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LOW-COST ADSORBENTS FOR A DYE UPTAKE FROM CONTAMINATED WATER MODELING OF ADSORPTION ISOTHERMS: THE LANGMUIR, FREUNDLICH AND ELOVICH MODELS

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1. Introduction

Dyes are widely used in many industries, mainly in the textile industry. Their effluents often cause serious environmental problem due to a deep color present in the discharge water that requires pretreatment for color elimination prior to its disposal into the river. Since large amounts of metal complexes are present in the dyes, the majority of commercial dyes are treated with difficulty by the conventional biodegradation or chemical oxidizing method. A liquid- phase adsorption of dyes from discharge streams by various adsorbents becomes one of the major treatments for such wastewater because this process is simple, inexpensive, and easy to design. Activated carbon, due to its effectiveness, is the most widely used adsorbent. Many researches [1, 2] have conducted numerous studies on the adsorption of dyes onto activated carbon.

Besides the activated carbon, the use of other low-cost adsorbents has also been the focus of the recent research. These include silica gel, clays, sawdust, peat, and fly ash [3, 4]. The activated clay and montmorillonite are porous matrix material, mainly containing silica and aluminum. Both clay minerals are inexpensive. The activated clay and acid-treated activated montmorillonite have been widely used in the decoloration process in the cooking oil industry [5, 6]. However, their use for the adsorption of dyes in aqueous solutions have not been well documented. Wu et al [7] and El-Geundi et al. [8, 9] reported that the basic dye can specifically be adsorbed onto activated clay. Hsu et al [10] found the activated clay to have an excellent affinity for dyes, and the degree of adsorbability depends really on the species of basic dyes, the temperature, and the relative concentrations of the adsorbate and adsorbent.

Clays of bentonite type are largely used in many industrial products, such as ceramics, paintings, coatings, drilling mud, etc. The adsorption of macromolecules on the surface of these materials modifies their properties, particularly the rheological ones, as well as the stability of argillaceous suspensions. All this has a significant technological interest, since the based clay suspensions are effective adsorbents for the retention of polymer wastes in solutions. They are also less expensive than the usual adsorbents, such as activated carbon. Thus, they were the topic of the great number of studies, particularly when used for dye retention [11, 12, 13].

Hence, natural clays, particularly 2:1-type phyllosilicates, possess very good sorbent properties because of their large specific area. There has been an increasing interest in the past decade in the use of these materials to remediate environmental contamination, and so to attenuate the penetration of organic pollutants into the soil. The replacement of natural inorganic exchange cations with larger organic cations through the ion-exchange reaction has been shown to yield the organoclays with organophilic properties. Hence, this simple modification has been proposed to improve the sorptive capacity of clays for organic compounds [14, 15]. The purpose of this investigation was to examine the equilibrium adsorption data by some theoretical models (the Langmuir, Freundlich and Elovich models) in order to study the mechanism of the dye (Quinalizarin) adsorption into Tunisian clay samples before and after some amendments.

2. Experimental and methods

2.1. Clay adsorbents

Bentonite employed as an adsorbent in the present study, collected from Zaghouan (North-eastern of Tunisia), was prepared as described earlier [16]. The most important properties are illustrated in Table 1.

In order to prepare a homoionic clay in Li^+, Na^+, K^+, NH_4^+, Ca^{2+}, Mg^{2+}, Fe^{3+} and Al^{3+} bentonite, the

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natural bentonite, which is first of all saturated with Na using 1M NaCl, was used to obtain M^{n+} saturated clays (M^{n+} are exchangeable cations) by treating the clay fractions with 1M chloride or nitrate (analytical grade) solution 7 times at 12 h intervals [17]. The most important properties of the eight produced clay adsorbents (bent-M^{n+}) are summarized in table 2.

Table 1. Most important properties of the used sample

$S_{BET}(m^2g^{-1})^{*}$	$S_S(m^2g^{-1})^{**}$	CEC (meq/100g)***	$V_{tot}(cm^3/g)^{****}$
72.2	504	89	0.00581
Structure formula: $\mathbf{Ca_{0,043}Na_{0,404}K_{0,16}(Si_{7,56}Al_{0,44})(Al_{2,933}Fe_{0,678}Mg_{0,476})O_{22}}$			

$^{*}S_{BET}$ is the surface, **Ss is the specific surface,
***CEC is the cation exchange capacity, **** V_{tot} is the total pore volume

We note that the chemical analyses of the samples were obtained using the X-ray diffraction studies (Philips Goniometer, PW1730/10, Cu Kα radiation). The cation exchange capacities (CEC) were estimated using the Kjeldhal method [18], the BET surface area (S_{BET}) and the total pore volume (V_{tot}) of the each sample measured according to (N_2, BET) method.

Table 2. The most important properties of produced clays

Samples (bent-M^{n+})	S_{BET} (m^2g^{-1})	CEC meq/100g	V_{tot} (cm^3/g)
bent-Na^{+}	72.2	61.5	0.00581
bent-NH_4^{+}	93.8	65.92	0.0551
bent-K^{+}	117	69.65	0.0264
bent-Li^{+}	101	72.3	0.0158
bent-Mg^{2+}	92	69	0.0112
bent-Ca^{2+}	82	65.38	0.00921
bent-Fe^{3+}	120	74	0.026
bent-Al^{3+}	122	64.8	0.019

2.2. Sorption experiments

In order to efficiently decompose and detoxify Quinalizarin (Quin), a toxic organic substance ($C_{14}H_8O_6$;, fig. 1) used to dye cotton, from industrial effluents, it was necessary to concentrate the substance in same way. Accordingly, the synthesized bentonites (homoionic) were used to remove this dye (Quin) from the textile industrial water owing to their high cation exchange capacity and specific surface area.

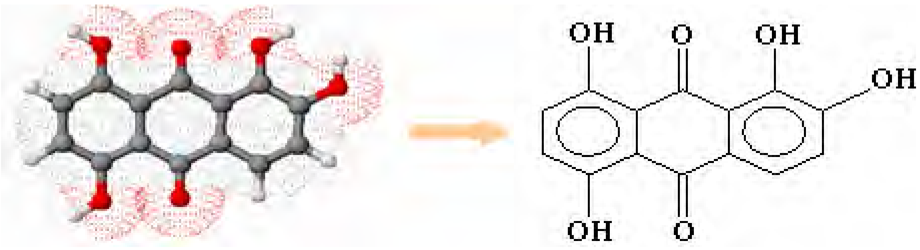


Fig. 1. Chemical structure of Quinalizarin

2.2.1. Adsorbate solution

The stock solutions containing 2.2×10^{-4} and 2.2×10^{-3} M of Quin, which amount to the cation exchange capacity (CEC) of 10% and 100% of the clay adsorbent at pH = 5, respectively, were prepared in the ethanol as eluent. We reported that Narine and Guy (1981) [19] suggested that the adsorption of various organic molecules was essentially independent of pH in the range of 4.5 to 8.5.

2.2.2. Batch adsorption

The adsorption experiments were carried out in 100 ml Erlenmeyer flasks by mixing 0.1 g of the clay adsorbent with the fixed volume (50 ml) of Quin of variable concentration. The mixture was shaken at room temperature on a reciprocating shaker for 1h. Previous studies indicated that this time period was adequate for the solutions to achieve the equilibrium state. After shaking, the supernatant was separated by centrifugation at 4000 rpm for 20 min. The residual dye concentration of Quin remained unadsorbed in the supernatant liquid was measured according to the UV and visible spectra, and its adsorbed amount was determined. By plotting this amount (Q_{ads}) as a function of the equilibrium concentration of the required substance (C_e), the isotherm of the each sample was obtained. All adsorption experimental measurements were duplicated. We note that the UV detector (multi-wavelength UV detector Waters 490) was adjusted at the wavelength of the maximum adsorption, λ_{max} = 238.29 nm.

2.2.3. Theoretical foundation

The adsorption equilibrium is usually described by an isotherm equation [20], whose parameters express the surface properties and affinity of the adsorbent at a fixed temperature and pH. The adsorption process is usually described by the following widely used isotherms [21, 22].

a) Freundlich isotherm

$$x/m = k_f.C_e^{\,n}$$

b) Langmuir isotherm

$$C_e/(x/m) = 1/a + b/a.C_e$$

where C_e is the equilibrium concentration of the solute (g.L^{-1}); x/m is the amount of the solute adsorbed per unit weight of the adsorbent (g.g^{-1} of clay); a and b are the Langmuir coefficients representing, respectively, the equilibrium constant for the adsorbate - adsorbent equilibrium and the monolayer capacity; k_f and n are the sorption constants of the Freundlich equation.

The linear Freundlich and Langmuir plots are obtained by plotting log x/m versus log C and C/(x/m) versus C, respectively, from which the adsorption coefficients could be evaluated.

The Langmuir equation is also used to obtain R_L, the dimensionless equilibrium parameter or the separation factor [23], from the expression:

$$R_L = 1/(1+aC_0),$$

where C_0 is the initial concentration of the adsorptive. For favorable adsorption, $0<R_L<1$; while $R_L>1$; $R_L=1$ and $R_L=0$, respectively, describe unfavorable, linear and irreversible adsorption [24].

c) *Elovich equation* [25], $(x/m)/Q_m = K_E\, C_e\, e^{-((x/m)/Q_m)}$, is also used successfully to describe the second order kinetics assuming that the actual solid surfaces are energetically heterogeneous [26, 27, 28].

The linear form is $\ln (x/m)/C_e = \alpha\,(x/m) + \beta$, Q_m is the maximum amount of the adsorbate adsorbed in the solid phase (mg/g), k_E, α and β are the Elovich coefficients.

3. Results and discussion

3.1. X-ray diffraction

The XRD patterns of the each sample (monoionic bentonite) before and after intercalation with dye molecules (Quinalizarin) were recorded and the positions of 001 reflection were registered.

In table 3 and fig. 2 the interlayer expansions (Es) are presented before and after the intercalation of this organic molecule in the each sample.

Compared with the d_{001} of untreated clay, that is prior the intercalation, which appears to be around 12 Å, it was suggested that quinalizarin molecules intercalate into the interlayer of the each sample with a monolayer arrangement, thus leading to the larger interlayer expansion.

Table 3. The interlayer spacing (Es) before and after intertercalation of dye molecules

Bent-M^{n+}	Bent-Na^+	Bent-k^+	Bent-Li^+	Bent-NH_4^+	Bent-Ca^{2+}	Bent-Mg^{2+}	Bent-Fe^{3+}	Bent-Al^{3+}
Es before intercalation (nm)	0.30	0.12	0.26	0.23	0.54	0.51	0.50	0.47
Es after intercalation (nm)	0.53	0.51	0.46	0.46	0.56	0.54	0.56	0.56

According to the published data [29], the organic molecules fit between the clay layers in two different ways (fig. 3):

1) Parallel arrangement:
  a -only one layer
  b - two layers
2) Arrangement at right angles or tilted:

a - only one layer
b- two layers

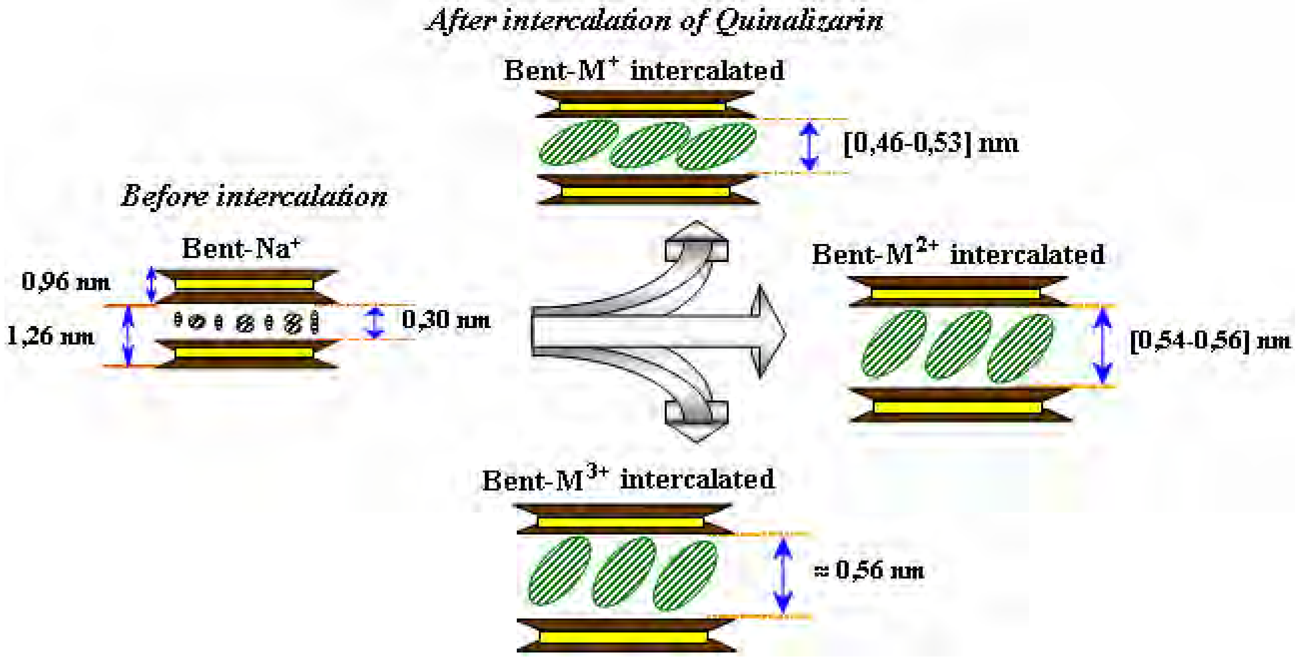


Fig. 2. Cartoon of interlayer spaces expansion (Es) before and after intercalation of organic molecule (quinalizarin); bent: bentonite, M^{n+}: exchangeable cation

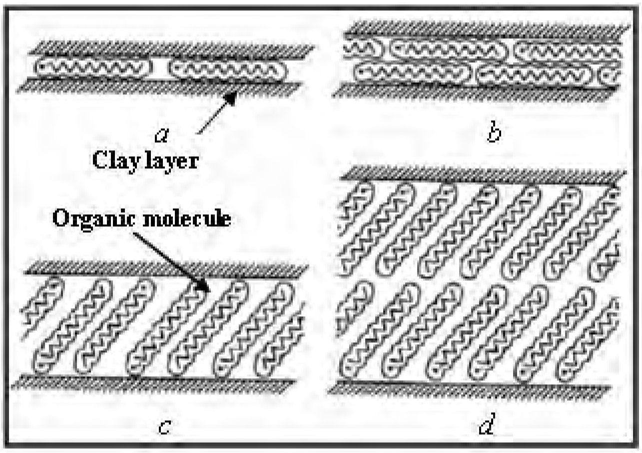


Fig. 3. Arrangement of organic molecule between clay layers

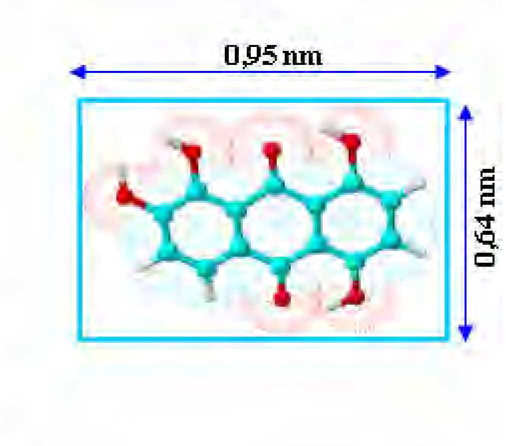


Fig. 4. Quinalizarin dimensions determined by software model MOPAC [30] *(Dewar 1985)*

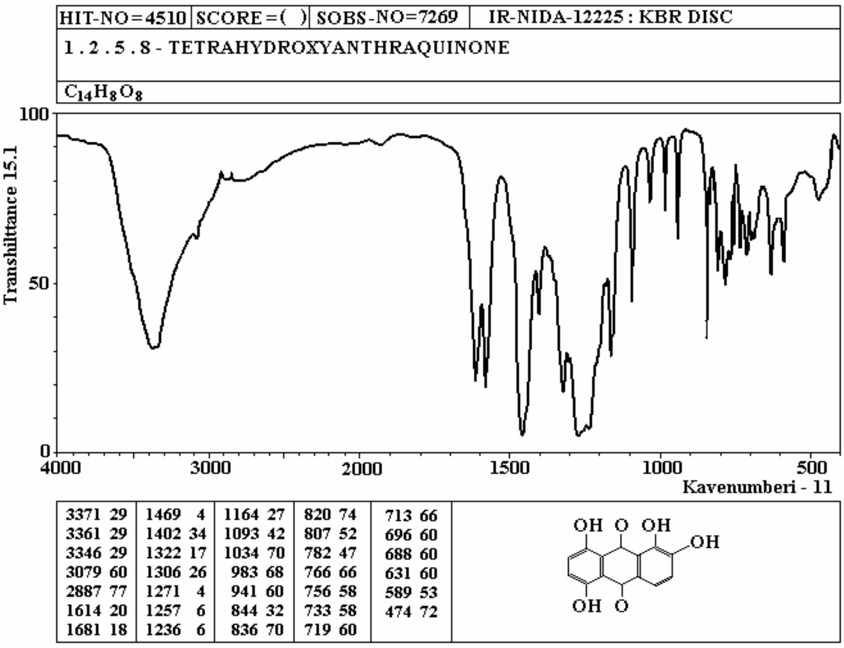
According to the software model CS Chem3D MOPAC [30] (the Dewar theory, 1985), this molecule has the length of 0.95 nm, the height of 0.64 nm and the thickness of 0.5 nm (fig. 4). The expansion of interlayer spacing before and after intercalation allows to deduce that the molecule inserts parallel to the clay layers with a small slope with only one layer.

Nevertheless, the XRD does not allow to include/understand the mechanism of the exchange phenomenon which occurs, or to determine the maximum quantity of quinalizarin adsorbed by the each sample. So, we had to apply other techniques, such as the infrared spectroscopy and UV-visible spectrophotometry.

3.2. Infrared analysis

The infrared spectra were recorded with Perkin-Elmer 783 dispersion spectrometer in the range of 4000 to 400 cm^{-1}. The samples were prepared in the form of KBr pellets by mixing 150 mg of KBr and 8 mg of phyllosilicate.

The infrared spectra of pure Quin (fig. 5) and the clay samples before and after intercalation (fig. 6) enhance the quinalizarin molecule into the clay interlayer. The presence of quinalizarin between the clay layers was detected by a slight shoulder at 1480–1580 cm^{-1} (the stretching C=C vibration in the aromatic ring) and a slight peak at 1600 cm^{-1} (the stretching C=O vibration in the aromatic ring), which do not appear in the IR spectra of the samples before intercalation and are the principal vibrational bands of this organic molecule. The vibration modes of the adsorbed quinalizarin occur in the range of 2000-1000 cm^{-1} (table 4).



HIT-NO=4510 | SCORE=( ) | SOBS-NO=7269 | IR-NIDA-12225 : KBR DISC

1 . 2 . 5 . 8 - TETRAHYDROXYANTHRAQUINONE

$C_{14}H_8O_8$

3371 29	1469 4	1164 27	820 74	713 66
3361 29	1402 34	1093 42	807 52	696 60
3346 29	1322 17	1034 70	782 47	688 60
3079 60	1306 26	983 68	766 66	631 60
2887 77	1271 4	941 60	756 58	589 53
1614 20	1257 6	844 32	733 58	474 72
1681 18	1236 6	836 70	719 60	

Fig.5. Theoretical IR Spectra of pur Quinalizarin

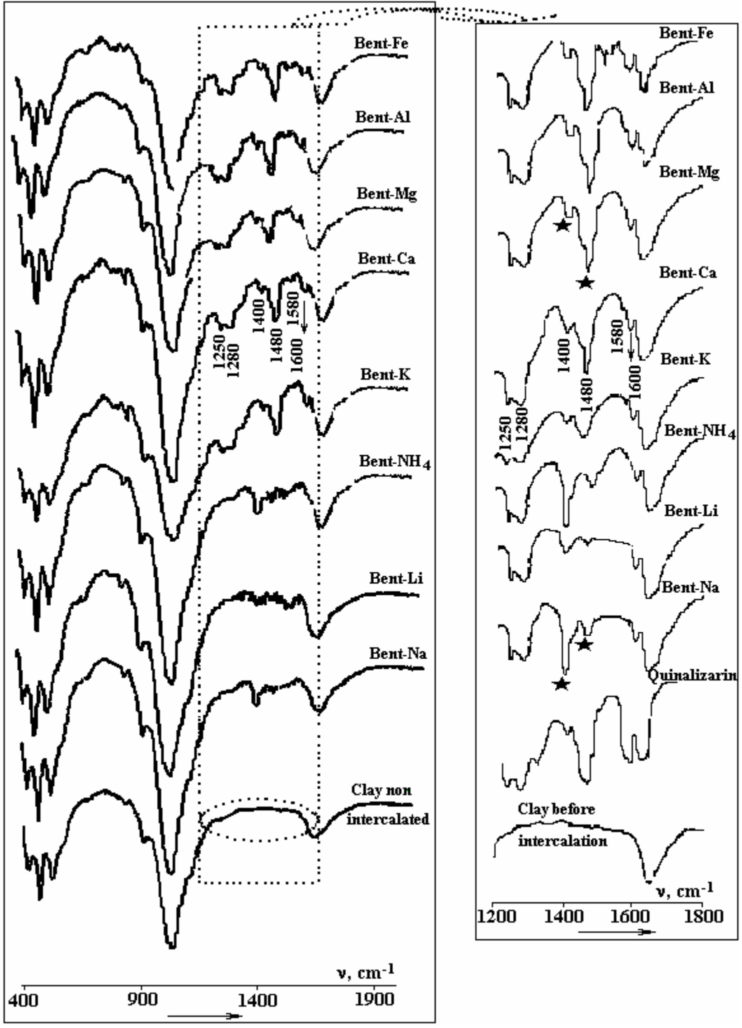


Fig. 6. IR spectra of pure Quin and of clay samples before and after intercalation of Quin (ben-M^{n+}, M^{n+} : exchangeable cation)

The vibration wavenumbers of the adsorbed quinalizarin onto the ion-exchanged smectites are found to be almost the same. However, we note a reversed in intensity vibration bands, which appear at 1480 and 1400 cm^{-1} on the level of the IR spectra of the samples saturated with monovalent cations (Na, NH_4 and Li), since the band around 1480 cm^{-1} becomes the least intense.

The IR spectroscopic studies of the adsorption of Quin by the clay adsorbents indicated that certain modes of the aromatic molecule, particularly the ring modes around 1600–1400 cm^{-1}, are very sensitithe oxygen atom to the Lewis acidic sites or to the exchangeable cations (direct or indirect through water bridges).

Table 4 . Vibration bands of Quinalizarin intercalated in the homoionic smectite

Attribution	$\nu(C_{ar}\text{-}O)$	$\nu(Ar\text{-}H)$	$\nu(C{=}C)_{Ar}$	$\nu(C{=}O)$	δ_{OH}
Vibration bands	1250-1280	****	1480-1580	1600	1400

3. Adsorption procedure

In the isotherm experiments, a fixed volume (50 ml) of quinalizarin with a variable concentration was equilibrated with 100 mg of a homoionic smectite powder prepared as described earlier [31] in centrifuge tubes. The mixture was shaken on a reciprocating shaker at the temperature of 24°C for 1 h. The previous studies indicated that this time period was adequate for the solution to achieve the equilibrium state. After shaking, the supernatant was separated by centrifugation at 4000 rpm for 20 min. The residual dye concentration of quinalizarin remained in the solution was measured according to the UV and visible spectra and the adsorbed amount was determined. By plotting this amount (C_{ads}) as a function of the equilibrium concentration of the required substance (C_{eq}), the isotherm of the each sample was obtained (fig. 7). All the adsorption experimental measurements were duplicated. We note that the UV detector (multi-wavelength UV detector Waters 490) was adjusted at the wavelength of maximum adsorption, λ_{max} = 238.29 nm using the ethanol as the eluent, and that the initial dye concentration was chosen to be equal the cation exchange capacity (CEC) of the clay fraction (2.2 x 10^{-4} M) prepared at pH 5.

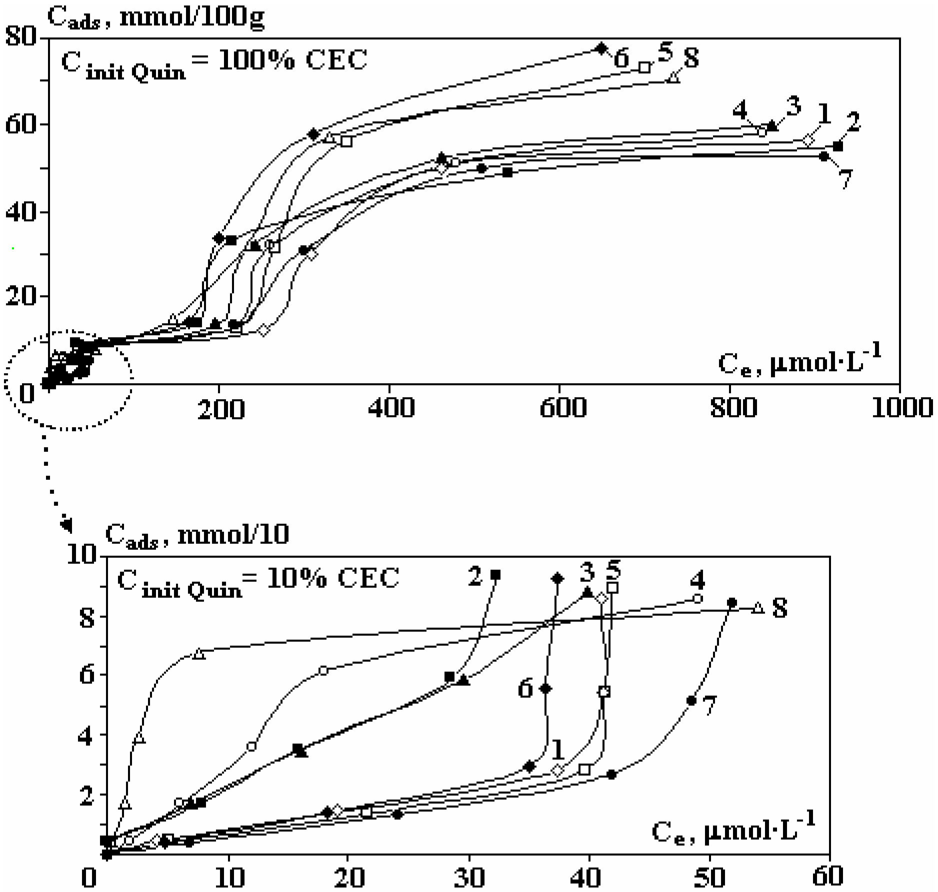


Fig. 7. Adsorption isotherms of Quinalizarin by bent-M^{n+}. 1 – Ca; 2 – Mg; 3 – Fe; 4 – Li; 5 – NH_4; 6 – Al; 7 – K; 8 – Na

We reported that the saturation level was reached, the high adsorption capacity (79 meq/100 g), close to the cation exchange capacity of the synthesized bentonite (89 meq/100g), indicates a strong interaction between the dye molecule and the adsorbent [32]. The lower specific adsorption is obtained using the bentonite saturated with potassium. The potassium ions suppressed quinalizarin adsorption to bentonite, probably by collapsing the clay lattices and preventing the expression of the interlayer-derived cation-exchange capacity. More quinalizarin was adsorbed by the bentonite made homoionic to various mono-, di- and trivalent cation [31], the sequence of the adsorption ability to the homoionic smectite was:
Bent-Al^{3+} >bent-NH_4^+ > bent-Na^+ > bent-Fe^{3+} > bent-Li^+ > bent-Ca^{2+} > bent-Mg^{2+} > bent-K^+.

As a result, the experimental equilibrium isotherms can be explained involving the cation exchange phenomena of quinalizarin in the clay interlayer space, since the maximum quantity of the adsorbed quinalizarin by the synthesized bentonite is close to the cation exchange capacity.

3.4. Modeling of the adsorbtion isoterms of Quinalizarin

The values of the adsorption coefficients computed from the Langmuir, Freundlich and Elovich plots, are given in table 4. Fig. 8 a, b, c depicts the the linear plots obtained according to the tree models (Langmuir, Freundlich and Elovich), respectively.

As can be seen in fig. 8, a,b,c and table 4, the experimental adsorption data for this metal fit with these tree models approximately in all the cases.

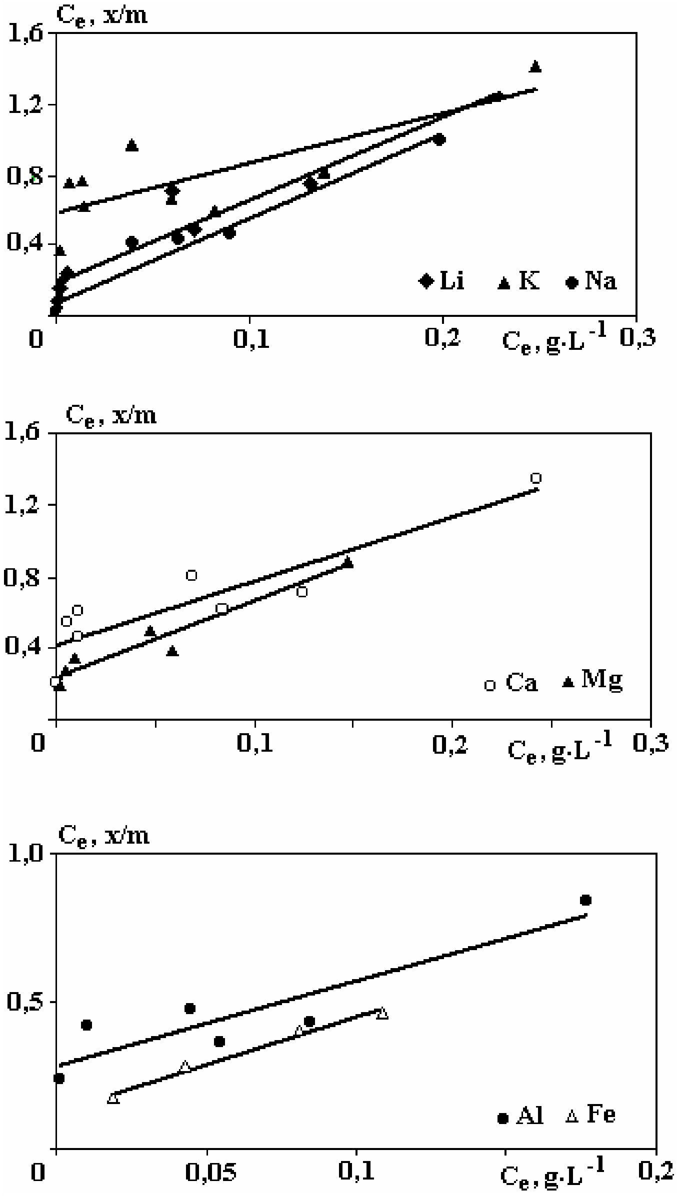


Fig.8$_a$. Langmuir plots for the adsorption of Quinalizarin by homoionic bentonite

We reported that the condition for the favorable adsorption is $0 < R^2 < 1$; while $R^2 > 1$ represents an unfavorable adsorption, and $R^2 = 1$ indicates the linear adsorption, as is depicted in Table 4; in all this cases $0 < R^2 < 1$. These tree models yield a good fit with the adsorption data and the adsorption coefficients agree well with the conditions supporting the favorable adsorption.

The adsorption data follow the empirical Freundlich isotherm (fig. 8,b) (R^2= 0.9–0.97), this isotherm does not yield any specific information about the mechanisms of adsorption related to the non-specific adsorption on heterogeneous solid surfaces. In terms of the Freundlich adsorption capacity, K_f, the highest value was found for bent-NH$_4$, and the lowest value for bent-k. The adsorption intensity given by the n should be less than unity and the values obtained in this work also confirm this fact (n = 0.3–0.8).

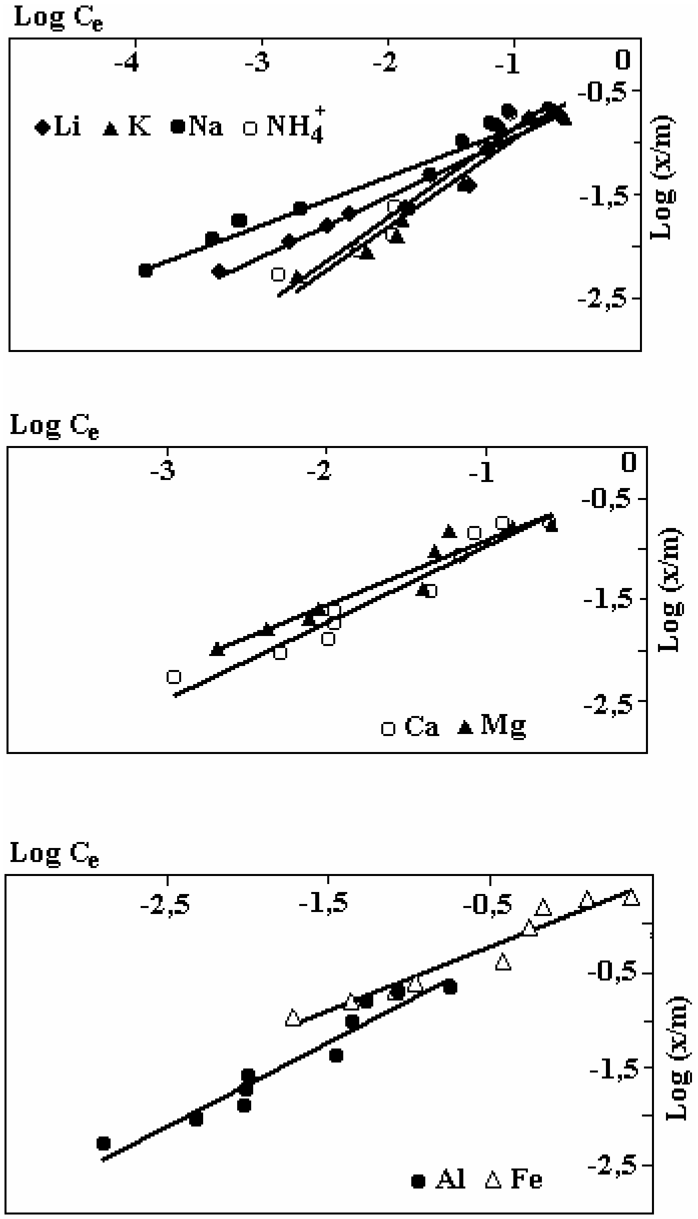


Fig. 8$_b$. Freundlich plots for the adsorption of Quinalizarin by homoionic bentonite

The Langmuir isotherm plots (fig. 8,a) have a little better linearity (R^2 = 0.9–0.99) compared to the Freundlich plots. The equilibrium coefficient b exhibits large values (b = 37.5–109.9 Lg^{-1}), which show that the equilibrium clay samples (a solid phase) + Quin (solution) = clay…Quin, is shifted predominantly to the right-hand side, i.e; towards the formation of the adsorbate-adsorbent complex. The Langmuir monolayer capacity a is appreciably large with its value from 8 to 26 Lg^{-1}. The separation factor R_L~ 0.999 in all the cases indicate that the adsorption of this molecule is favorable on the clay adsorbents; we can consider that the linear adsorption occurs, since $R_L \cong 1$.

The Elovich constants, α and K_E (table 7), depend significantly on the amount of the adsorbent with the adsorption rate constant α being much more sensitive. Since α represents the initial rate of adsorption, the results indicate that the rate could be enhanced many times by increasing the adsorbent amount, i.e. by providing a large surface area for the interaction. The Elovich equation has been found useful in describing predominantly chemical adsorption on highly heterogeneous adsorbents, since in all the cases R^2~0.9.

Table 5. Langmuir, Freundlich and Elovich parameters

Paramèters de Langmuir							
échantillon	*Na-bent*	*Li-bent*	*K-bent*	*Mg-bent*	*Ca-bent*	*Fe-bent*	*Al-bent*
a (Lg^{-1})	18	26	8	14	13	11	12
b (Lg^{-1})	43,96	37,5	109,9	68,13	67,14	42,43	39,38
R^2	0,93	0,99	0,99	0,99	0,95	0,77	0,9

Paramètres de Freundlich								
éch	*Na-bent*	*Li-bent*	*K-bent*	*Mg-bent*	*Ca-bent*	*Fe-bent*	*Al-bent*	*NH_4-bent*
K_F	0,033	0,157	0,03	0,127	0,113	0,035	0,037	0,975
n	0,8	0,32	0,577	0,33	0,367	0,7	0,7	0,75
R^2	0,97	0,9	0,97	0,95	0,9	0,9	0,91	0,94

Paramètres de Elovitch							
échantillon	*Na-bent*	*Li-bent*	*K-bent*	*Mg-bent*	*Ca-bent*	*Fe-bent*	*Al-bent*
K_E	575,2	59,53	16,4	30,48	9,622	2,366	18
α (g/mg)	17,37	9,521	6,8	8,25	5,144	0,744	5,188
R^2	0,91	0,93	0,95	0,92	0,94	0,94	0,93

4. Conclusions

The synthesized homoionic bentonites have a great tendency to remove Quinalizarin from solutions. The removal process is very rapid and spontaneous.

According to the Langmuir, Freundlich and Elovich equations, these homoionic bentonites have the highest capacity for adsorption, they have the greater potential for dye removal by adsorption mechanisms in an ion exchange form accompanied by the surface adsorption mechanism. These three models yield a good fit with the adsorption data, and the adsorption coefficients agree well with the conditions supporting the favorable adsorption

From the practical point of view, this clay exhibits a high potential to adsorb Quinalizarin and so other dyes, consequently any other pollutant, since its natural sources are available with high potentiality of use and low cost.

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Received 07.05.08

Summary

The adsorption of organic molecule to clay minerals has been reported by several investigators. Bentonite, which is made homoionics by mono-, di- and trivalent cations was used to remove the dye, an organic molecule (Quinalizarin) used to dye cotton. Equilibrium data were obtained by the batch technique. The results show the largest adsorption capacity of the homoionic bentonite; the saturation level was reached, the high adsorption capacity (79 meq/100 g), close to the cation exchange capacity of the synthesized bentonite (89 meq/100g), indicates a strong interaction between the dye molecule and the adsorbent. Adsorption onto the clay was essentially immediate and correlated with the cation exchange-capacity (CEC). The type of exchangeable cations indicate that adsorption was primarily to the negatively charged sites on the clay. The amount of the adsorbed color was found to be a function of the type of exchangeable cations; it drops off in the following order: Bent-Al^{3+} > bent-NH_4^+ > bent-Na^+ > bent-Fe^{3+} > bent-Li^+ > bent-Ca^{2+} > bent-Mg^{2+} > bent-K^+. The Langmuir, Freundlich and Elovitch models were employed to examine the equilibrium adsorption data. In all cases, the results show that all adsorption systems could be adequately fitted by these three models. It is seen that the linear fit is fairly good for these three models, the correlation coefficients R^2 was found to be equal to 0.9 in approximately all cases.
