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Treatment of Crude Oil Wastewater Using Low-cost Modified Jordanian Kaolin Sorbent

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Abstract

This work uses batch experiments to investigate the adsorption capacity of the low-cost modified Kaolin adsorbent to remove crude oil from refinery wastewater. Kaolin modification is carried out using lauric acid. Equilibrium and Kinetic analysis were studied to investigate the adsorption mechanism. The experimental work has conducted at 60^0C; at several contact times, different adsorbent amounts to find the optimum conditions. Results show an increase in the adsorption rate with increasing contact time. The maximum removal efficiency is obtained at about 80 min. No significant change in the equilibrium concentrations is achieved when increasing time after adsorption equilibrium is reached. Three equilibrium models were studied, Langmuir, Freundlich, and Temkin isotherms. Results show that the Langmuir model isotherm fits well with the experimental data with R^2=0.997; the R_L value is less than one, suggesting that the crude oil adsorption onto modified Kaolin is favourable. Furthermore, three kinetic models are studied to understand the mechanism of adsorption. Among these models, pseudo-second-order models fitted the experimental data perfectly.

Paper type: Research paper

Keywords: Wastewater, crude oil wastewater, Kaolin, batch, equilibrium, isotherm kinetics**.**

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Introduction

Human activities, like Industries, are the vast effluents' primary source. Several industries produce oil wastewater, including metallurgical, transportation, petrochemical, food processing and petroleum refineries (Keramati and Ayati, 2019). Oil spillage and evacuation are significant threats to human health and ecosystems. Thus, oil wastewater treatment is essential for a sustainable environment (Liang *et al.*, 2019). Oil emulsified in water at a range of 100–1000ppm is considered a significant water pollutant. (Yatsufusa *et al.*, 2009). Reports suggest that the maximum oil concentration allowed for a safe environment flowing is only about 5−40mg/L. The oil wastewater is composed of dispersed oil 20–150µm, free oil >150µm, and emulsified oil <20µm (Tanudjaja *et al.*, 2019). Different methods have been used for rapid oil wastewater treatment (Wang *et al.*, 2013), such as oil skimmers, booms (Yu *et al.*, 2014) bioremediation (Swannell *et al.*, 1996) controlled burning (Gao *et al.*, 2017), physical diffusion (Cheng *et al.*, 2011), solidifiers (Basak *et al.*, 2012), dispersants (Kujawinski *et al.*, 2011), membranes technology (Hudaib *et al.*,2022; Howarter *et al.*, 2009) and adsorption (Ji *et al.*, 2009; Adebajo *et al.*, 2003; Chai *et al.*, 2020; Caponi *et al.*,2017). Adsorption has been considered the most low-cost, available, and recyclable for oil/water removal (Stolz *et al.*, 2016; Chu *et al.*, 2012; He *et al.*, 2013). Several types of adsorbent materials have been investigated for pollutant removal. Clay minerals are one promising material that has been studied for their adsorption capacity to treat different types of pollutants like oil wastewater.

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Kaolin is a clay mineral composed of two layers of tetrahedral oxygen silicate (SiO_4) and octahedral alumina. It has excellent chemical stability, low expansion characteristics, and good exchange capacity (Uddin *et al.*, 2016; Emam *et al.*, 2017). Modification is a method used to improve the Kaolin quality by adding some essential materials to the adsorbent to increase its ability to remove pollutants; Kaolin is an excellent absorbent potential for removing crude oil wastewater as it is safe, cheap, and it is highly available (Emam *et al.*, 2017; Mustapha *et al.*,2019; Panda *et al.*,2010). This work-modified Kaolin was investigated as a potential low-cost adsorbent for crude oil wastewater treatment. A batch adsorption study was conducted to investigate the potential capacity of Kaolin to remove crude oil. The equilibrium and kinetic data obtained were analyzed.

1 Materials and Methods

1.1 Materials

Lauric Acid was purchased from the local chemical store, methanol and hexane were purchased from Merck Company, Kaolin stones were collected from the Mahes area in Salt, Jordan, and Crude oil samples were from Jordan Refinery Company.

1.1.2 Modified Kaolin preparation

First, Kaolin was grinding and sieved to a specific size of about 2.7mm, and then washed with distilled water carefully about 4-5 times and dried in an oven. A 150ml of methanol was added to 0.1 molarity of lauric acid; the mixture was stirred vigorously at 130˚C for approximately 15 minutes until the lauric acid was dissolved entirely. The Kaolin was added to the mixture at the same temperature for about 6 hours; after that Kaolin was filtered from the solution, and hexane drops were added to remove the excess amount of lauric acid; finally, Kaolin was dried in the oven at 200ºC, as shown in **Figure 1**, then they were kept in a closed clean bottle.

1.1.3 Crude oil wastewater

Crude oil used in this experiment was collected from the refinery, 0.1g of crude oil was dispersed in 1 litre of distilled water using surfactants to assure oil homogeneity in water, and the resultant crude oil wastewater viscosity is 0.89cP and the pH is 8.2.

1.1.4 Kaolin characterization

Raw and modified kaolin was analyzed using Fourier Transform Infrared Spectroscopy (FTIR) instrument; model PerkinElmer, Spectrum Two L1600400, FT-IR/DTGS. The chemical composition of the raw kaolin used as an adsorbent in this study was analyzed using Thermo Fisher XRF Instrument, model ARL™ SMS-Omega.

Fig.1 Sample of Kaolin.

1.2 Batch adsorption techniques

The adsorption study was conducted using batch equilibrium. A group of batch experiments was studied using different modified Kaolin adsorbent amounts of 1.5 to 4g, and from 1 to 400ppm for crude oil wastewater at 60ºC. the mixture was stirred at 180 rpm for 5 hours to assure reaching the equilibrium state, the solution sample parameters like pH and temperature were kept constant. Kaolin's capacity for removing oil from wastewater and the adsorbent dosage effect was studied. Each experiment was carried out twice, and the results were averaged. The resultant solution was tested using a UV-Vis spectrophotometer (Varian Cary 100) at a maximum wavelength of 358 nm.

2 Results and Discussion

2.1 Raw Kaolin characterization (XRF)

Raw kaolin mineral investigations were carried out using X-ray fluorescence spectroscopy analysis (XRF) to investigate the chemical compositions. The elements obtained were expressed as oxides, as presented in **Table 1**. The main composition of the Kaolin is silica at about 67.35wt%, iron oxides (hematite) at about 17.48wt%, and alumina at about 8.62wt%.

Table 1 XRF of raw Kaolin samples.

Kaolin Compositions	Al_2O_3	CaO	Fe_2O_3	K_2O	MgO	SO_3	SiO_2	Loss of ignition
Wt (%)	8.62	0.65	17.48	0.27	0.22	0.14	67.35	5.27

2.2 FTIR Analysis

The Fourier transform infrared spectroscopies (FTIR) spectra of raw Kaolin and modified Kaolin are shown in **Figure 2**. Raw Kaolin functional groups are listed in **Table 2**. After Kaolin modification, a new peak at 1643 cm^{-1} confirms the presence of C=O stretching vibration, suggests the grafting of lauric acid to kaolin, and the interactions between Kaolin and lauric acid. The spectra of lauric acid showed characteristic peaks for the stretching vibration of $-CH_2$ and $-CH_3$ groups at 2936.3 cm^{-1} and 2890.7 cm^{-1} (Jiang *et al.*, 2018). The spectra of modified Kaolin presented some little shifts of prominent peaks compared with raw Kaolin. Additionally, there are clear

new peaks in the spectra of modified kaolin. These changes explain that there is any reaction between Kaolin and lauric acid, which justifies the adsorption feasibility (El Mouhri *et al.*, 2020).

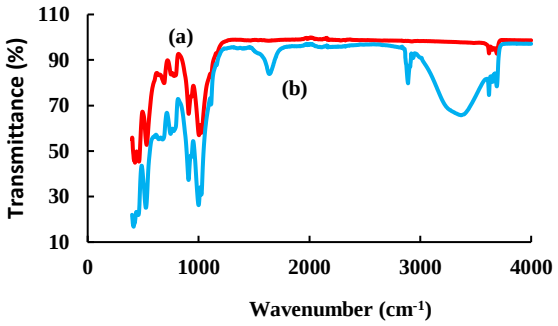

Fig.2 FTIR spectra of (a) raw Kaolin and (b) modified Kaolin.

Table 2 Raw kaolin FTIR spectra (Panda *et al.*,2010; Belver *et al.*,2002; Chai *et al.*,2020; Peter *et al.*,2018)

Functional group	Wave number (cm^{-1})
OH-	3620
Si–O	1057.71
Si-OH	912.76
Si-O-Al	782.3
Si–O–Si	691.5
Al-O	566

2.3 Equilibrium investigation

The adsorption capacity at equilibrium q_e and the equilibrium concentration C_e for the adsorption of crude oil from the wastewater by Kaolin adsorbent at 60°C are shown in **Figure 3**.

The equilibrium data are shown in Fig. 3, which resulted from batch experiments after mixing with different modified Kaolin masses ranging from 1 to 4g with the crude oil wastewater samples. To ensure reaching the equilibrium state, Kaolin was added to the crude oil wastewater, and well stirred in a mixer at 180rpm for 6 hours. The temperature was kept constant at 60°C using the shaker water bath. Then, the Kaolin was separated by filtration, and the amount of crude oil residue was determined. Fig. 3 shows that the crude oil adsorbed per adsorbent at equilibrium. It revealed that the equilibrium adsorption capacity of Kaolin q_e decreased with increasing the concentration C_e of the crude oil wastewater at equilibrium.

2.3.1 Langmuir adsorption isotherm

The Langmuir model analyses the monolayer of the homogeneous adsorption process. The model reveals the same adsorption sites and, thus, has identical adsorption energy (Doğan *et al.*, 2000). The standard linearized Langmuir model is given by equation (1).

$$\frac{C_e}{q_e} = \frac{1}{K_a Q_m} + \frac{C_e}{Q_m} \quad (1)$$

where C_e is the equilibrium concentration of the crude oil, and q_e is the amount of the crude oil adsorbed per unit mass of adsorbent at the equilibrium. The Langmuir equilibrium parameters Q_m and K_a illustrate the highest adsorption gained at equilibrium and the binding energy of the adsorbate to the adsorbent, respectively (Tran *et al.*, 2017). The linear plot of the Langmuir model is shown in **Figure 4**. As shown in Fig. 4 the R^2=0.997 calculated is fitted with the experimental data with a positive slope, the obtained values of Q_m is equal to 3.47x10^{-5}, and k_a=862.6.

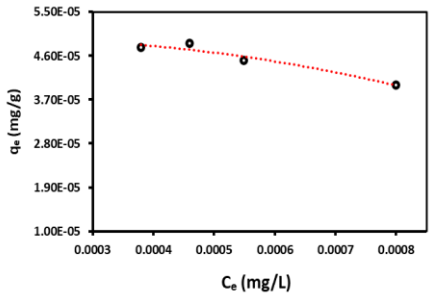

Fig.3 The equilibrium data conducted for modified Kaolin adsorption.

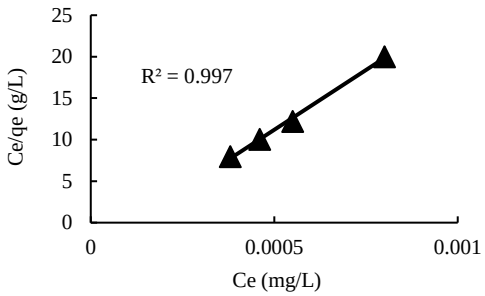

Fig. 4 Langmuir isotherm at 60°C.

2.3.2 Freundlich adsorption isotherm

Freundlich isotherm has applied for non-ideal heterogeneous adsorption using an empirical approach from the experimental results for heterogeneous non-ideal adsorption. Freundlich equation is represented by equation (2).

$$\log q_e = log\ K_f + \frac{1}{n} log\ C_e \quad (2)$$

where K_f is the Freundlich isotherm constant illustrating the amount of the crude oil that adsorbed. When K_f value is high, then the adsorbent has a higher adsorption capacity. $1/n$ showed the intensity of the crude oil adsorbed on modified Kaolin. Moreover, $1/n$ value explained the adsorption process type. If its value is below 1, it is suggested that a monolayer adsorption type, but if it is above 1, a postulate of cooperative adsorption. The Freundlich isotherm is shown in **Figure 5** and **Table 3**. The obtained K_f and *1/n* values from the straight line's equation are 7.61x10^{-6}mg$^{1-1/n}$ L$^{1/n}$ g^{-1}, and -0.2338, respectively. *1/n* value leads to the adsorption type (Altaher *et al.*, 2015). When it is less than 1, the adsorption process can be considered as a chemical one. However, when it is bigger than 1, as in this adsorption process which is -0.2338, suggesting a chemical adsorption.

2.3.3 Temkin adsorption isotherm

The Temkin isotherm model postulates that the interaction between the adsorbent and sorbent can be estimated by linear plotting, a decrease in the adsorption heat is noticed. The binding energies can be explained that the adsorption mechanisms being distributed uniformly. Temkin isotherm is given according to equations (3) and (4) (Hei Ing *et al.*, 2015).

$$q_e = B_1 \ln a_t + B_1 \ln C_e \quad (3)$$

$$B_1 = RT/b_t \quad (4)$$

Where b_t is a constant for the heat of sorption, and a_t is the Temkin isotherm constant. Temkin model data is plotted in **Figure 6**; results revealed a good correlation coefficient, with R^2=0.971, which means a good experimental data fitting.

The adsorption energy for the Temkin model, b_t=-2.47x10^3 is negative for crude oil adsorption, indicating that the adsorption is highly endothermic.

Table 3 summarizes the three equilibrium models tested on the experimental data. Results showed that Lanngimure is the best-fit model for the adsorption of crude oil on Kaolin with the highest correlation coefficient at R^2=0.997. To estimate the affinity between the adsorbent and adsorbate using the equilibrium parameter R_L expressed in the following equation (Malik *et al.*, 2004).

$$R_L = \frac{1}{(1+K_a C_i)} \quad (5)$$

Where K_a is the Langmuir constant and C_i is the initial concentration of the crude oil in wastewater. The value of R_L suggested the type of Langmuir isotherm to be irreversible if R_L=0, linear if R_L=1, unfavourable if R_L>1, or favourable if 0<RL<1) (McKay *et al.*, 1982). The R_L value for this study was found equal to 0.536, suggesting that crude oil adsorption on modified Kaolin is favourable.

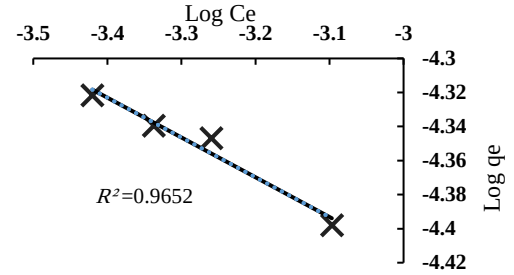

Fig. 5 Freundlich isotherm at 60 °C

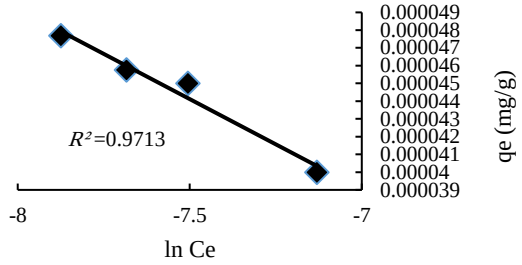

Fig. 6 Temkin isotherm fitted the equilibrium adsorption data.

2.4 Adsorption kinetics

2.4.1 Pseudo-first order

Pseudo–first-order kinetic model assumed that the solute sorption rate is proportional with time-related to the difference between the saturation concentration of sorbent and the amount of sorbent taken with time. Usually, the experimental data follows the pseudo-first-order rate equation; thus, the controlling step is diffusion through the interface. The pseudo-first-order model given by equation (6) (Chiou *et al.*, 2003).

$$log(q_e - q_t) = log(q_e) - \left(\frac{k_1}{2.303}\right) t \quad (6)$$

where q_e and q_t are the total amounts of the crude oil in wastewater adsorbed at equilibrium and at time t, and k_1 is the first-order rate constant. **Figure 7** presents the linear behaviour for the Pseudo-first order model. The rate constant, k_1 is given in min^{-1}, and the correlation coefficient R^2 is 0.9749.

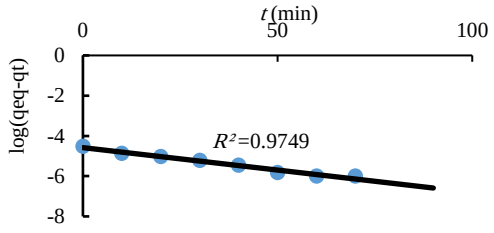

Fig. 7 Pseudo-first-order model.

Table 3 Equilibrium adsorption isotherm parameters.

Langmuir isotherm	Freundlich isotherm	Temkin isotherm
R^2=0.997	R^2=0.9697	R^2=0.9713
K_a=862.6 (L/mg)	K_f=7.61x10^{-5} (mg $^{1-1/n}$ L $^{1/n}$ g^{-1})	b_t=-2.47x10^6 (J/mol)
Q_m=3.47x10^{-5}	$1/n$=-0.2338	a_t=20.1 (L/mg)
	n=-4.28	

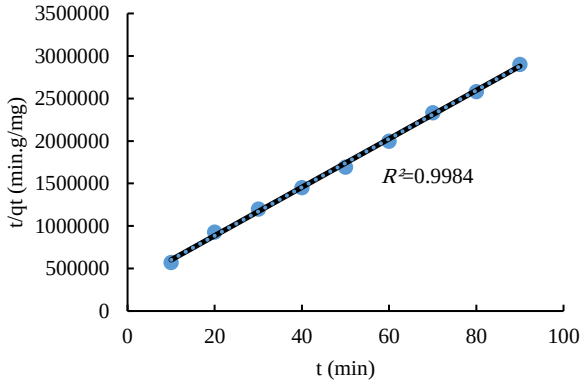

Fig. 8 Pseudo-second order model.

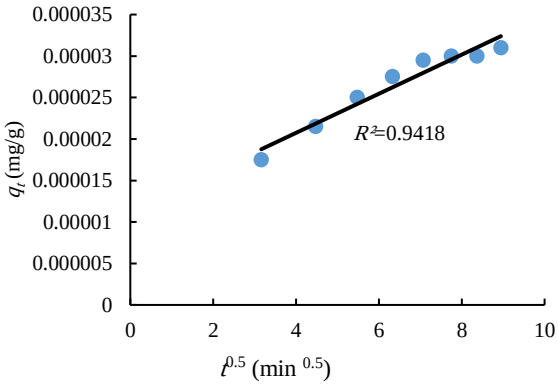

Fig. 9 Intraparticle diffusion model.

2.4.2 Pseudo-second-order kinetic model

This model is indicating that chemical sorption is the limiting step rate. The linear form of the pseudo-second-order adsorption is presented in equation (7) (Ho Y-S, 2006).

$$\frac{t}{q_t} = \frac{1}{k_2\, q_e^2} + \frac{1}{q_e}\, t \qquad (7)$$

where k_2 is the pseudo-second-order rate constant. **Figure 8** shows the linear plot of the pseudo-second-order kinetic model. The resultant rate constant, k_2 and correlation coefficient, R^2 are 2.56x10^{3}g/mg.min, and 0.9984, respectively. Fig.8 shows that the pseudo-second-order kinetic model was well-fitted to the experimental data. Moreover, the Pseudo-first order is assigned for more physical adsorption than chemical (Ngah *et al.*, 2005). Thus, the pseudo-second-order model better describes the crude oil wastewater adsorption on Kaolin than the pseudo-first-order model.

2.4.3 Intraparticle diffusion model

The intraparticle diffusion linear equation is presented in equation (8) (Santhi *et al.*, 2010).

$$q_t = k_{id} t^{0.5} + C \qquad (8)$$

where k_{id} (mg/g.min$^{-0.5}$) is the intraparticle diffusion model rate constant, and C is a constant expressing the boundary layer's thickness. Generally, the boundary layer effect is greater for a large value of C. **Table 4** shows the three kinetic models plotted for the adsorption experiment. The high correlation coefficient R^2=0.9984, reveals that pseudo-second-order well fitted the experimental measurement. Hence, pseudo-second-order might be a limiting process in the adsorption process.

Table 4 Adsorption kinetics model parameters.

Pseudo-first-order	Pseudo-second-order	Intraparticle diffusion
R^2 =0.9749	R^2=0.9984	R^2=0.9418
k_1= 0.0514 (min^{-1})	k_2=2.56x10^3 (g/mg.min)	k_{id}=2x10^{-6} (mg/g.min$^{-0.5}$)

Conclusions

Jordanian Kaolin is cost-effective and locally available adsorbent has been presented in this study. Kaolin was modified with lauric acid and used to treat crude oil wastewater. Results showed that the adsorption of crude oil onto modified Kaolin increases with the amount of adsorbent, and the maximum removal efficiency was obtained within about 80min. Three equilibrium adsorption models were fitted the equilibrium data; Langmuir, Temkin, and Freundlich isotherms. Langmuir model showed the best fit of experimental data. Moreover, the R_L value was less than one, suggesting that the crude oil adsorption onto modified Kaolin is favourable. Three kinetic models were studied to understand the adsorption mechanism. Among these models, pseudo-second-order models fitted the experimental data perfectly.

Nomenclature

C_e =equilibrium concentration of crude oil [mg/L]

q_e	=adsorption capacity at equilibrium	[mg/g]
K_a	=binding energy of the adsorbate to the adsorbent	[L/mg]
K_f	=Freundlich isotherm constant	[mg $^{1-1/n}$ L $^{1/n}$ g^{-1}]
a_t	=Temkin isotherm constant	[L/mg]
b_t	=adsorption energy for the Temkin model	[J/mol]
k_1	=the first-order rate constant	[min^{-1}]
k_2	=the pseudo-second-order rate constant	[g mg^{-1} min^{-1}]
k_{id}	=the intraparticle diffusion model rate constant,	[mg/g.min$^{-0.5}$]

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