on a magnetic agitator (BS-21, Heidolph, Germany) to provide the designed agitation speed. Each experiment was conducted by treating 100 mL of wastewater under the effects of MCM-41 nanoparticle dosage, speed of the stirrer, and reaction time. After each experiment, the sample was filtered to separate nanoparticles. The filtrate was then analyzed using an oil analyzer to determine the final concentration of COD pollutant.

EXPERIMENTAL DESIGN

The design of treatment experiments was maintained using the RSM-CCD method. Analysis of results, modeling, and optimization was conducted using a statistical program (Minitab). The influence of the operational variables shown in Table 1 on the COD removal efficiency was investigated. The actual and coded values of these variables are shown in Table 2, where the rotatability is 1.732. The total number of runs is 19 based on the RSM-CCD method [29,30].

TABLE 1. Operational variables

Parameters	Ranges
X_1: NP dosage (g)	0.025–0.10
X_2: Reaction time (min)	5–120
X_3: Stirring speed (rpm)	100–250

TABLE 2. Actual and coded variables

Natural variable (Xi)	Coded variables				
	−1.732	−1	0	1	1.732
X_1: NP dosage (g)	0.02	0.04	0.06	0.08	0.1
X_2: Reaction time (min)	5	30	62	96	120
X_3: Stirring speed (rpm)	100	130	175	175	250

A mathematical correlation related the response required to the operating variables using Eq. (3) [4]:

$$Y = B_0 + \sum_{i=1}^{q} B_i X_i + \sum_{i=1}^{q} B_{ii} X_i^2 + \sum_{i}\sum_{j} B_{ij} X_i X_j \tag{3}$$

Where X_i, X_j, and X_q are designated to the operating variables; B_o, B_i, and B_{ij} are the regression coefficients, and Y is the studied response.

Several models were used to study the behavior of adsorption such as Langmuir, Freundlich, and Temkin isotherm models (Eqs. 4 to 7), which are the commonly used models for studying adsorption involving the evident interaction between the MCM-41 NPs adsorbent and COD pollutant [26, 27].

$$\frac{C_{eq}}{q_e} = \frac{1}{q_m} C_{eq} + \frac{1}{K_L q_m} \tag{4}$$

Where q_m and K_L are the adsorption capacity (mg/g) and adsorption energy (L/mg), respectively.

The favorability of this model could be obtained from using the RL indicator (Eq. 5), which characterized the Langmuir adsorption isotherm to be favorable, unfavorable, irreversible, or linear when R_L is less, more, or equal to 0 or 1, respectively [27].

$$R_L = \frac{1}{1+K_L C_i} \tag{5}$$

Where C_0 is the highest initial dye concentration (mg/L). The Freundlich linear form is given in Eq. 6:

$$\ln q_e = \ln K_f + \frac{1}{n} ln\ C_{eq} \tag{6}$$

Where n is the adsorption intensity, and Kf is the adsorption capacity of the sorbent. The Temkin model could be represented as follows:

$$q_e = \frac{RT}{b} \ln K_T + \frac{RT}{b} \ln C_e \tag{7}$$

Where K_T and b are the Temkin isotherm constant (L/mg) and the heat of adsorption (J/mol), respectively. R and T are the universal gas constant (kJ mol^{-1} K−1) and absolute temperature (K). Assuming that B=RT/b.

RESULTS AND DISCUSSION

Table 3 shows the obtained findings of the experimental values of COD removal efficiency in the batch process. The observed COD removal values ranged from 55.02% to 93.77%.

TABLE 3. Results of the studied variables

Run	**X_1: NP dosage (g)**	**X_2: Reaction time (min)**	**X_3: Stirring speed (rpm)**	**Y_{CODR} %**
1	0.04	30	130	81.05
2	0.08	30	130	84.01
3	0.04	30	220	82.88
4	0.08	30	220	86.50
5	0.04	96	130	84.82
6	0.08	96	130	82.10
7	0.04	96	220	70.04
8	0.08	96	220	92.86
9	0.02	62	175	70.43
10	0.1	62	175	55.02
11	0.06	62	100	85.60
12	0.06	62	250	83.77
13	0.06	5	175	75.60
14	0.06	120	175	82.49
15	0.06	62	175	93.00
16	0.06	62	175	93.77

continued on next page.

TABLE 3. continued

Run	X1: NP dosage (g)	X2: Reaction time (min)	X3: Stirring speed (rpm)	YCODR %
17	0.06	62	175	91.98
18	0.06	62	175	93.00
19	0.06	62	175	92.61

REGRESSION MODEL

Based on the findings listed in Table 3, the regression equation (Eq. 3) had developed with regard to real factors showing the interaction between the studied variables and the COD removal efficiency (Eq. 8):

$$\text{COD removal \% (YCODR)} = -90.7 + 2442\,X_1 + 0.502\,X_2 + 1.160\,X_3 - 27180\,X_1^2 - 0.00420\,X_2^2 - 0.00383\,X_3^2 + 2.72\,X_1\,X_2 + 3.64\,X_1\,X_3 - 0.00070\,X_2\,X_3 \quad (8)$$

The Pareto chart shown in Fig. 1 revealed that the square effect of NPs dosage and reaction time is insignificant because they exceed the Pareto parameter (2.306) of signification. Therefore, their effect will omit, and Eq. 7 will be presented as follows:

$$\text{COD removal\% (YCODR)} = -90.7 + 2442\,X_1 + 0.502\,X_2 + 1.160\,X_3 - 0.00383\,X_3^2 + 2.72\,X_1\,X_2 + 3.64\,X_1\,X_3 - 0.00070\,X_2\,X_3 \;(R^2{=}83\%) \quad (9)$$

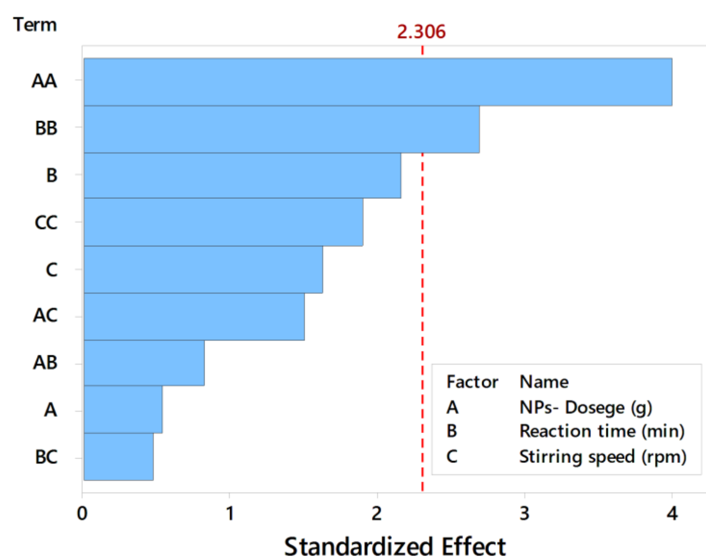

FIGURE 1. Pareto chart of the studied variables ($\acute{\alpha} = 0.05$)

EFFECT OF NP DOSAGE

As shown in Fig. 2, the removal efficiency of COD pollutant increased sharply with the increase of the dosage of MCM-41 nanoparticles. This observation indicates that the activated sites inside the nanoparticles assist adsorption occurring between MCM-41 nanoparticles and COD pollutant. However, the excessive addition of nanoparticles will tend to decrease the treatment efficiency because of the agglomeration of nanoparticles, and then the activated sites will minimize, which will cause the fast decrement of COD removal efficiency. This result is consistent with our previous work [15] and Peymanfar et. al. 2018 [20].

The mathematical relation (Eq. 10) between the studied response and the variable of MCM-41 nanoparticles in the case of mean values of other operating variables is as follows:

$$\text{COD removal\%} = 2937\ X_1 - 23753\ X_1^2\text{: } R^2 = 99.2\%. \quad (10)$$

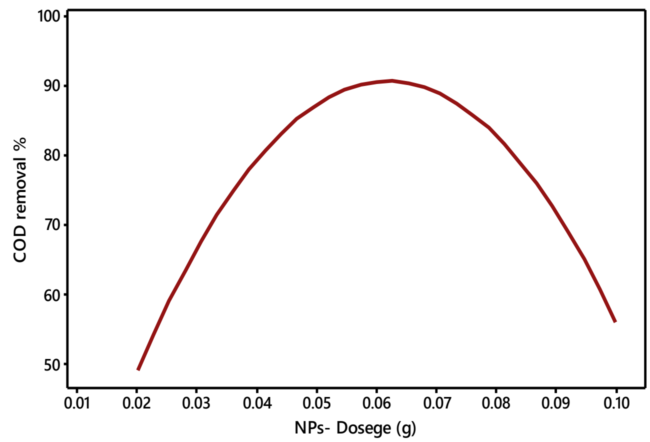

FIGURE 2. COD removal from refinery wastewater vs. NP dosage at a reaction time of 62 min and stirring speed of 175 rpm

EFFECT OF REACTION TIME

Fig. 3 shows the direct relation between the COD removal response and reaction time until the mean value of the studied variable of the reaction time was reached, which was then slightly minimized. The decrement of removal efficiency may refer to desorption that occurred when the activated area of pores inside the nanoparticles used had become saturated. This finding is consistent with [28]. Eq. 11 shows the correlation between the COD removal efficiency and reaction time:

$$\text{COD removal \%} = 2.537\ X_2 - 0.017\ X_2^2\text{: } R^2 = 93.3\%. \quad (11)$$

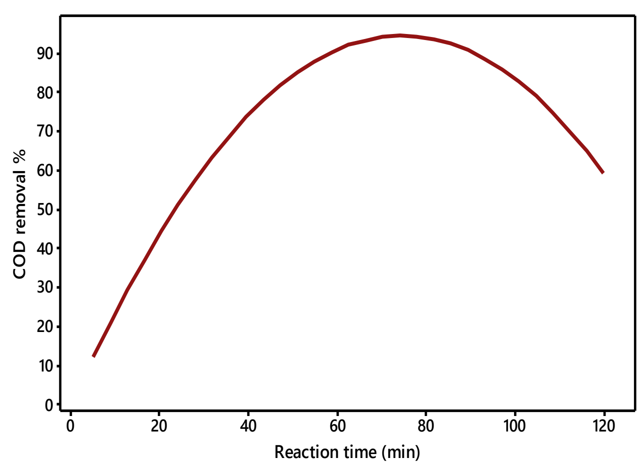

FIGURE 3. COD removal from refinery wastewater vs. reaction time at NP dosage of 0.06 g and stirring speed of 175 rpm

EFFECT OF STIRRING SPEED

The behavior of COD removal depending on the variation of the stirring speed at the mean values of NP dosage and reaction time is shown in Fig. 4. The slight increase in agitation speed had a positive effect on the removal efficiency until it reaches approximately 175 rpm, and then the removal process tends to minimize with the increase of the mixing speed because the interaction between nanoparticles and pollutants will become more difficult as the agitation speed increases, and then the adsorption process will be less efficient [15, 28]. The mathematical relation between COD removal response and the mixing speed is presented in Eq. 12.

$$\text{COD removal \%} = 0.961\ X_3 - 0.003\ X_3^2\text{: } R^2 = 98.3\% \quad (12)$$

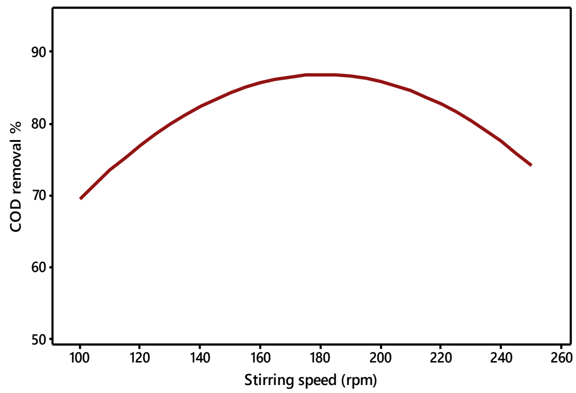

FIGURE 4. COD removal from refinery wastewater vs. stirring speed at NP dosage of 0.06 g and reaction time of 62 min

ADSORPTION STUDY

The results obtained from the study of Langmuir, Freundlich, and Temkin isotherm models (Fig. 5 and Table 4) revealed that the Langmuir isotherm model is more fitted than the other two models. The sorption sites have equal and favorable affinity to COD pollutants, where the maximum adsorption capacity is 333.33 mg/g, which corresponds to a complete monolayer coverage on the surface of the MCM-41 nanoparticle. Moreover, adsorption was physical based on the Freundlich isotherm constant (n>1). The heat of adsorption decreases with the increase of the active area along the reaction time because of the indirect nanoparticle–COD interactions and uniform distribution of the binding energy.

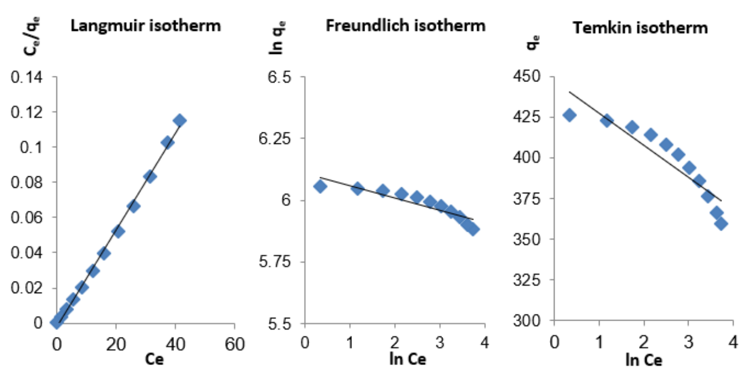

FIGURE 5. Adsorption isotherm models for COD removal from refinery wastewater (257 ppm COD/L, 0.06 g NP dosage, 175 rpm)

TABLE 4. Specifications of adsorption isotherm models (257 ppm COD/L, 0.06 g NP dosage, 175 rpm)

Models	Parameters	Correlations
Langmuir	q_{max} = 333.33 mg/g b= 1.304 L/mg R_L= 0.003 R^2 = 0.998	y = 0.0028 x − 0.002
Freundlich	K_F= 449.90 mg/g n>1 R^2 = 0.822	y = −0.0049 x + 6.109
Temkin	B=19.666 kJ/mol K_T=0.0001 L/mg R^2 = 0.837	y = −19.666 x + 447.09

OPTIMIZATION

The optimization results are shown in Fig. 6, where the composite desirability (D) is equal to 1, which indicates an accurate measurement of the optimum values of the operating variables. This result indicates the best performance of the refinery wastewater treatment process by using MCM-41 nanoparticles. The optimum values of the operating variables (Fig. 9) are 0.06 g, 64.24 min, and 174.24 rpm for NPs dosage, reaction time, and stirring speed, respectively, which produce 99.58% of COD removal efficiency.

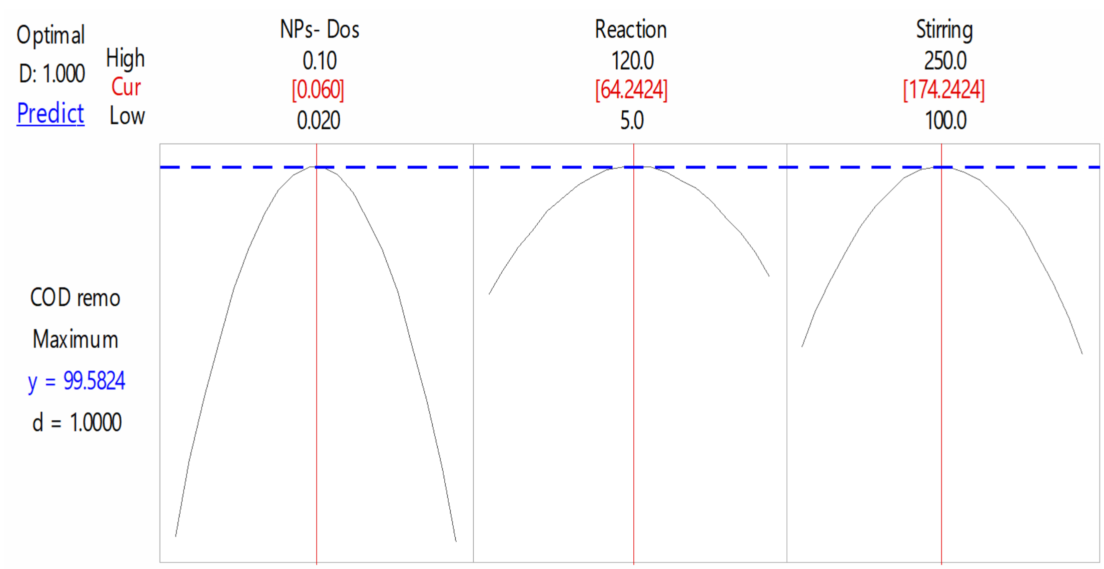

FIGURE 6. Optimization determination of the studied responses

CONCLUSIONS

Millions of cubic meters of wastewater are discharged every year to the environment. A high percentage of these quantities are released from crude oil refinery plants. Many conventional treatment methods are used to eliminate pollutants from oily wastewater, but they suffer from different disadvantages such as cost and maintenance. The present work uses nanotechnology by using MCM-41 nanoparticles to remove COD from real oily wastewater discharged from Al-Dora refinery plant. The results indicate the effective use of this type of nanoparticles under the effect of their dosage, reaction time, and stirring speed. The optimum values of the operating variables are 0.06 g, 64.24 min, and 174.24 rpm for NP dosage, reaction time, and stirring speed, respectively, which produce 99.58% of COD removal efficiency. The adsorption mechanism is consistent with the Langmuir isotherm model, which is more fitted than Freundlich and Temkin isotherm models. Moreover, the adsorption is physical and favorable.

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