Removal of Direct 50 Dyes from Aqueous Solution Using Natural Clay and Organoclay Adsorbents

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Received 25, February, 2014
Accepted 15, June, 2014

Abstract

In this study, hexadecyltrimethylammonium bromide (HDMAB) - bentonite was synthesized by placing alkylammonium cation onto bentonite. Adsorption of textile dye such as direct Yellow 50 on natural bentonite and HDMAB -bentonite was investigated. The effects of pH, contact time,dosage clay and temperature were investigated experimentally .The Langmuir and Freundlish isotherms equations were applied to the data and values of parameters of these isotherm equations were evaluated. The study indicated that using 0.2 g of HDMAB (hexadecyltrimethylammonium bromide) lead to increase the percentage removal(R%) from 78% for pure bentonite to 99 %. The optimum pH value for the adsorption experiments was found to be pH=3 and therefore all the experiments were carried out at this pH value. The pseudo-second-order kinetic model agrees very well with the experimental results.Different thermodynamic parameters such as Gibb's free energy, enthalpy and entropy of the on-going adsorption process have also been evaluated. The thermodynamic analyses of the dye adsorption on organoclay indicated that the system was endothermic in nature .

Key words: direct yellow50, adsorption, bentonite, organoclay.

Introduction:

The pollution of water resources with industrial effluents containing organic compounds and toxic substances is a matter of great concern[1]. Environmental contamination has been pointed as one of the greatest problems of modern society, mainly due to the population explosion and the increased industrial activity[2-3]. Dyes have been used long in dyeing, paper and pulp, textiles, plastics, leather, cosmetics and food industries. Effluents discharged from these industries poses certain hazards and environmental problems. Wastewater from these industries may present an eco-toxic hazard and introduce the potential danger of bioaccumulation, which may eventually affect human beings.

Various techniques have been employed for the removal of dyes from wastewaters. The most widely used methods for removing color effluents from water include chemical precipitation, ion-exchange, osmosis, ozonation, solvent extraction, adsorption, membrane filtration, but only that of adsorption is considered to be superior to other techniques[4-7]. The uses of bentonite in wastewater treatment have received increasing attention and currently offers a very attractive method for pollution remediation. Besides it is plentiful inexpensive and available in many countries. Bentonite is a Smectite, which is a 2:1 layered silicate ,the inner layer is composed of an octahedral sheet which is situated

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between two tetrahedral sheets. The substitutions of Al^{3+} for Si^{4+}in the tetrahedral layer and Mg^{2+} or Zn^{2+} for Al^{3+} in the octahedral layer result in a net negative surface charge ion on the clay. The charge imbalance is offset by exchangeable cation such as H^+,Na^+, or Ca^{2+}on layer surfaces[8].Because of the repulsion interactions between the negatively charged surface of clays and anionic dyes , only few studies on the adsorption of acid dyes have been carried out using bentonite as an adsorbent. Surface properties of natural clay can be easily modified by exchanging their inorganic cations with quaternary ammonium cations[9-10] . This may result in an increase in the inter – lamellar spacing and exposure of new sorption sites of the clay. More importantly, the substituted organic cations are weakly hydrated and as the inorganic cations are progressively replaced by the organic cations, the surface properties of a clay may change considerably from highly hydrophilic to increasingly organophilic (hydrophobic)[11-13].

In the present work, bentonite modified with hexadecyltrimethylammonium bromide was prepared and the ability of clay to remove anionic dye by adsorption, has been considered. The effects of contact time, initial dye concentration and pH on the amount of colour removal were investigated. The equilibrium experimental data were fitted into Langmuir and Freundlich equations to determine the best isotherm correlation. In addition ,the kinetics and thermodynamic of the process were also investing at end.

Materials and Methods:

Adsorbents .

Natural bentonite.

The bentonite clay was supplied from the state company for Geological Survey and Mining – Iraq. The bentonite had the following composition with particle size less than (75) µm. Information and analysis for the surface were supplied by the same company and described below in Table(1). The molecular formula of bentonite could be written as:$Mg_2Al_{10}Si_{24}O_{60}$ (OH) $_{12}$[Na, Ca].[13]

Table.(1) Chemical specifications of *Natural bentonite*.[13]

Constituent	SiO_2	Al_2O_3	CaO	MgO	K_2O	Na_2O	Fe_2O_3	L.O.I
Wt%	56.77	15.67	4.48	3.42	0.60	1.11	5.02	12.49

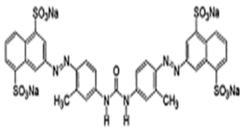
modified bentonite

Three samples of these modified bentonite were prepared .The preparation of modified benonite involves mixing different weight (0.01,0.05,0.1)g of modifier (HDTAB), with (10)g of natural bentonite and (100)ml of deionized water. This content was shacked about 3 hr. at 25 °C and then separated and dried at 105 °C

Adsorbate

Direct yellow 50 is an organic compound that is used as a dyestuff .It is traditionally used as a dye for materials such as silk, leather, and paper . It is known high solubility in water, λ max = 398nm[14]. **Manufacturing Methods :** 3-Aminonaphthalene-1,5-disulfonic acid diazo, and a coupling m-Toluidine, then the light gasification. The properties of the dye is listed in Table (2)

Table (2) The physical and chemical properties of DY50

Specification		Structure of dye
Empirical formula	$C_{35}H_{24}N_6Na_4O_{13}S_4$	
Molar mass	956.63	
Class	Double Azo	
Solubility	Water	

Batch mode adsorption :-

Batch mode adsorption studies for individual dye was carried out to investigate the effect of different parameters such as adsorbate concentration, adsorbent dose, temperature and pH. Solution containing 50 ml adsorbate and 0.2 g adsorbent was taken in250 ml capacity conical flask and agitated at 200 rpm in water bath shaker at predetermined time intervals. The adsorbate solution was centrifuge at (3000 rpm) for (30min) .The percentage of dye removal (R in %) and quantity of DY50 (Qe) was calculated using Eq. 1 and 2 respectively. [15]:

$$R\% = \frac{(C_o - C_t)}{C_o} \times 100 \text{ ----------(1)}$$

$$Q_e = \frac{C_0 - C_e}{m} * V \text{ ------ (2)}$$

where C_0 and Ce are the initial and the equilibrium concentrations (mg L^{-1}) of DY50 in solution, respectively. Q_e ,is the amount of dye adsorbed per unit of clay mg/g, V is volume L of solution and m is the mass of adsorbent (g) used in each r experiment . The residual dye concentrations of each solution were determined by measuring their characteristic absorbance using a double beam UV-Vis Spectrophotometer (Schimadzu, uv-160A) at a wavelength (398nm). The spectrum for 50 mg/L DY50 adsorption was shown in Figuer. 1. The calibration curve at this wavelength was established as a function of DY 50 dye concentration in Figuer . 2

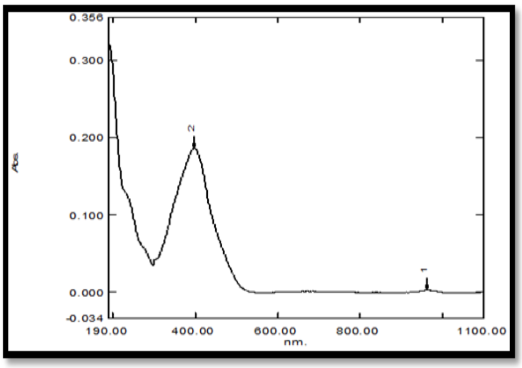


Fig.(1) UV-Visible absorption spectrum for (10) mgL^{-1} DY50 dye solution

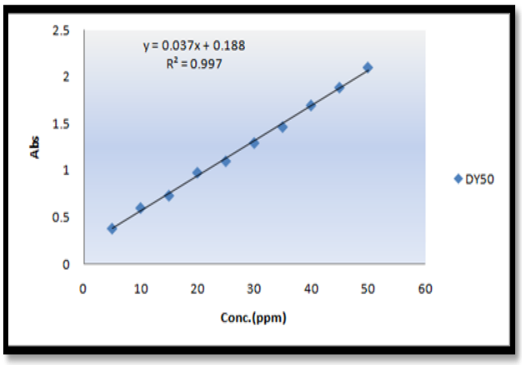


Fig .(2)Calibration curve of DY50 dye (5-50) ppm ,pH 7, 25C^0.

The Effect of Adsorption Parameters

To examine the effect of temperature, adsorption experiments were conducted at 25, 30,35 40, and 45°C, respectively. The influence of the initial pH was studied at values: 3, 5, 7 and 9. Dye solution pH was adjusted using 0.1M NaOH and 0.1M HCl solutions and measured using a Meter Lab, pH meter. Adsorbent dosage used in this study was in the range 0.1 to 0.7 g L^{-1}.

Results and Discussion:

The Effect of Modifier

The adsorption of 50 mgL^{-1} direct yellow 50 on modified bentonite is shown in Figuer.3. The results indicated that the R% of 50 mgL^{-1} direct yellow 50 dye increase from 99.1% to 99.38% when 0.1 g HDMAB added to 10 g natural bentonite .As a conclusion, the best amount of modifier was 0.1g HDMAB.

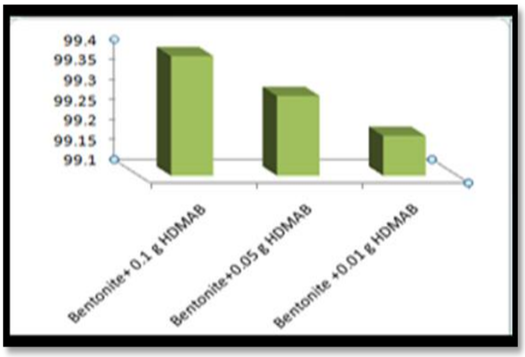


Fig.(3)The Effect of adding different amount of modifier to bentonite on Removal percentage 50mg/L DY50

The Effect of Shaking Time:-

The time-dependent behaviour of dye adsorption was examined by varying the contact time between adsorbate and adsorbent in the range of 5-60 min. The concentrations of dye was kept as 50 mg/Land the amount of adsorbent added was 0.2 g. The adsorption data for the uptake of DY50 versus contact time was represented in Figuer4. The removal of direct dye by adsorption increased with time and attained a maximum value in 40 min and thereafter it remained constant for all the concentration studied .This mean that uptake attained equilibrium at 40 min for natural and modified bentonite at the same conditions. Therefore, a 40 min shaking time was found to be appropriate for maximum adsorption and was used in all subsequent experiments[14]. Although the rate behavior of both bentonite and modified bentonite was similar, but it has been found that under identical conditions the modified bentonite presented higher removal percentage [16-18]

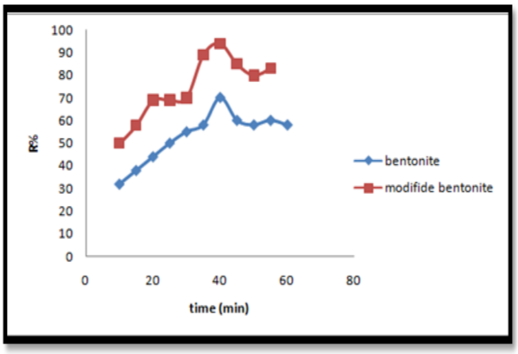


Fig .(4)The Effect of contact time on DY50 adsorption on natural bentonite and Modified bentonite.

The increase in the adsorption capacity of modified bentonite may be attributed to that ,in the modified form (HDTMA-Bentonite) the clay surface become organophilic and interact strongly with organic compound. In dye adsorption may be enhanced hydrophobic interaction between the adsorbed dye molecule and HDTMA-Bentonite leading to the formation of organic phases consisting mostly of the C_{16} hydrocarbon group into which solutes are partitioned thus the degree of sorption of dye depends on the amount of the surfactant ions in the bentonite inter layer sites [19].

The Effect of Adsorbent Dosage

The effect of adsorbent mass on the percentage removal of DY50 dye using an initial dye concentration of 50 ppm is examined. The studied adsorbent dosages were0.1, 0.2 ,0.3, 0.4 ,0.5,and 0.6 g as shown in Figuer.(5). It can be noticed that increasing the bentonite dosage gradually increase the percentage removal of DY50 . The increase in the extent (R%) of DY50 was found to be insignificant after 0.2 g .The R% for natural bentonite and modified bentonite are 78% and 96% respectively .

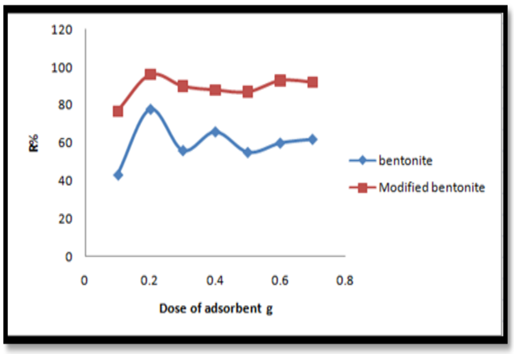


Fig.(5)The Effect of the adsorbent mass on the adsorption of 50 mg L^{-1} DY50

The Effect of pH on Adsorption

The effect of pH on DY50 adsorption onto natural and modified bentonite was studied through tests carried out on solutions of various pH as shown Figuer(6). It showed that the adsorption of dye DY50 onto modified bentonite increased with decreasing solution pH and reach to highest value at pH=3. This is due to high electrostatic attraction between the positively charged (high concentrations of H^+) surface of the adsorbents and anionic dye [20].When the pH is increased; the electrostatic repulsion increases and the adsorption rate get decreased. However, the adsorption of DY50 dye on natural bentonite was not affected.

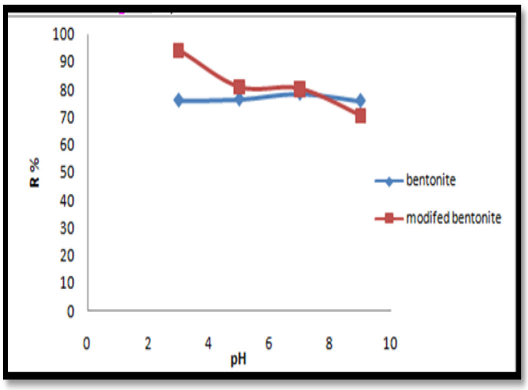


Fig. (6)The Effect of the pH on the adsorption of 50 ppm, $25C^0$,0.2g clay

The Effect of Temperature

The effect of temperature on the equilibrium adsorption capacity of DY50 onto Modified bentonite was investigated at a concentration of (5-50) mg /L at 25, 30,35, and 40 °C. The results were represented in Figuer.7 It can be seen that with the increase of temperature, the adsorption capacity for modified bentonite was slightly increased. This observation revealed that the adsorption process is slightly endothermic [21].This suggesting the adsorption behavior was insensitive to the changes of temperature in the range investigated [22]

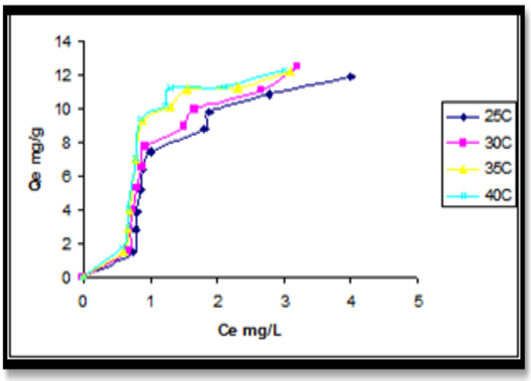


Fig.(7) Adsorption isotherm at different temperatures of DY50 Dye on HDMAB –Bentonite

Thermodynamics of Adsorption

The equilibrium constant, K_{eq} has been calculated with the help of the equation (3),where Q_e is the amount of adsorbed dye at equilibrium (mg / kg) and C_e is equilibrium concentration of dye in solution (mg / L). K_{eq} was then used to calculate the standard free energy (ΔG°)using equation (4). ΔH° and ΔS°were determined from the slope and intercept of Van't Hoff's, equation (5) [23-24] .The calculated standard free energy change (ΔG°), standard enthalpy change (ΔH°) and entropy change (ΔS°) parameters at different temperatures are presented in Table (3)

$$K_{eq} = Q_e / C_e \text{ ------------(3)}$$
$$\Delta G^\circ = - RT \ln K_{eq} \text{ ------------(4)}$$
$$\ln K_{eq} = - \Delta H^\circ / RT + \Delta S^\circ / R \text{------(5)}$$

The linear plot of $\ln K_{eq}$ versus 1/T is shown in Figure (8) .The values indicating that the adsorption of DY50 is spontaneous and thermodynamically favorable . The ΔH° positive values indicate that the adsorption of DY50 dye onto organoclay was an endothermic process, which was supported by the increase of adsorption of the dye with increase in temperature. Furthermore, the (ΔS°) positive indicates that the degrees of freedom increased at the solid – liquid interface during adsorption of the direct dye [25]. Generally, Δ*G*° for physisorption is between -20 to 0 kJ/mol and for chemisorption is between -80 to -400 kJ/mol[26-27].So that the adsorption of DY50 onto the given adsorbent is physisorption type.

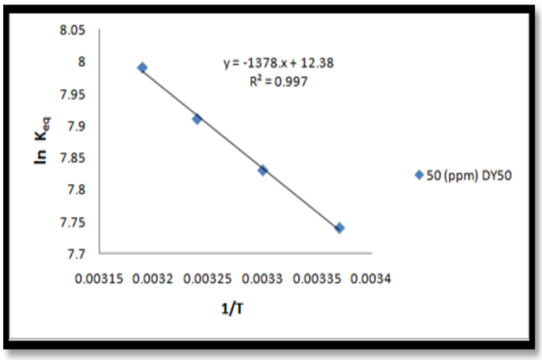


Fig (8) Values of 1/*T* and ln K_{eq} for the DY50.

Table(3). Thermodynamic parameters of DY50 adsorption on organoclay at 50 mg/L , and pH 3

ΔH kJ mol^{-1}	ΔS, J mol^{-1} K^{-1}	ΔG kJ mol^{-1}			
		25°C	30°C	35°C	40°C
20.33	40.5	-19.40	-21.15	-22.03	-22.9

Adsorption isotherms

Adsorption isotherms play a very important role for understanding adsorption mechanism.The variation of the dye concentration with the same mass of the adsorbent is used to determine an adsorption mechanism. Figure(9) shows the adsorption isotherm which was of S-type .This indicating a vertical or flat orientation of dsorbate , and the adsorbate is mono functional.The Langmuir (Langmuir, 1918) and Freundlich (Freundlich, 1906) adsorption isotherm models were applied to describe the dye-organoclay system ,equations (5),(6) [28-29].

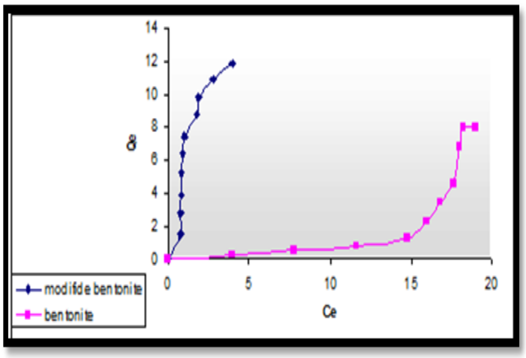


Fig. (9) Adsorption isotherms of DY50 on bentonite and modified clay at 25 ^{0}C pH7

$$C_e/Q_e = 1/Q_m K_L + C_e/Q_m \text{ -----(5)}$$
$$\log Q_e = \log K_f + 1/n \log C_e \text{------(6)}$$

Where Q_m is Langmiur constant related to the capacity and K_L is related to the energy of adsorption, 1/n and K_f are Freundlich constants related to the intensity of adsorption and adsorption capacity respectively. Figures (10) and (11) show the linear plots of Langmuir and Freundlich respectively. To confirm the favorability of the adsorption process, the separation factor (R_L) is calculated by using the [equation(7)]and presented in Table (4). The values of R_L are found to be between 0 and 1 and confirm that the ongoing adsorption process is favourable . R_L values indicate that the type of isotherm is irreversible (R=0) , favorable ($0 < R_L < 1$) , linear (R_L=1) or unfavorable ($R_L > 1$) [30] .

$$R_L = \frac{1}{1+K_L C_0} \text{ -----(7)}$$

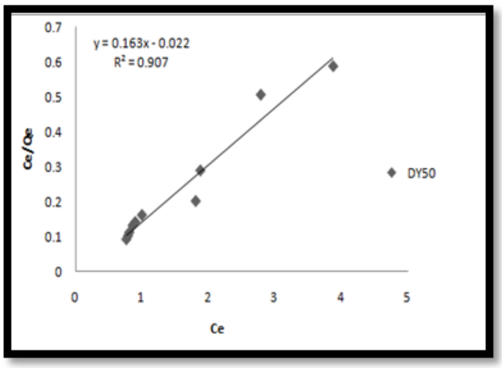


Fig.(10). The linear plot of Langmuir isotherm at 25 ^{0}C

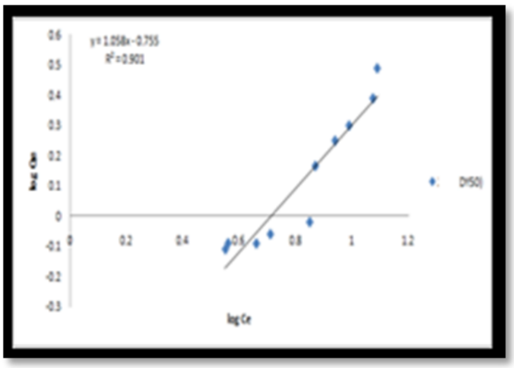


Fig. (11). The linear plot of Freundlich isotherm at 25 ^{0}C

The values of *n*>1 indicate favourable adsorption conditions. The values of linear *R*^{2} coefficient were high (> 0.9) for Freundlich isotherm indicating the useful values of its constants .So the adsorption isotherm for the present system is explained better by Freundlich isotherm model.

Table (4) The parameters of Freundlich and Lungmier isotherms

<table>
<tr><td rowspan="2">Freundlich parameter</td><td>T (°C)</td><td colspan="2">1/n</td><td colspan="2">K_F</td><td>R²</td></tr>
<tr><td>25</td><td colspan="2">0.5 22</td><td colspan="2">0.6 17</td><td>0.971</td></tr>
<tr><td rowspan="2">Langmuir parameter</td><td>T (°C)</td><td>q _L</td><td colspan="2">K_L</td><td>R_L</td><td>R²</td></tr>
<tr><td>25</td><td>0.923</td><td colspan="2">0.866</td><td>0.190</td><td>0.909</td></tr>
</table>

.Kinetics investigation

In order to determine the adsorption kinetics of dye, the pseudo-first-order, pseudo-second order and intrapartical diffution kinetics models were tested. The pseudo-first order kinetic model of Lagergren is routinely used in the linearized form:[31]

$$\log(q_e - q_t) = \log q_e - k_1 t \text{-------- } (8)$$

where q_t is the amount of adsorbed dye onto the adsorbent at time t and k_1 is the rate constant of first-order adsorption.The pseudo-second-order kinetic mode in its integrated linear form is.[32]

$$t / q_t = 1 / q_e^2 k_2 + 1 / q_e t \text{ --------} (9)$$

where k_2 is the rate constant of second -order adsorption.The intra-particle diffusion kinetic model based on the equation proposed by Weber and Morris is[33].

$$q_t = k_D t^{1/2} + C \text{ --------} (10)$$

where k_D is the rate of diffusion and C is the intercept. Kinetic parameters obtained after subjecting experimental data to the three kinetic models are shown in Table (5), while the linear plots of the above three equations are shown in Figuers. (12).(13),and,(14). According to the values of correlation coefficient, the 2nd order model is found to be more suitable to describe the adsorption kinetic data than the 1st order model for the adsorption system . Similar behavior has also been reported in the literature[34].

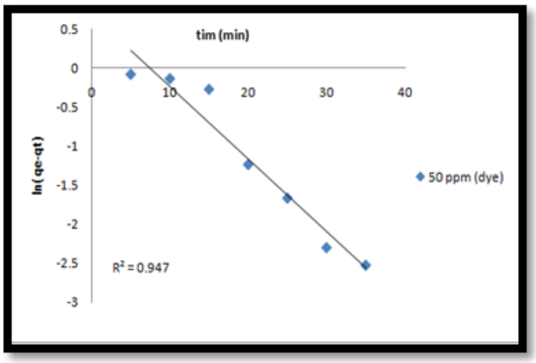


Fig. (12) The liner kinetic plot for pseudo-first order

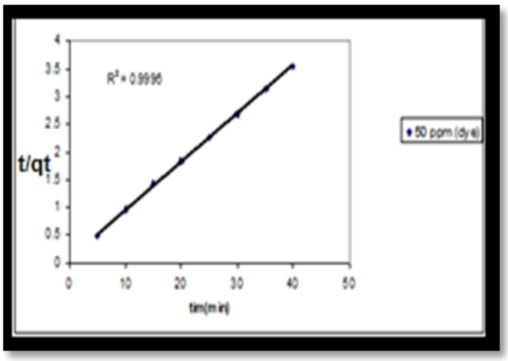


Fig. (13) The liner kinetic plot for pseudo-second

Although the regression of intraparticle diffusion was linear, the plot didn't pass through the origin, suggesting that adsorption involved intraparticle but wasn't the only rate-controlling step [35]The values of k_D , which are less than k_2, have also been confirmed that the intraparticle diffusion was rate-controlling step.

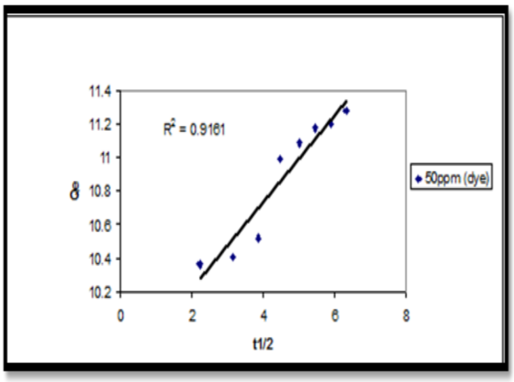


Fig. (14) The liner kinetic plot for intraparticle diffusion

Table (5) The values of the constants of the three model of kinetic for 50 ppm dye

pseudo-first - order			pseudo-second – order			Intrapart-yical diffusion	
q_e(calc.)	K_1	R^2	q_e(calc.)	K_2	R^2	K_D	R^2
0.973	0.666	0.947	1.66	0.087	0.9996	0.053	0.919

Conclusions:

The present work shows that organoclay is an effective adsorbent for the removal of synthetic yellow dye DY50 from aqueous solutions. The amount of dye adsorbed is dependent on amount of the adsorbent USED and increased slightly in lower pH (pH = 3), in the studied dye concentration range. With the increase in the temperature, the amount adsorbed dye increased, indicating that the adsorption of dye onto organoclay is an endothermic process. The best correlation was obtained using the pseudo-second -order, second-order kinetic model. Equilibrium data were also fitted well to the Freundlich isotherm model for low initial dye concentrations.

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ازالة الصبغة المباشرة DY50 على سطح طين البنتونايت والبنتونايت العضوي من محلولها المائي

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الخلاصة:

في هذا البحث تم تحضير بنتونايت عضوي من خلال استبدال الكاتيون اللاعضوي بالكاتيون العضوي (alkylammonium) تم دراسة تاثير كل من الدالة الحامضيه (pH) وزمن التماس وكميه المادة ألمازة على عملية امتزاز الصبغة (Direct yellow 50) على كل من طين البنتونايت الطبيعي والعضوي المحضر كلا على انفراد . طبقت ايزوثرمات لنكماير وفرندلش على النتائج العملية على طين البنتونايت العضوي ثم حسبت المعاملات الخاصة بهذه المعادلات . ودلت نتائج الدراسة ان الطين العضوي المحضر كان أفضل إزالة من الطين الطبيعي بنسبه إزالة وصلت إلى 99% . وكانت الظروف المثلى لإجراء عملية الامتزاز بالنسبة للدالة الحامضية هي pH=3 . تم حساب الدوال الثرموديناميكيه ΔH° و ΔS° , ΔG° لعملية الامتزاز من خلال دراسة تاثير درجات الحرارة ودلت نتائجها على أن عملية الامتزاز كانت تلقائية وماصة للحرارة ، بينما دلت النتائج الحركية على ان عملية الامتزاز تتبع معادلة الدرجة الثانية الكاذبه .