Removal of Birlliant Green Dye From Aqueous Solution by Adsorption Onto Modified Clay

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Abstract

Application of a Fe-bentonite nano clay (Fe-BNC) as modified clay has been investigated for the removal of birlliant green (BG) from aqueous solutions. Atomic force microscope measurements give a detailed information on pore shape and pore size distribution about the clay. These measurements show that the average diameter of the improved clay is 346.84 nm. Batch adsorption experiments were carried out for the removal of (BG) from aqueous solutions onto Fe-BNC.

Equilibrium data were fitted to Freundlich and Langmuir isotherm equations and the isotherm constants were determined. Thermodynamic parameters such as free energy, entropy and enthalpy, have been calculated.

For the modified clay the study of adsorption rate constants has been carried out using lagergrens first order rate equations for the adsorption processes and it is found to follow first order rate kinetics. In addition, an activation energy of sorption has also been determined.

Keywords : Adsorption, Birlliant green dye, bentonite.

Introduction

Dyes and coloring materials are frequently used in some industries such as; textile, dye stuff manufacturing, leather tanning, food preparation, paper production that produce a high colored waste effluents [1]. The discharge of effluents is one of the potential sources of contamination and pollution [2], and also can consume the dissolve oxygen in water which is required by aquatic life. Some of these dyes were found to be toxic to some organisms and cause allergic dermatitis, skin irritation and mutation in man. It was reported that dyes are resistant to light and moderate oxidative agents, so they cannot be completely removed by conventional biological treatment process, including activated sludge and anaerobic digestion [3].

Birlliant green (BG), is known to be used in several industries including leather products, dying wool, silk and jute. It is also used in distilleries [4]. In addition to its high risk of cancer and many other diseases to human, this dye is highly toxic to the microbial populations in BG-containing water, therefore it is so important to develop a system that can be used to remove this dye from the water. Precipitation, coagulation, ozonization, ion exchange as well as adsorption methods are the conventional methods used for the removal of this dye [5]. Among the various treatments available for the removal of dyes, adsorption technique is one of the most effective treatment methods due to simplicity and high efficiency, as well as the availability of a wide range of adsorbents. Some materials like fly ash, clays, alumina, activated carbon and some agricultural byproducts have been as adsorbents for removal of dyes from industrial wastewaters [6]. It was found that the use of the clay materials as an adsorbent has many advantages in comparison of other adsorbant due to the low cost, lack of toxicity and good potential for ion exchange. The clay materials have a high specific surface area which gave these materials unique properties and were used in several application [7]. Lately, many studies have reported the use of nano-particels and cause a significant breakthrough in many areas of applied sciences. In particular, nano-particles smaller than 100 mm were found to be important in natural systems due to the high surface area, surface activity and their associated properties of adsorbing to organic and other trace metal contaminants [8]. This work examines the efficiency of using modified nano-clays to remove BG by measuring the adsorption data.

Materials and method

Materials

Adsorbate :All chemicals (such as Fe(NO3)3.9H2O and Na2CO3) used in the experiments were analytical grad chemical and were obtained from Merck. Stock solutions of the test reagent were made by dissolving the dye in distilled water. The dye, Birlliant green sulfate, chemical formula: $C_{27}H_{34}N_2O_4S$. Molar mass= 482.65, λ_{max}: 625 nm (measured value) was supplied by Merck. The chemical structure of birlliant green sulfate is shown in Figure (1)

Adsorbent: The adsorbent, bentonite clay was collected from Al-Sufra in Al-Rutba, Iraq. It was supplied by geological survey and mining-Iraq. The components of this clay are SiO_2 (56.77%), Al_2O_3 (15.67%), MgO (3.42%), K_2O (0.06%), Na_2O (1.11%), Fe_2O_3 (5.12%), CaO (4.48%) and loss of Ignition L.O.T (12.49%).

Preparation of Fe-bentonite nano composite

The Fe-BNC was prepared through the following steps [9]. Firstly an aqueous dispersion of bentonite clay was prepared by adding 10 gm bentonite clay to 500 ml H_2O under vigorous stirring for (3 hrs.) at room temperature. Secondary, sodium carbonate was added slowly as a powder into a vigorously stirred (0.2 M) solution of iron nitrate for (3 hrs.) such that a molar ratio of (1:1) for $[Na^+]/[Fe^{+3}]$ was established. Thirdly, 500 ml solution obtained from the second step was added drop by drop into the dispersion of bentonite clay prepared in the first step under vigorous stirring. Fourthly, the suspension was stirred for (3 hrs.) followed by ageing at 100ºC in an autoclave for (48 hrs). Finally, 1000 solution containing Fe-bentonite nano clay was obtained [10]. The modified clay was separated from the solution by filtration, then dried in oven at 100 ℃ and stored in desiccators.

Characterization measurement

The atomic force microscopy (AFM) study carried out by (AA 3000, Angstrom Advanced IUC. USA) Fig.(2) shows typical AFM Top-view of (I) clay, (II) Fe-BNC. The results show that the average diameter for the clay is 699.22 nm, while the pore diameter of Fe-bentonite clay is about (300-400)nm with an average of 346.84 nm, it means that the pore diameter of the Fe-BNC is smaller than that of the clay**.**

Methods

Adsorption studies

The adsorption of BG on to the modified clay was investigated in a batch system. Equilibrium experiments were carried out by containing 0.5 gm of modified clay to 50 ml of dye solution of different initial concentrations (5-20 ppm). A series of round bottom flasks were then shaken at a constant speed of 100 rpm in a shaker (CRIFFIN FLASK SHAKER) for 30 min (Equilibrium time) at four temperatures in the range (283-303)K. The dye solution was separated from the adsorbent by filtration residual concentration of dye in supernatant was estimated spectrophotometrically by monitoring the absorbance at 625 nm λ_{max} using UV-Vis spectrophotometer (UV-1800 shimadzu). Amount of adsorbed dye molecules per gm of solid was determined as follows:

$Q_e = (C_o - C_e)\, V/W$…..………(1)

Where, C_o is the initial concentration of BG (mg/L), C_e is the equilibrium concentration of dye (mg/L), V is the volume of the solution (L) and (W) is the mass of the Fe-BNC (g). The removal efficiency (R) is defined as: [11]

Removal of dye (%) = $[(C_o - C_e)/C_o] \times 100$…..………(2)

Isotherm modeling

The isotherm models of Langmuir [12], Freundlich [13] were fitted to describe the equilibrium adsorption.

Langmuir isotherm

$Q_e = K_L\, C_e/ 1 + aC_e$…..………(3)

The linear form of the Langmuir isotherm equation is represented in the following formula:

$C_e/Q_e = 1/K_L + a/K_L.C_e$…..………(4)

Where Ce is the supernatant concentration at equilibrium state of the system ($mg.L^{-1}$), Q_e is the amount of dye adsorbed per unit weight of adsorbent ($mg.g^{-1}$), assuming a mono layer of adsorbate up taken by the adsorbent, K_L, a is the Langmuir affinity constant. Langmuir equation used to evaluate thermodynamic parameters of the ongoing process and also to know the nature adsorption.

Freundlich isotherm The empirical Freundlich equation based on sorption on heterogeneous surface is given by equation:

$Q_e = K_f\, (C_e)^{1/n}$…..………(5)

Where K_f is the freundlich constant related with adsorption capacity $mg.g^{-1}\, (mg.L^{-1})^{-1/n}$ and n is the freundlich exponent (dimensionless).

This model is rearranged to the linear form by taking logarithms on both sides.

$\log Q_e = \log K_f + 1/n \log C_e$…..………(6)

Batch kinetic studies
Rate constant study

The specific rate constant for each adsorption process was calculated using Lagergren's first order rate expression [14]

$\ln (q_e-q_t) = \ln q_e - K_{ad}\, t$ ….……….(7)

Where q_e (mg.g^{-1}) and q_t (mg.g^{-1}) are the amounts of dye adsorbed at equilibrium and at any time t (min) respectively. K_{ad} is the rate constant of adsorption (min^{-1}).

Results and Discussion

Effect of initial dye concentration

The effect of the dye (BG) concentration on adsorption properties of the bentonite and Fe-BNC are shown in Table (1) and Figure (3). It is apparent that the percentage removal of dye increases with increase in initial dye concentration [15], probably due to the greater availability of exchangeable sites for the adsorbents. Nonetheless, adsorbate saturation of the adsorbent sites may occur on increasing concentration of the dye solution causing no further adsorption of the dye molecules.

As seen from the values of removal efficiency of the two adsorbents the R% values of the Fe-BNC larger than that of the bentonite clay. This indicates that Fe-BNC is effective for the removal of (BG) from aqueous solution

Effect of temperature

Fig. (4) shows the sorption kinetics of BG removal at 283, 293 and 303 K by plotting the dye uptake capacity q_t versus time at the initial dye concentration of (10 ppm). Increasing the temperature reduced the sorption capacity of Fe-BNC clay. Thus, when increasing the temperature from 283 K to 303 K the removal of dye decreased, this may be due to a tendency for the dye molecules to escape from the solid phase to the bulk phase with an increase in temperature of the solution [16].

During kinetic study it has been seen that the removal of dye was rapid in the initial stages of constant time and gradually with time until equilibrium. This decreasing removal rate towards the end, suggests formation of monolayer coverage of dye molecules on the outer surface of the adsorbent and pore diffusion onto inner surface of the adsorbent particulars through the film due to continuous agitation maintained during the experiments [17].

The rate constant for the adsorption of dye on modified clay was determined using Lagergren equation [7]. The kinetic data of adsorption BG (C_o= 10 ppm) onto the modified clay was shown in Table (2).

The straight line plots of (ln q_e-q_t versus t), Fig. (5) confirm that process of removal is governed by first order kinetics. The values of K_{ad} were determined by slops the graphs of Fig. (5) were listed in Table (3). All the fits show very good correlation coefficients. Fig. (6) shows a linear relationsship between the logarithm of rate constant and the reciprocal of temperature. The activation energy for the adsorption process E_a, was calculated using the Arrhenius equation [18]

$\ln K_{ad} = \ln A – E_a/RT$ ….……….(8)

Where A is referred to as the Arrhenius factor. R is the universal gas constant (8.314 J.mol^{-1}.K^{-1}), T is the absolute temperature (K). The rat constant K_{ad} listed in Table (3) was applied to estimate the activation energy of the adsorption. A value of (11.626 K.J.mol^{-1}) for Ea was obtained from the slop of an ln K_{ad} versus 1/T plot with a R^2 of 0.981. Because the value of E is high, it is concluded that the adsorption kinetic of (BG) onto Fe-BNC involved a chemical reaction in the adsorption process

Adsorption isotherm

The linear plots of C_e/Q versus Ce show that the adsorption obeys Langmuir isotherm model [19], Fig. (7), and indicates formation of mono layer of the dye around adsorbent particles and once a dye molecule occupies a site, no further adsorption takes place at that site. K_L and a were determined from the slop and intercept of the plot and are presented in Table (4).

The essential characteristic Langmuir isotherm can be expressed by a dimensional constant called equilibrium parameter, R_L [20] that is defined by

$$R_L = \frac{1}{1+K_L C_o} \text{.........(9)}$$

Where, K_L is the Langmuir constant at ($L.mg^{-1}$) and Co is the initial concentration.

The value of R_L indicates the shape of the isotherm to be either unfavorable ($R_L > 1$), linear ($R_L = 1$), favorable ($0 < R_L < 1$) or irreversible ($R_L = 0$). As shown in Table (5), R_L value decreases with the concentration and its values between 0 and 1 at different concentrations indicate favorable adsorption of dye onto Fe-BNC clay. linear plot of ln Q_e versus ln C_e shows that the adsorption follows freundlich isotherm model as well Fig. (8), K_f and n were calculated from the intercept and slop of the plot. The fitting results, i.e isotherm parameters and the coefficients of determination, R^2 are shown in Table (4). It can be seen in Fig. (8) that freundlich isotherm fits the data better than Langmuir isotherm. This is also confirmed by the high value of R^2 in case of freundlich (0.99) compared to Langmuir (0.962).

The freundlich constant n is primarily related to the strength of adsorption. Given fixed C_o and K_f the smaller the constant, the stronger the adsorption bond. As shown in Table (4), the (n) values of all the clays sample are less than (1), indicating that for this adsorbent the increased adsorption would promote the clay sorption capacity, and predominant adsorption mechanism would be chemical adsorption rather than physical adsorption [21].

The value of K_f increased on the increase of temperature indicating that higher temperature favored BG sorption on to Fe-BNC clay [22]. Fig. (9): Shows that the adsorption isotherms of BG on Fe-BNC clay is S-type according to the Giles classification.

Thermodynamic parameters

To calculate thermodynamic parameters of the ongoing process, another Langmuir form equation used [23]:

$$\frac{C_e}{Qe} = \frac{1}{b} + C_e \text{...........(10)}$$

Where b is the maximum adsorption quantity for dye solutions at different temperatures and could be obtained from the plot of C_e/Q_e vs. C_e fig (7)

The relation between log b and 1/T fig (10) is used to estimate ΔH and ΔS using the following formulas:

$$b = a \exp\left(-\frac{\Delta H}{RT}\right) \dots\dots\dots..(11)$$

$$a = \exp(\Delta S/R) \dots\dots\dots(12)$$

$$\log b = \frac{\Delta S}{2.303R} - \frac{\Delta H}{2.303R}\frac{1}{T} \dots\dots\dots..(13)$$

The ΔG value was calculated from the following equation:

$$\Delta G = \Delta H - T\Delta S \dots\dots\dots\dots..(14)$$

The estimated thermodynamic parameters are listed in Table (6). The positive value of enthalpy change (ΔH) confirms an endothermic nature of the enduring process whereas the negative values of free energy (ΔG) suggest feasibility and spontaneous nature of the process. The values of (ΔG) decrease with the increase of temperature indicated that the adsorption was more favorable at higher temperature. Positive values of entropy (ΔS) revealed that the adsorption process was irreversible and random at the solid/liquid in interface during the sorption of BG onto and Fe-BNC and showed that a good affinity of the adsorbent materials towards the dye, also the positive value of ΔS suggested some structural change of BG and Fe-BNC clay, and favored complexion and sorption stability [24]. Similar results were also observed on the adsorption of some dyes on to de-oiled soya [25], bentonite [26], neem leaf [27] and hen feathers [28].

Conclusion

It can be concluded that Fe-NBC can be successfully used as low cost adsorbents for removal of BG from waste waters. Equilibrium data were fitted to freundlich and Langmuir adsorption isotherm models.

The thermodynamic parameters indicated that the adsorption is feasible, endothermic and spontaneous in nature. Kinetic studies were verified for lagergrens first order, and the adsorption of (BG) follows first order Kinetics more.

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Table (1): Removal efficiency for the adsorbents used in the adsorption of BG.

Co (ppm)	5	10	15	20	25
R% for bentonite	91.24	95.56	96.62	97.47	97.90
R% for Fe-BNC	97.63	98.42	98.70	99.05	99.20

Table (2): Kinetic data of adsorption (10 ppm) BG dye onto Fe-BNC at temperature range (283-303) K.

Temp. (K)	Time (min)	q_e x 10^{-3}	q_t x 10^{-3}	q_e-q_t x 10^{-4}	(ln q_e - q_t)
283	5 10 15 20 30	9.45	9.29 9.33 9.37 9.39 9.45	1.60 1.20 0.80 0.60 0	-8.70 -9.03 -9.43 -9.72
293	5 10 15 20 30	9.41	9.265 9.30 9.34 9.371 9.41	1.45 1.10 0.70 0.39 0	-8.839 -9.115 -9.567 -10.1519
303	5 10 15 20 30	9.364	9.251 9.266 9.295 9.331 9.364	1.13 0.98 0.64 0.33 0	-9.088 -9.231 -9.660 -10.31

Table (3): Rat constant at different temperatures

T/K	283	293	303
K_{ad} (min.$^{-1}$)	0.0668	0.0805	0.088
Ea (K.J.mol^{-1})		11.626	

Table (4): Freundlich and Langmuir parameters for BG adsorption on Fe-BNC clay

T(K)	Langmuir isotherm			Freundlich isotherm		
	K_L (L/mg)	a (mg/g)	R^2	K_f (mg.g^{-1})	n	R^2
283	0.1886	3.562	0.923	0.0962	0.7174	0.998
293	0.2288	3.994	0.914	0.1011	0.7233	0.999
298	0.2323	3.919	0.962	0.1036	0.7219	0.999
303	0.2541	4.055	0.953	0.1050	0.7179	0.998

Table (5): Analysis of Langmuir isotherm at 283 K

Initial dye concentration Co (ppm)	5	10	15	20	25
R_L	0.5256	0.3585	0.2763	0.2096	0.1699

Table (6): Thermodynamic parameters for the adsorption of BG onto Fe-BNC

T(K)	ΔG ($J.mol^{-1}$)	ΔH ($J.mol^{-1}$)	ΔS ($J.mol^{-1}.K^{-1}$)
283	-2976.7	5169.7	28.786
293	-3264.5	=	=
298	-3408.5	=	=
303	-3552.4	=	=

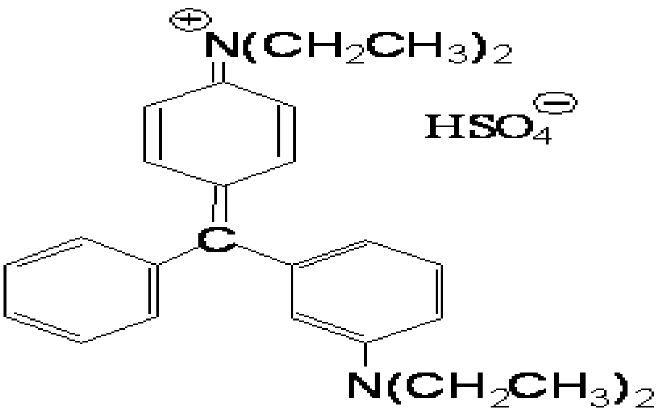

Fig. (1): The chemical structure of birllint green sulfate.

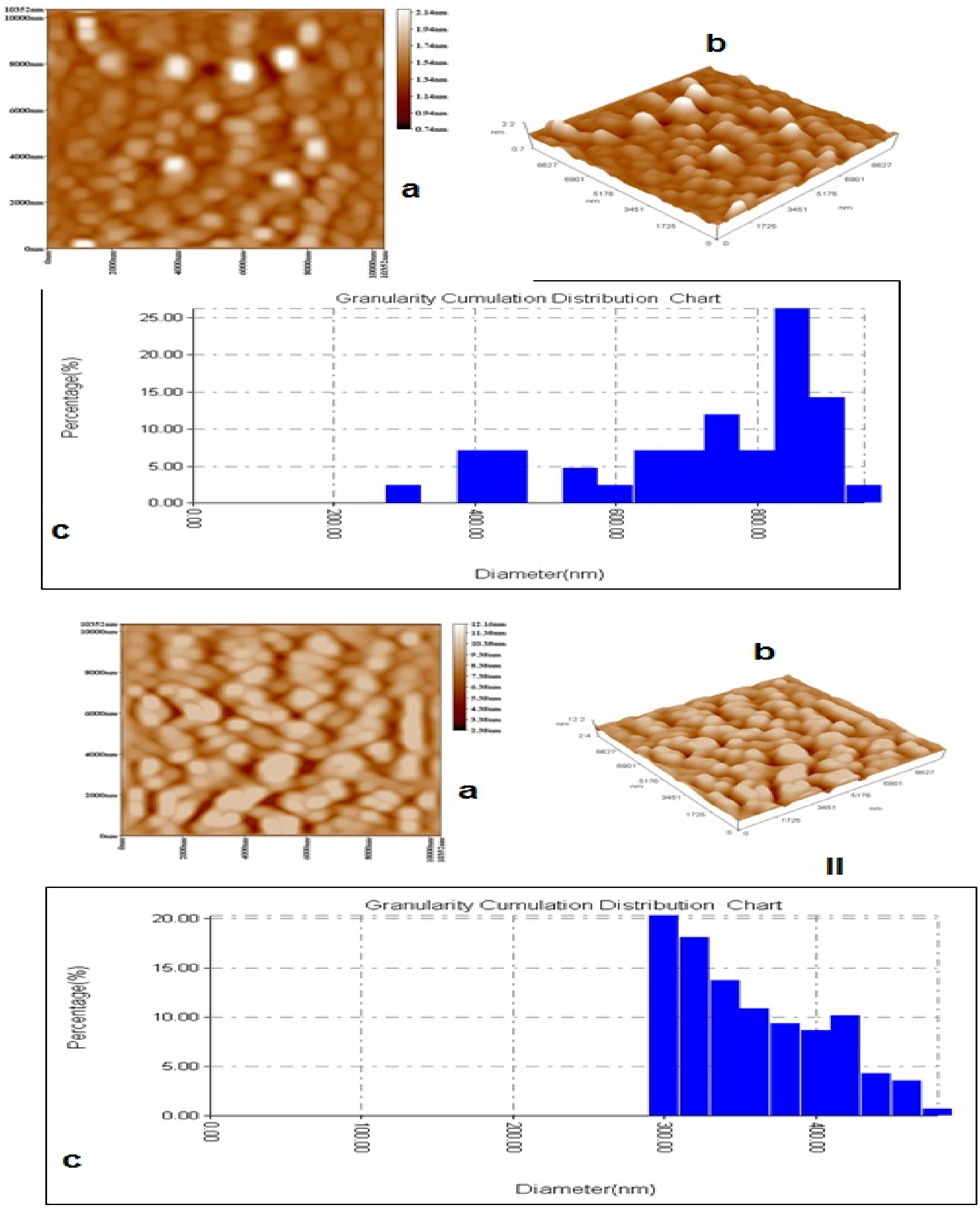

Fig. (2):(I) AFM images of clay. (II) AFM images of Fe-BNC clay
(a)The 2D, cross section (b) 3D and (c) Pore size distribution diagram

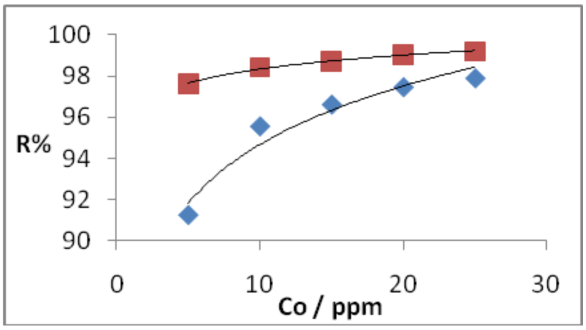

Fig. (3): Effect of initial dye concentration on equilibrium adsorption of BG on to (I) bentonite (II) Fe-BNC

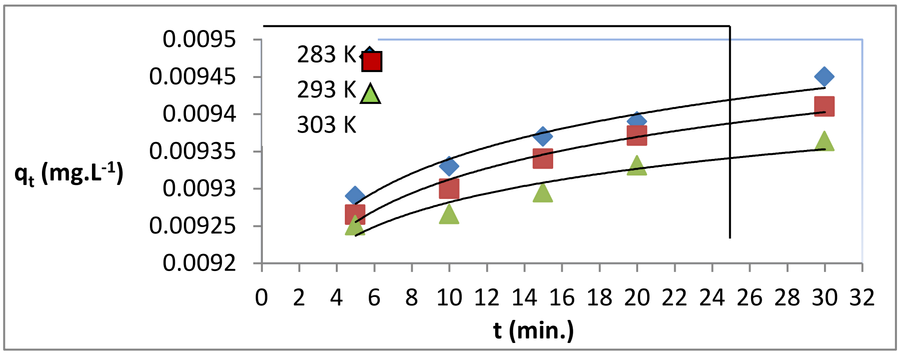

Fig. (4): Kinetics of BG uptake by Fe-BNC at several initial temperatures

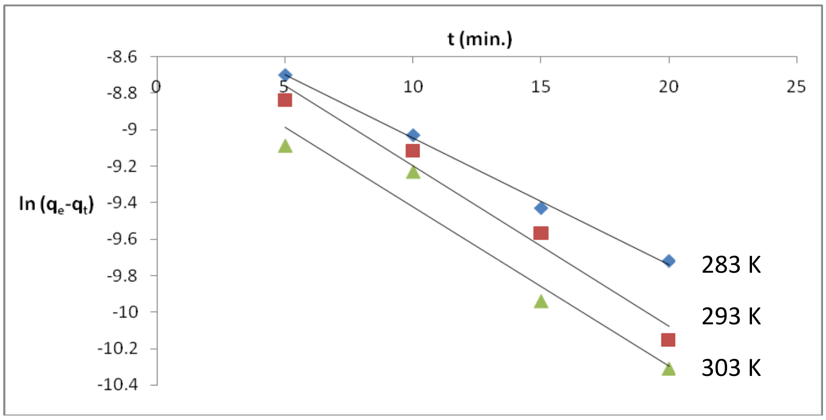

Fig. (5): Lagergrens plot for kinetic modeling of the adsorption process of BG on Fe-BNC clay at different temperatures

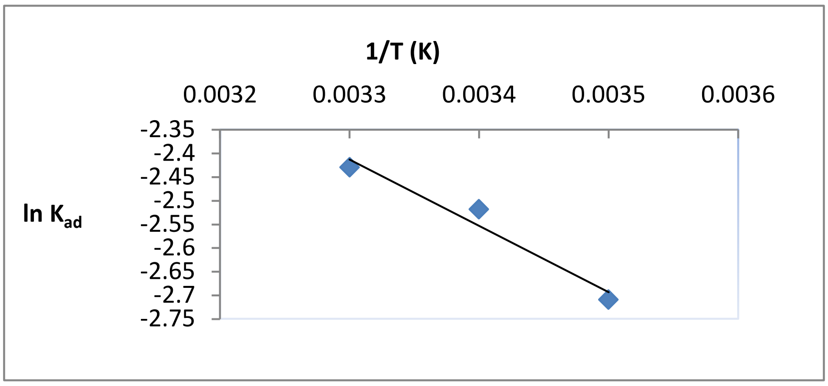

Fig. (6): Arrhenius equation of adsorption of BG on Fe-BNC

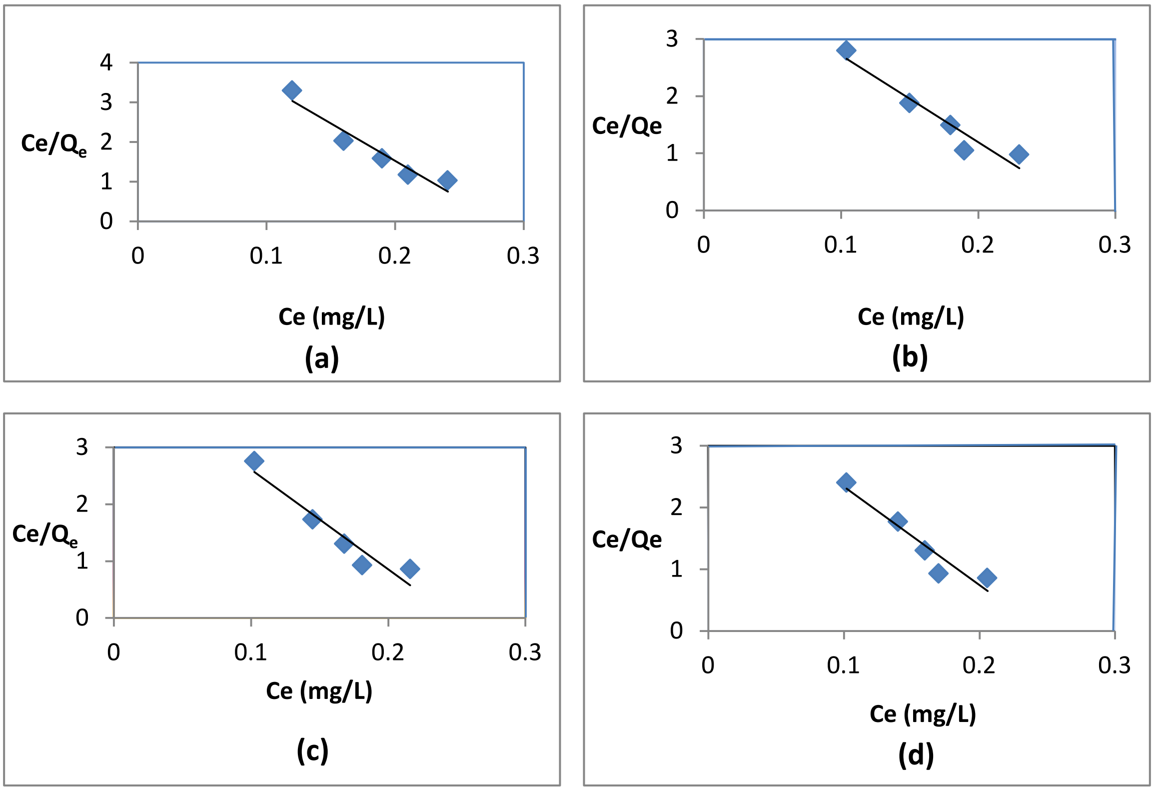

Fig. (7): Langmuir's isotherm plot for the adsorption of BG on Fe-BNC [(a) 283, (b) 293, (c) 298, (d) 303K]

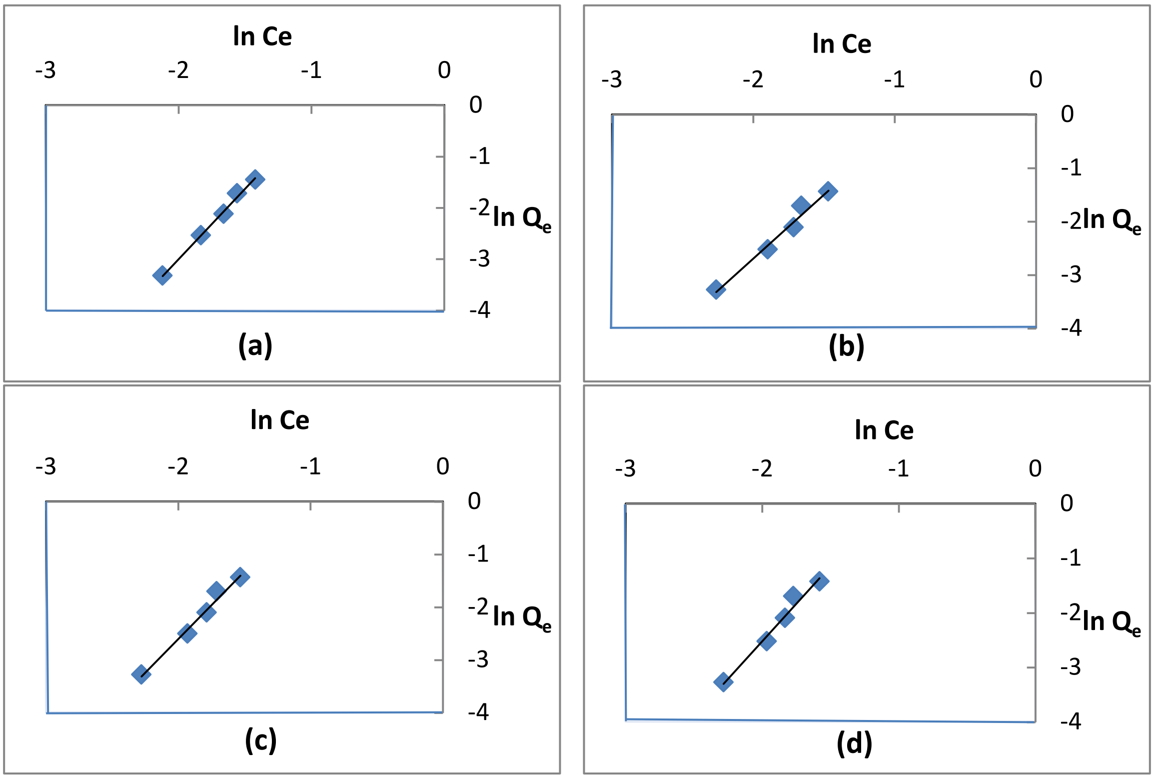

Fig. (8): Plot of Freundlich adsorption isotherms of BG on Fe-BNC [(a) 283, (b) 293, (c) 298, (d) 303K]

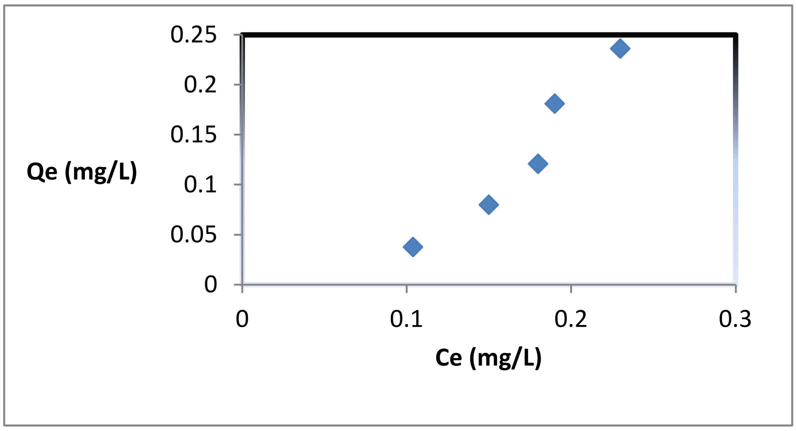

Fig. (9): Adsorption isotherm of BG on Fe-BNC at 298 K

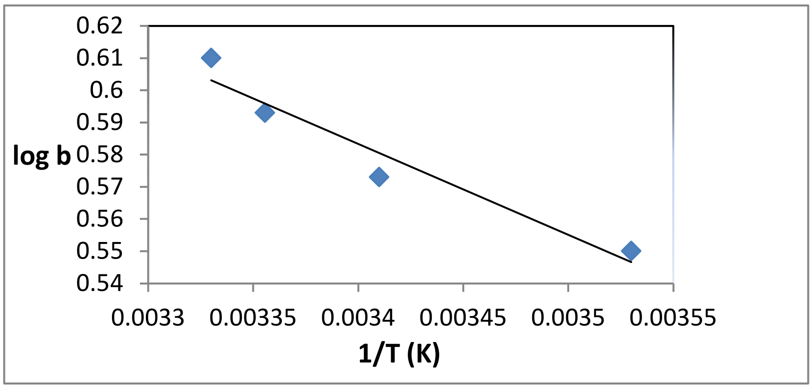

Fig. (10): Relation of log b against 1/T where T/K

ازالة الصبغة الخضراء اللمـــاعة من محاليلها المائيـة بإمتزازها على سطح الطين المحور

دنيـــا عيـــدان محمـــد
قســـم الكيميــــاء / كليـــة العلـــوم / جامعـــة بغــداد

استلم البحث في : 21 شباط 2013 ، قبل البحث في : 10 حزيران 2013

الخلاصة

درس استعمال استخدام طين البنتونايت النانوي كطين محور لازالة الصبغة الخضراء اللماعة من محاليلها المائية. واستخدم مجهر القوة الذرية لغرض الحصول على شكل المسام وتوزيعه ، وتبين ان معدل قطر المسام للطين المحور هو 346.81. واجريت وجبات من تجارب الامتزاز على سطح الطين المحور لازالة الصبغة من محاليلها المائية. وتبين ان الامتزاز يتبع متساوي درجة الحرارة لكل من لنكماير وفرندلش، واوجدت قيم الثوابت لكل منهما. وامكن حساب قيم الدوال الثرموديناميكية مثل الطاقة الحرة ، والانتروبي ، والانثالبي. واوجد ثابت السرعة لعملية الامتزاز بأستخدام معادلة لكركرين وتبين أن عملية الامتزاز تتبع حركية المرتبة الاولى فضلاً عن ذلك تم تعيين طاقة التنشيط لعملية الامتزاز.

الكلمات المفتاحية : الامتزاز، الصبغة الخضراء اللماعة، البنتونايت.