J Nanostruct 16(3): 3027-3038, Summer 2026

RESEARCH PAPER

Activation of Flint Clay Surface and Study of its Adsorption Efficiency for Methyl Orange Dye

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ARTICLE INFO

Article History:
Received 24 February 2026
Accepted 27 April 2026
Published 01 July 2026

Keywords:
Acidic activation
Adsorption
Flint clay
Isotherm
Methyl orange

ABSTRACT

This article presents an improvement in the properties of environmentally friendly, inexpensive, and readily available surfaces for use in addressing a significant environmental problem: pollution from dyes generated by various industrial processes. The surface of Iraqi flint clay was acidically activated and used to adsorb methyl orange dye from its aqueous solutions. The clay surface was characterized before and after acid activation using several techniques, including Fourier transform infrared spectroscopy, X-ray diffraction spectroscopy, X-ray disperse energy spectroscopy, and field-emission scanning electron microscopy. The adsorption of methyl orange dye using flint clay before and after activation was performed in batch system under controlled operating conditions (weight of adsorbent surface, contact time, initial dye concentration, and temperature). The amount of adsorption of methyl orange dye was found to be higher at higher temperatures, with the q_e value for the raw clay being 45 mg/g and for the activated clay q_e value of 30 mg/g. The process data were analyzed kinetically and found to follow the pseudo-second order kinetic model, while the process data were analyzed isothermically and found to follow the Freundlich isotherm model. The thermodynamic functions of the adsorption process were assigned and found to be a non-spontaneous, endothermic, and random process on the raw clay, while it became more spontaneous, random, and endothermic, reflecting the success of the acid activation process in improving the efficiency of the Iraqi flint clay surface in removing the methyl orange dye from its aqueous solutions.

How to cite this article
Abd S., Radhi I., Abbas A. Activation of Flint Clay Surface and Study of its Adsorption Efficiency for Methyl Orange Dye. J Nanostruct, 2026; 16(3):3027-3038. DOI: 10.22052/JNS.2026.03.003

INTRODUCTION

Water contaminants that make it unfit for human consumption include organic compounds, inorganic, microorganisms, and chemical waste. Ten to fifteen percent of the more than 700,000 tons of dyes produced worldwide each year are released into the environment through wastewater. Strongly colored dye concentrations of 10–200 mg/L are frequently found in industrial wastewater, which has detrimental effects on water quality [1]. Contaminated healthcare waste is usually produced by all facets of medical facilities.

Since water is present in all forms of biomass, its importance to life is enormous. However, by introducing different pollutants into aquatic ecosystems, human activity is reducing the amount

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of clean water available. Resource extraction, oil spills, agricultural practices, nuclear waste, and industrial waste are particularly some of the sources of water pollution. This industry generates a significant amount of effluent and consumes substantial amounts of water, particularly during the wet processing of materials, which presents environmental challenges. The effluent frequently contains hazardous dyes that are bad for the environment and people, which has led to a lot of research being done to remove these pollutants from water bodies [2,3].

Chemical dyes are colored organic or inorganic substances that acquire their color from their molecular structure, which contains chromophores (color-carrying groups) (color auxiliaries), and they function they absorb certain wavelengths of light in the visible spectrum and are classified into many types according to their composition and uses, such as acidic, cationic, azo, and reactive dyes. They are used in textiles, inks, plastics, and food, with significant development since the discovery of the first synthetic dye in the nineteenth century. byinfectious diseases and may have detrimental effects on public health [4,5].

Multiple decontamination techniques are used, including filtration, chemical oxidation, chemical coagulation, precipitation, electrochemical decontamination, electrocoagulation, photocatalytic decomposition, ultrasonic and biological decomposition, and adsorption, to remove dyes [6]. When examining the strengths and weaknesses of each technique, adsorption might be a good idea. Numerous research organizations have published reports about it widely—adsorption is preferred for removing dyes from aqueous environments. Adsorption is one of the most important and widely used techniques for removing pollutants from their aqueous solutions due to its high efficiency and the simplicity of the technology used compared to other methods, as well as its low economic cost. The adsorption process is considered an attractive alternative for treating polluted water, especially if the adsorbent surface is natural, inexpensive, readily available, environmentally friendly, and does not require additional treatment steps. One of the first steps that must be available for the effective adsorption process is to obtain adsorbent surfaces that are characterized by high selectivity, large capacity, and a large surface area, and they must also be inexpensive and available [7]. But it also has drawbacks, like the toxicity of spent adsorbents and the need for chemicals for desorption, which suggests that pollution is transferred rather than eliminated [8-11]. Methyl Orange (MO) is a common pH indicator in the textile industry. However, these approaches frequently have issues with time consumption, high costs, and complexity. Because of its ease of use, affordability, and insensitivity to toxins, adsorption is preferred for the removal of MO.

Abdel Fattah Mohamed Al-Kharrar and others studied the extraction rate and adsorption isotherms of methyl orange and crystal violet dyes on the surface of dry peanut shells and prepared charcoal. A UV-Vis spectrometer was used to estimate the dye concentration before and after adsorption. The results showed that the adsorption isotherms were similar to those of Freundlich. The (s) type, according to Giles' classification, is inconsistent with the Langmuir equation. It was also found that the adsorption or removal rate of the studied dyes increases with the amount of adsorbent. The effect of temperature on adsorption was also investigated [12].

Farah Amalina and others studied the adsorption of orang-methyl ions on kaolinite clay from both thermodynamic and kinetic perspectives. The process involved both adsorption and malfunction, but in some instances, release occurred. Frondissement constants and their intensity were calculated. Demonstrate the calculation of thermodynamic coefficients, both systematic and non-automatic, within a temperature range of 298-328 K. Three dynamic computational models were used: the constant effect, the constant..., the adsorption coefficient, and the removal coefficient [13].

This study investigated the use of modified bentonite as a low-cost and effective adsorbent for removing basic and acidic dyes (methylene blue MB and methyl orange MO) from wastewater using two experimental methods: batch and continuous. The bentonite used in this study was local (calcium type) bentonite in its natural form, modified by replacing the replaceable calcium ions in natural bentonite with a basic surfactant (cetyl trimethyl ammonium bromide, CTAB). The study then investigated its adsorption behavior as an effective adsorbent for dye removal by converting its hydrophilic to organophilic nature [14].

Another study aimed to test the efficiency of modified red clay as an adsorbent material, given its abundance in the composition of Iraqi soil, in removing textile dyes and heavy metals. It also aimed to study the adsorption of dyes (Methyl blue (M.B) and Methyle orange (M.O) on the surfaces of modified red clay as an adsorbent material, and to study the conditions affecting the efficiency of the adsorption process for dyes M.B. and M.O. in terms of the effect of the acidity function, the concentration of the adsorbent and the adsorbent material, temperature, and time, and to adjust them to give the highest adsorption of the dye [15]. The primary focus of this study's novelty lies in the examination of a new adsorbent used for the removal of MO from aqueous solutions by activating the surface of the flint clay.

MATERIALS AND METHODS

Preparation of Clay Powder

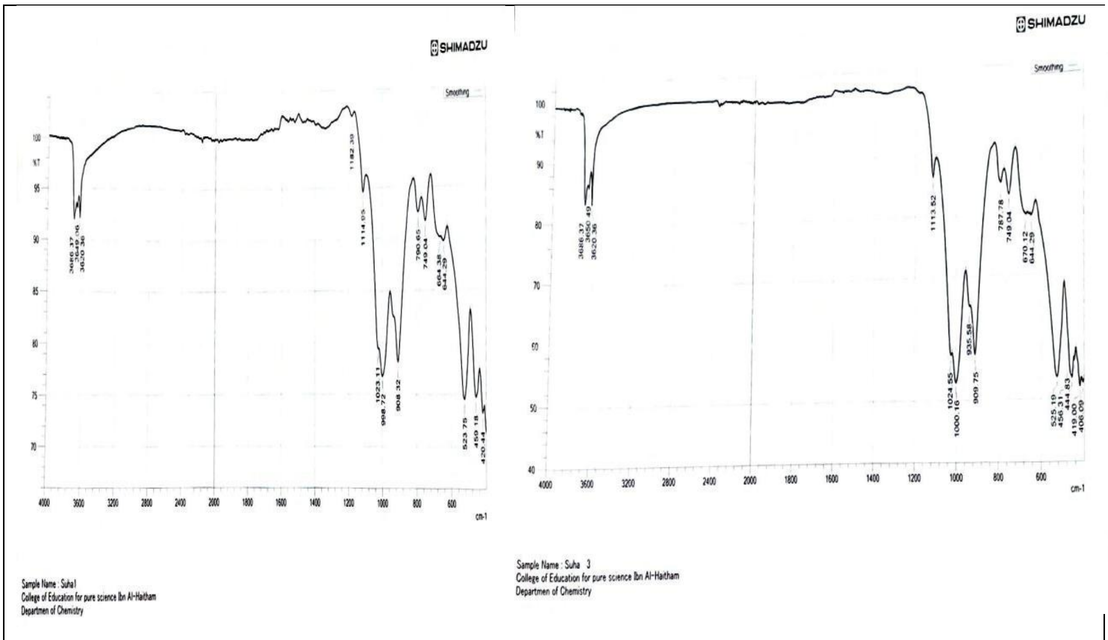
The flint clay was obtained from the Iraqi Geological Survey Company. The surface of the flint clay was prepared by washing it with a sufficient quantity of distilled water several times to remove foreign materials and water-soluble materials. It was then dried in an oven (Daihan Labtech, Korea) at a temperature of 100 °C for 24 hours, then sieved and stored. The particle size of the flint clay surface was chosen at 75 µm in the experiments related to this study. The raw flint surface was characterized after preparation using (FTIR, XRD, EDX, SEM) techniques.

Chemical Activation of Flint Clay Surface

The surface of flint clay was chemically activated using 600 ml of 1M hydrochloric acid (BDH) in a 1L beaker. 30 g of dried flint was added to the acid solution with continuous stirring at 50 °C for three hours. The mixture was left overnight to remove the acid and was washed with distilled water, stirring only for half an hour. This process was repeated several times until the pH of the solution reached 7. The resulting product was then dried at 105°C for four hours and heated to 300°C. The activated clay surface was characterized after preparation using FTIR, XRD (Shimadzu, Japan), SEM (FE-SEM ZEISS model: Sigma VP-UK). and EDX (EDX Oxford instruments, UK) techniques.

Adsorption Test

Adsorption experiments were conducted by placing a specific weight of flint clay surface, both before and after activation, with 10 ml of methyl orange dye solution (BDH) under varying operating



Before

after

Fig. 1. FT-IR spectra of Flint clay before and after activation.

conditions: different surface weights (0.05, 0.1, 0.15, 0.2, 0.25 g), different contact times (5, 15, 30, 45, 60, 90, 120 min), different initial dye concentrations (20, 40, 60, 80, 100 mg/L), and different temperatures (318, 308, 298, 288 K). The flasks (dye solution + surface weight) were placed in a shaking water bath (Labtech, South Korea) at a speed of 120 rpm. The concentration of methyl orange dye was determined by separating the molecule from the solid using a centrifuge. The molecule was then analyzed for absorbance using a UV-Vis spectrometer (UV-Vis) (Shimadzu, Japan) at λ_{max}=464nm. The amount and percentage of methyl orange dye adsorbed at time t (q_t) were calculated in mg/g according to the relationship [16]:

$$q_t = \frac{(C_\circ - C_t)V}{m} \qquad (1)$$

$$\%A = \left(\frac{C_\circ - C_t}{C_\circ}\right) \times 100 \qquad (2)$$

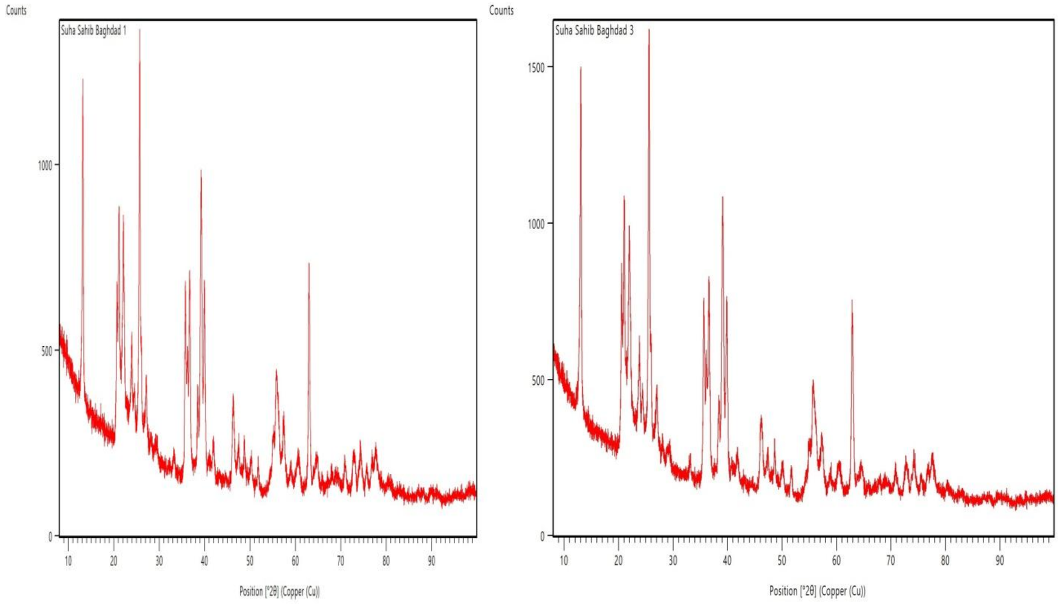
C =concentration at t=0 and t, m= weight of adsorbent, V= volume of dye solution (L).

RESULTS AND DISCUSSION

The surface of Iraqi flint clay was diagnosed before and after acid activation using several techniques (FT-IR, XRD, SEM, EDX) to determine the type of changes that occur on the surface and how this affects its behavior in adsorption processes·

FT-IR analysis

Analysis of FT-IR for flint clay was performed before and after acid activation, represented by the Fig. 1 respectively. Fig. 1 shows the presence of several peaks on the surface of the flint clay, as follows: the peak at (3649 cm^{-1}) is due to the stretching of the hydroxyl group, and a group of peaks in the region (900-1100 cm^{-1}) is due to (Si-O), while the group of peaks in the region (≤900) is due to (Si-O-Al, Al-OH-Al). However, Fig. 1 of the surface of the flint clay after acid activation shows relative changes in all peaks in terms of intensity and shape, with no peaks appearing or disappearing. This indicates the success of the acid activation process of the flint clay by distorting the uniformity of its layers without breaking them, which may reflect a tangible change in the surface behavior [17,18].



Before

After

Fig. 2. XRD spectra of Flint clay before and after activation.

XRD analysis

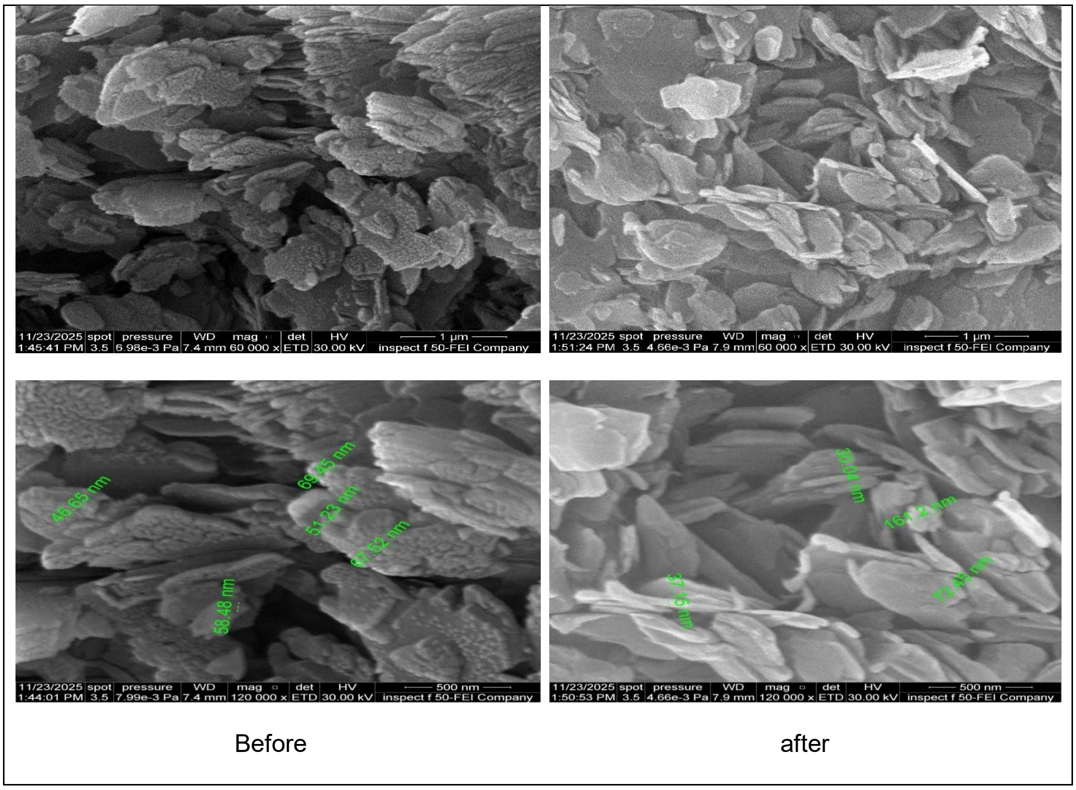
Analyses of XRD were performed on flint clay before and after acid activation, represented by Fig. 2 respectively. Several distinct peaks were found and included in Table 1. It was observed that most of the 2theta values of the flint clay surface decreased, in addition to a decrease in the (d) values of the distance between crystal planes, as well as changes in the (FWHM) values. This resulted in a decrease in crystal volume after the acid activation of the flint clay, indicating changes in the clay surface without destroying the clay's structure and composition. This is consistent with the results of FT-IR analysis. This reflects a tangible activity in the behavior of the clay surface [19,20].

SEM analysis

SEM analyses were performed on flint clay before and after acid activation by taking images at different magnification levels as shown in Fig. 3, where it is observed that flint clay before activation consists of homogeneous vertical conical clumps

Table 1. XRD data of characteristic peaks of flint clay before and after activation.

Flint					Flint-AC				
[°2θ]	d-spacing (A)	Rel-int	FWHM	Crystallite Size only [Å]	[°2θ]	d-spacing (A)	Rel-int	FWHM	Crystallite Size only [Å]
13.1499	6.72733	78.49	0.2399	321	13.029	6.78946	77.62	0.2597	300
21.1099	4.20518	57.21	0.3249	188	21.9508	4.04596	47.8	0.522	118
25.6655	3.46816	100	0.2838	265	25.5572	3.48261	100	0.2688	275
39.1352	2.29996	53.37	0.2807	298	39.0809	2.30303	63.91	0.4127	201
62.968	1.47493	65.34	0.2843	307	62.8363	1.47771	39.78	0.2866	342
Average d	3.635112	Average Crystallite Size		275.8	Average d	3.619754	Average Crystallite Size		247.2



Before

after

Fig. 3. SEM images of Flint clay before and after activation.

with an average particle size of 38 nm, but after acid activation, a large distortion occurs in these clumps, reflecting a decrease in homogeneity resulting in particles with an average size of 75 nm, which confirms the success of the change and activation process [21].

EDX analysis

Analysis of EDX was performed on flint clay before and after acid activation through Fig. 4. It is observed that the main constituent elements of flint clay before modification are (Al=38.12%, Si=27.76%, O=34.12%) while for flint clay after acid activation they are (Al=37.45%, Si=28%, O=34.55%). In addition, the slight changes in the proportions of the constituent elements indicate that the activation process did not change the composition of the clay but rather modified its surface properties. Also, the presence of only the main constituent elements of the clay indicates the purity of the clay sample after the activation process [22,23].

Adsorption test

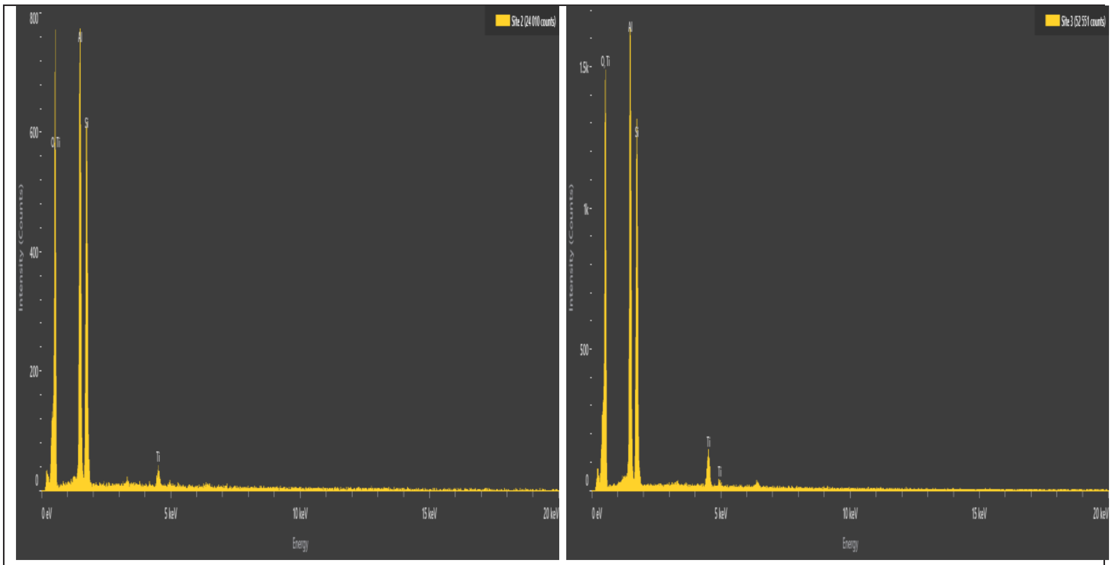
The adsorption experiments of methyl orange dye from its aqueous solutions on the surface of flint clay were carried out before and after acid modification in batch system and under working conditions of surface weight of the adsorbent, contact time, initial concentration of the dye and temperature, as follows.

Effect of weight

The adsorption experiments of methyl orange dye (100 mg/l) from its aqueous solutions by the flint surface before and after acid activation were carried out under the influence of a range of adsorbent surface weights (g0.005-0.025) and at an equilibrium time of (60 min) and a temperature of (298 K) through Fig. 5, where it was found that the ideal weight for the best percentage of adsorption of methyl orange dye to the flint clay surface before and after activation is 0.02 g [24].

Effect of time

The adsorption experiments of methyl orange dye (100 mg/l) from its aqueous solutions by the flint surface were carried out before and after acid activation under the influence of time periods (0-120 min) and at the weight of the adsorbent surface (0.02 g) and a temperature of (298 K) through Fig. 6. It was found that the amount of dye adsorbed increases significantly during the first 5 minutes due to the presence of the largest possible number of available adsorption sites. However, over time, the adsorption process



Before

after

Fig. 4. EDX spectra of Flint clay before and after activation.

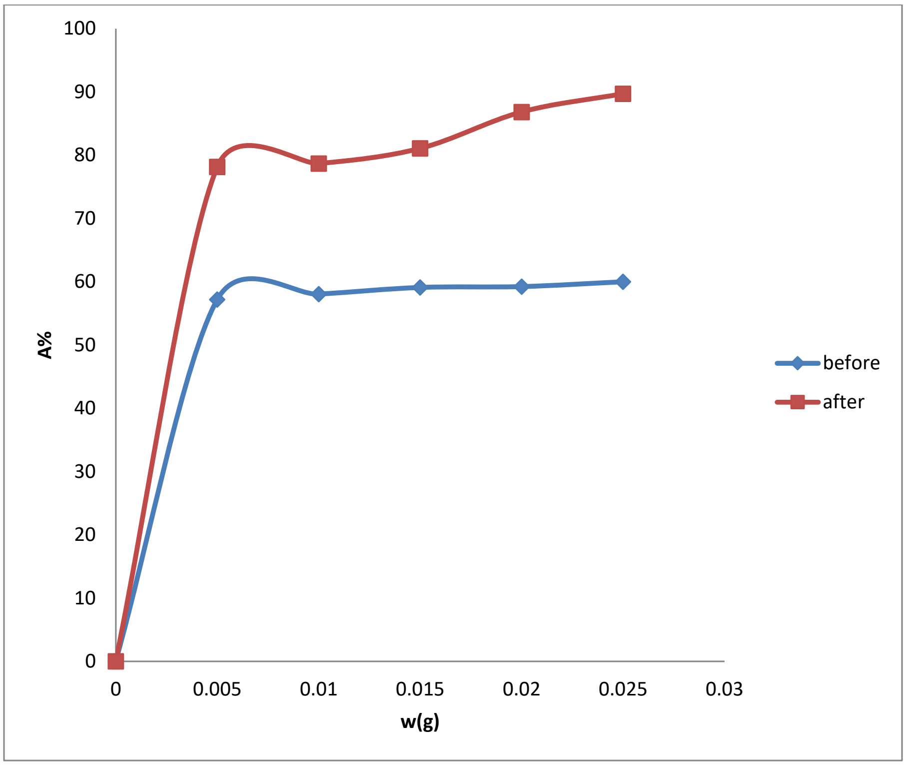


Fig. 5. Effect of weight flint clay before and after activation on A% of methyl orange dye.

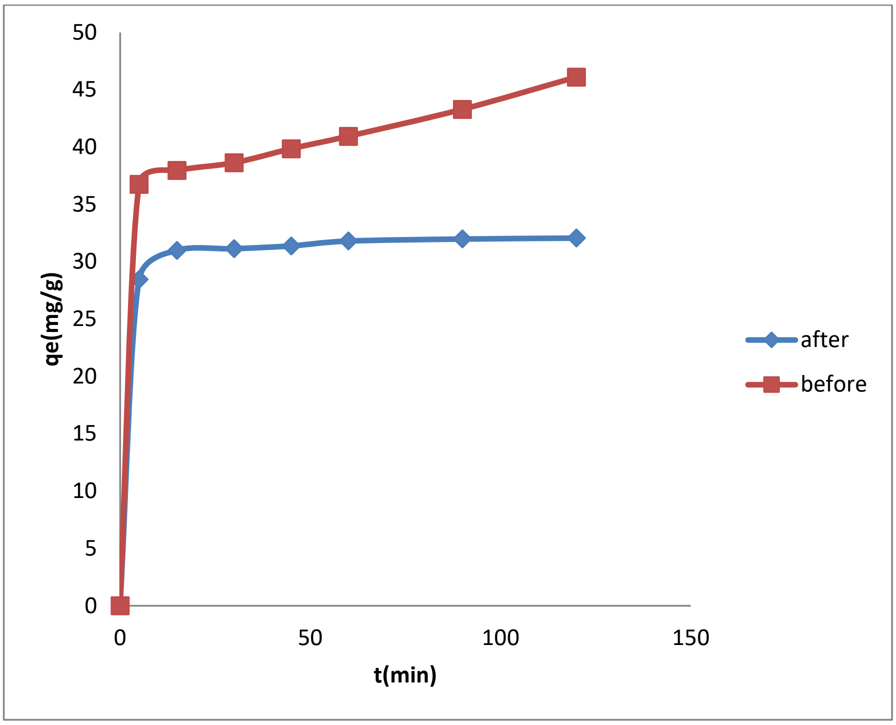


Fig. 6. Effect of contact time on adsorption of methyl orange dye by flint clay before and after activation.

begins to slow down until the surface saturation level is reached, resulting in the best amount of adsorption of methyl orange dye by the flint clay, which is (>30 mg/g), while the activated flint clay is (>40 mg/g) at an equilibrium time of (60 min) for both surfaces. This improvement in the amount of dye adsorbed by the flint clay after activation is due to the increase in the number of adsorption sites, the increase in surface area, and the cleaning of pores resulting from the effect of the acid molecules on the clay surface and its structure [25].

Kinetic study

The kinetic analysis of the experimental data for the adsorption of methyl orange dye by flint clay before and after acid activation was performed by applying pseudo-first order and pseudo second-order kinetic models according to the Eqs. 3 and 4 [26]:

$$\mathrm{Ln}(q_e - q_t) = \mathrm{Ln}q_e - k_1 t \qquad (3)$$

$$\frac{1}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \qquad (4)$$

Where (q_e, K_1, K_2) are kinematic constants calculated from the graphical representation of Eqs. 3 and 4 in Fig. 7 and included in Table 2.

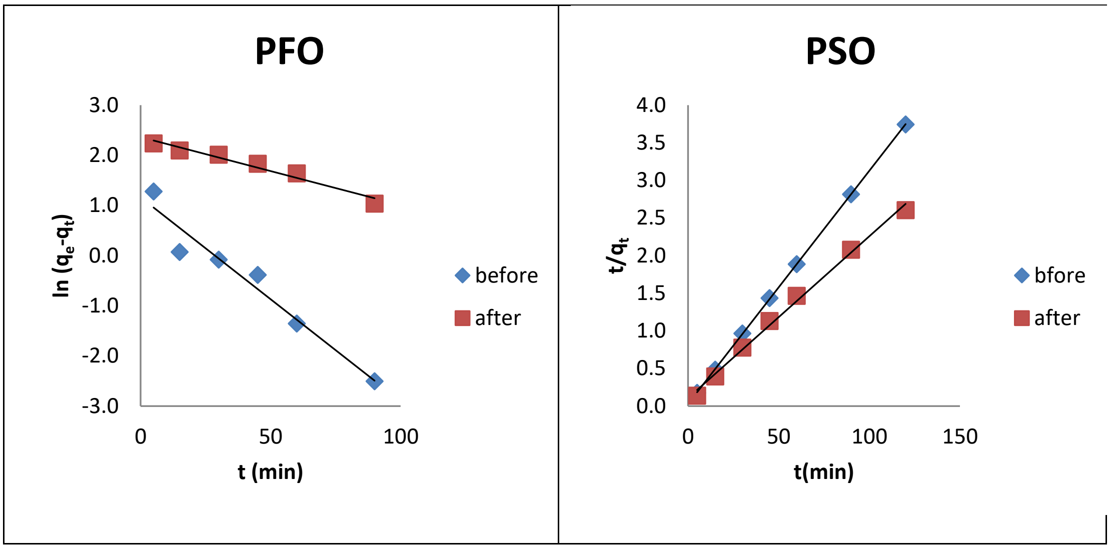


Fig. 7. A plot of pseudo first and second order for methyl orange dye adsorption by flint clay before and after activation.

Table 2. PFO and PSO kinetics constants for adsorption of MO dye on flint clay before and activation.

Surfaces	298 K			
	Pseudo- first order			
	k_1 (min^{-1})	$q_{e(mg/g)}$ (cal)	$q_{e(mg/g)}$ (exp)	R^2
Flint	0.020	12.561	32.058	0.989
AC-FLINT	0.014	10.633	46.094	0.961
	Pseudo-second order			
Surfaces	k_2(g. mg^{-1} .min^{-1})	$q_{e(mg/g)}$ (cal)	$q_{e(mg/g)}$ (exp)	R^2
Flint	0.005	44.053	32.058	0.997
AC-FLINT	0.004	46.512	46.094	0.995

It is observed from the Table 2 that the experimental data kinetically follow the pseudo second-order model more than the pseudo first-order model because it has a higher correlation coefficient value and is closer to the experimental and theoretical values of the amount of dye adsorbed than to the false first-order model. This indicates that the type of adsorption is of a chemical nature.

Effect of concentration

The adsorption experiments of methyl orange dye were carried out at a range of initial concentrations (40-100 mg/l) of its aqueous solutions by the flint surface before and after acid activation at a surface weight of (0.02 g) and an equilibrium time of (60 min) and at a temperature range of (288-318 K). Isotherm Gels were applied through Fig. 8. It was found that the adsorption of methyl orange dye by the flint clay surface followed the S1 class before activation and S3 after activation, indicating that the adsorption process occurred through the orientation of the methyl orange dye molecules in a vertical or weakly oblique manner towards the clay before activation, and then the orientation increased more strongly towards the activated clay, reflecting the improvement in surface properties as a result of the acid activation process. On the other hand, we note that the amount of adsorbed material increases with increasing initial concentration of the dye and increasing temperature, indicating that the adsorption process preferentially occurs at high temperatures, as the highest adsorption amount for the raw clay is (30 mg/g) and for the activated clay is (45 mg/g). This also indicates that the adsorption of methyl orange dye on flint clay before and after acid activation is of an endothermic nature [27].

The equilibrium data for the adsorption of methyl orange dye by flint clay before and after acid activation were analyzed by applying Langmuir and Freundlich isotherms according to the Eqs. 5 and 6 [28,29]:

$$\frac{C_e}{q_e} = \frac{1}{q_m . k_L} + \frac{C_e}{q_m} \qquad (5)$$

$$Lnq_e = Lnk_F + \frac{1}{n} LnC_e \qquad (6)$$

Where (k_L, K_f, q_m, n) are kinematic constants calculated from the graphical representation of Eqs. 5 and 6 and included in the Tables 3 and 4.

Tables 3 and 4 shows that the Langmuir isotherm model is unsuitable for the adsorption process of methyl orange dye on the flint clay surface before and after activation, despite the relatively high correlation coefficient values for the raw clay and the presence of negative adsorption quantities. This reflects a state of extreme disorder resulting

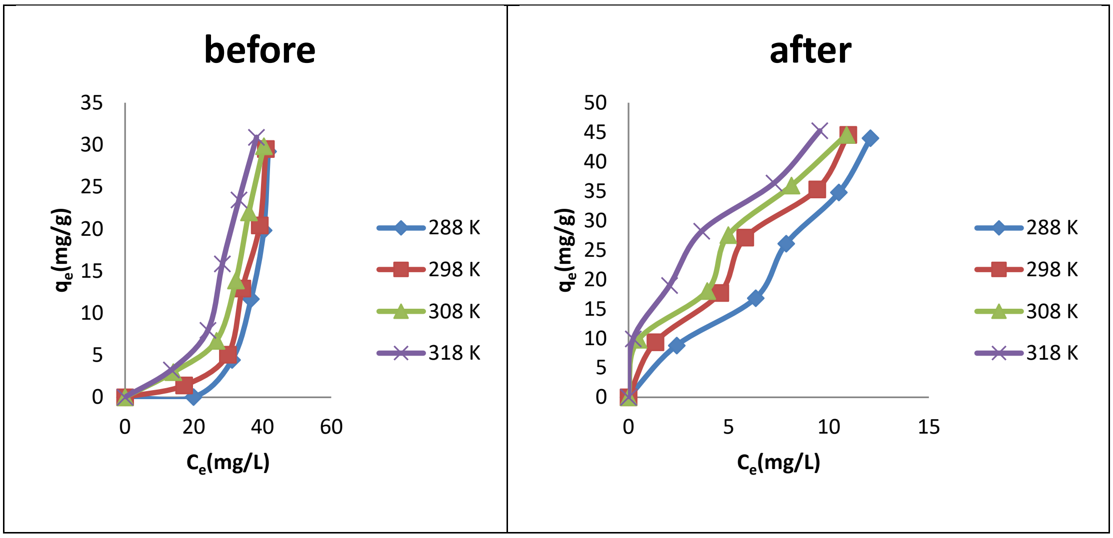


Fig. 8. Effect of concentration for methyl orange dye adsorption by flint clay before and after activation at different temperatures.

from the weak adsorption process occurring on the surface. In contrast, the Freundlich isotherm model is well-suited, with the constants (k_F, n) increasing with increasing temperature. This indicates an anothermic adsorption process occurring on heterogeneous surfaces. Based on the values of (n), where (n<1) is for adsorption before activation and (n>1) is for adsorption after activation, this demonstrates the preference for and spontaneity of adsorption after acid activation, despite the fact that both the dye and the surface are negatively charged [30].

Thermodynamic study

Thermodynamic data such as Gibbs free energy ΔG°, standard enthalpy changes ΔH° and standardentropy changes ΔS° were estimated using the following Eqs. 7-9:

$$\Delta G^\circ = -RTLnk_{eq} \quad (7)$$

$$k_{eq} = \frac{(C_i - C_e)V}{C_e.m} \quad (8)$$

$$\Delta G^\circ = \Delta H^\circ - T\Delta S^\circ \quad (9)$$

Where: K_{eq} is the equilibrium constant for the adsorption process, R: is theuniversal gas constant, T: absolute temperature (K), C_i and C_e: are the initial and the equilibrium concentrations (mg/L) of the adsorbate, respectively, V: is the CR solution's volume (L) and m: is the mass of the adsorbent (g). The standard entropy ΔS° and enthalpy ΔH° can be calculated from the intercept and the slope of the line between ln K_{eq} versus 1/T using Van't Hoff equation [31]. As shown in Fig. 9, Values of the thermodynamic data are given in Table 5.

$$Lnk_{eq} = \frac{\Delta S^\circ}{R} - \frac{\Delta H^\circ}{RT} \quad (10)$$

It is observed from Table 5 that the equilibrium constant values for the adsorption process increase with increasing temperature, indicating that the process is endothermic and preferential at high temperatures. On the other hand, the equilibrium constants for the adsorption process are higher on the surface of activated flint clay than on the raw flint clay, reflecting the ease and efficiency of adsorption, which is higher for the surface of activated flint clay compared to the surface of raw flint clay at all studied temperatures. It is also observed that the values of ΔG are positive, reflecting the difficulty and lack of residue in the adsorption process of methyl orange dye on the surface of flint clay, while conversely, the negative values of ΔG indicate the ease of adsorption on the surface of activated flint clay. It is also observed that the values of ΔH are positive, indicating that the adsorption process on the surface of flint clay before and after acid activation is endothermic in nature, which is consistent with the kinetic results. It is also observed that the values of ΔS are positive, indicating the randomness of the

Table 3. The calculated constants and correlation coefficients (R^2) for Langmuir model adsorption of MO dye onto flint clay before and after activation.

T/K	Flint			AC-flint		
	k_L	q_m	R^2	k_L	q_m	R^2
288	-0.710	-0.025	0.913	-0.004	-769.231	0.013
298	-0.0235	-2.1124	0.970	0.059	103.093	0.565
308	-0.027	-7.794	0.906	0.182	60.606	0.715
318	-0.022	-7.868	0.938	0.483	50.000	0.897

Table 4. The calculated constants and correlation coefficients (R^2) for Freundlich model adsorption of MO dye onto flint clay before and after activation.

T/K	Flint			AC-flint		
	K_F	n	R^2	K_F	n	R^2
288	0.000000342	0.207	0.987	3.428	1.021	0.959
298	0.00007	0.292	0.953	7.052	1.370	0.965
308	0.008	0.463	0.929	12.460	2.083	0.925
318	0.009	0.452	0.968	16.656	2.503	0.973

Table 5. The calculated thermodynamic parameters for adsorption of MO dye onto flint clay before and after activation.

T/K	Flint				flint-AC			
	ΔG° (k J.mol^{-1})	ΔH° (k J.mol^{-1})	ΔS° (J.mol^{-1} K^{-1})	k_{eq}	ΔG° (J.mol^{-1})	ΔH (J.mol^{-1})°	ΔS° (J.mol^{-1} K^{-1})	k_{eq}
288	0.849	3.717	0.0100	0.701	-3.093	6.052	0.0318	3.639
298	0.815		0.0097	0.720	-3.472		0.0320	4.060
308	0.776		0.0096	0.739	-3.610		0.0314	4.094
318	0.563		0.0099	0.808	-4.111		0.0320	4.735

adsorption process on all studied surfaces [32].

CONCLUSION

We conclude that the acid activation of the flint clay surface was successful using several techniques (FT-IR, XRD, SEM, EDX). This success is evidenced by changes in the clay surface and its pores, resulting in an improvement in the amount of dye adsorbed. For the raw clay, the adsorbent value (qe = 30 mg/g), while for the activated clay, it reached (qe = 45 mg/g) at the preferred high temperatures. The adsorption data follow a pseudo-second-order kinetic model, indicating a chemical adsorption process. Conversely, the adsorption process follows a Freundlich isotherm model, suggesting that the flint clay surface before and after acid activation is heterogeneous. Furthermore, we conclude that the thermodynamic characterization of the adsorption process for methyl orange dye is non-spontaneous, endothermic, and random on raw flint clay, while it becomes spontaneous, more random, and endothermic in the activated flint clay. Therefore, we conclude that the efficiency of the flint clay surface in removing methyl orange dye from its aqueous solutions is improved as a result of acid activation.

ACKNOWLEDGEMENTS

The authors thank the (Department of Chemistry - College of Education for Pure Sciences, Ibn Al-Haytham - University of Baghdad) for their assistance with tools, equipment, and laboratories.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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