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Adsorption of 2,4,6-trichlorophenol on bentonite modified with benzyldimethyltetradecylammonium chloride

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ABSTRACT

The objective of this study is to evaluate the performance and capacities of the bentonite of Maghnia, modified with benzyldimethyltetradecylammonium chloride, to remove the organic pollutant 2,4,6-Trichlorophenol (TCP). The modified sample was studied by X-ray diffraction (XRD) technique, infrared spectroscopy (FTIR), and thermogravimetric analysis (TGA) methods. The best removal rate (99.52%) was obtained at 19°C, pH 4, solution concentration of 50 mg/L, stirring speed of 180 rpm and contact time of 60 min. The results were well fitted by both Langmuir and Freundlich isotherm models and the pseudo-second-order is the best model to describe the process.

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Capsule Summary: The desorption tests showed that 7.24% of the TCP is released into the solution, confirming the effectiveness of adsorbent.

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INTRODUCTION

Natural bentonites are clay materials widely used for the removal of toxic metal ions, dyes, chlorophenols, medicaments (Al-Anber, 2010; Baghel et al., 2006; Bhattacharyya and Gupta, 2007; Putra et al., 2009; Zheng et al., 2008; Liu and Zhou, 2010). Challenge of chemical treatment and the cost of using sodium bentonite have led many laboratories to focus on the search for new adsorbents based on natural materials. In this context, we used crude bentonite in this study to promote an abundant adsorbent material based on Algerian clay for the treatment of polluted water.

The adsorption of organic pollutants by clays receives particular attention from researchers who have

focused on modifying the surface of this natural material by exchanging the inorganic interlamellar cations with organic cations such as quaternary alkylammoniums (Xi et al., 2007; Daoudi et al., 2013).

The modification of the bentonite by the surfactants increases the interlayer space and improves its adsorbent power, so organophilic bentonite can have various applications (Kaufhold et al., 2007; Yilmaz and Yapar, 2004; Zaghouane-Boudiaf and Boutahala, 2011; Zhu and Zhu, 2008).

This study focuses on the modification of crude bentonite (crude-B) from the Hammam Boughrara deposits the Algerian west (Maghnia) part by benzyl dimethyl tetradecylammonium chloride (BDTAC) and its use for elimination of the organic pollutant 2,4,6- trichlorophenol (TCP). There is little work done on the modification of the clays by the surfactant BDTAC for the elimination of the pollutants that is why we chose it for our study of adsorption while the choice of the molecule TCP was motivated by its toxicity because it has been classified by the EPA as one of the 21 most toxic pollutants (EPA, 2002). Particular attention has been paid to the TCP, judging by the countless work carried out for its elimination (Hameed et al., 2008; Zaghouane-Boudiaf and Boutahala, 2011), but to our knowledge, no research has been carried out on crude bentonite modified with BDTAC.

The modified clay was characterized by infrared (IR) spectroscopy to confirm the existence of molecules of organic nature and by thermogravimetric analysis (TGA) to evaluate the thermal stability of this material and by X-ray diffraction (XRD) to quantify the expansion of the interfoliar distance after intercalation of the BDTAC molecules. The kinetic and equilibrium data of adsorption studies were processed to understand the adsorption mechanism of TCP onto the modified clay.

MATERIAL AND METHODS

Chemical composition

The determination of the percentage content of the chemical composition of the bentonite was carried out using a SIEMENS X-ray fluorescence spectrophotometer (S.R.S. 3000). The results obtained are: 65.2% SiO₂, 17.25% Al₂O₃, 2.10% Fe₂O₃, 1.20% CaO, 3.10% MgO, 2.15% Na₂O, 0.60% K₂O, 0.20 % TiO₂ and 8.20% of loss on the ignition with 900 °C. The chemical composition shows that we have a tendency alumina silicate with a high SiO₂ content. The SiO₂ / Al₂O₃ report of bentonite which is equal to 3.77 is in agreement with that of montmorillonite which is between 2 and 5.5 (Bouyakoub, 2009; Mering, 1975).

The cation exchange capacity of the crude-B is 80 meq/100 g. The reactants used were BDTAC and 2,4,6-Trichlorophenol (TCP) were purchased from Sigma-Aldrich (99% purity) and used for the preparation of the solutions without any purification. 2,4,6-Trichlorophenol ($Cl_3C_6H_2OH$) has a molecular weight of 197.45 g/mol and BDTAC ($C_{23}H_{42}CIN$) has a molecular weight of 368.04 g/mol.

Preparation of the organophilic sample

An amount of Benzyldimethyltetradecylammonium chloride is dissolved in 0.5 liters of distilled water at 80°C, with stirring for 3 hours. A mass of 2.5 g of adsorbent was added to the surfactant solution. The concentration of surfactant is 2.0 CEC of the bentonite (the organoclay was washed free of chloride anions as determined by use of AgNO₃) and was dried at 80°C for 24 hours, then kept in the shelter from all external contamination. The kinetic study was realized by shaking the optimal mass of crude-B-BDTA, beforehand determined, in the aqueous solution of TCP to 50 mg /L.

Optimization of adsorption parameters

The parameters influencing TCP adsorption on crude-B-BDTA such as Temperature, pH, concentration of TCP

solution, contact time and mass of the modified bentonite were examined. The pH of solutions was adjusted by adding a few drops of concentrated solutions of HCl or NaOH 0.1M. The contact time was studied between 10 and 90 minutes at room temperature under an agitation speed of 180 rpm. Samples of 5 mL were centrifuged and the concentration of TCP was determined using a wavelength (λ max = 290.64 nm) spectrometer (Lambda 45-UV-Vis).

The adsorption percentage is calculated according to equation (1).

Adsorption (%) =
$$\left(\frac{C_i - C_e}{C_i}\right)$$
. 100 (1)

Where, Ci and Ce are the initial and equilibrium concentrations of TCP.

RESULTS AND DISCUSSION

Characterization

In X-ray diffraction profile of crude and modified bentonite shown in figure1, there are peaks of impurities minerals like quartz, cristobalite and feldspart. The crude bentonite has a d-spacing of 14.75 Å; the interlayer spacing increased to 28.23 Å when modified with BDTA. This indicated that the surfactant Benzyldimethyltetradecylammonium entered in the interlayer space of the bentonite by ion exchange.

Thermal analysis was used to explore the structure of the Crude-B-BDTA 2xCEC complex. Figure 2 shows the multiple steps of the material's decomposition; a significant loss of weight is observed near 50, 250, 370 and 700°C which can be attributed to desorption of physically adsorbed water and the dehydroxylation of the aluminosilicate layer, respectively. It was also noted that the degradation starts from 300°C, which shows that this material is suitable for any use in this temperature range. However, no loss of mass is observed between 700 and 850°C, which testifies to the Thermal stability at high temperatures.

The IR spectra of the Bentonite and the BDTAmodified Bentonite are shown in Figure 3. The existence of new absorption bands relating to the presence of the organic molecules in the Crude-B-BDTA sample confirms the actual insertion of the surfactant into the bentonite. The two bands 2850 and 2926 cm⁻¹, observed on organo-bentonite, can be assigned to the symmetric and antisymmetric stretching vibrations of the methylene group (Zaghouane-Boudiaf and Boutahala, 2011; Zhou et al., 2007). The band at 1650 cm⁻¹ is assigned to water bending modes within the clay interlayer (Boufatit et al., 2007). The bands at 3440 and 1630 cm⁻¹ are due to H-O-H stretching and bending vibrations of adsorbed water respectively (Yuehong et al., 2010). The strong attenuation of the intensity of the absorption bands relative to the OH group, indicate the replacement of the H₂O molecules by those of BDTAC. The presence of this band in organophilic materials may be due to the existence of OH groups in the polycationic interfoliar complexes. The absorption band at 1490 cm⁻¹ is attributed to ammonium ion



Fig. 1: X-ray diffraction spectrum of crude-B and the crude-B-BDTA



Fig. 2: TG curves of the crude-B and the crude-B-BDTA



Fig. 3: IR spectrum of crude-B and the crude-B–BDTA

(Akçay, 2004). Figure 4 shows the IR spectrum of the BDTAC surfactant alone.

Optimization of adsorption parameters

Influence of adsorbent mass

The results of the mass variation of the Crude-B-BDTA adsorbent (10-90 mg) indicate that the removal percentage is maximum (92.93%) for a 70 mg clay mass, but beyond this

value, the influence of the weight becomes almost constant as shown in Figure 5. The increase in the percentage of elimination can be explained by the fact that, when the mass of the adsorbent is increased, the specific surface area and the number of adsorption sites are increased and hence the elimination of the TCP.

Influence of initial TCP concentration on the adsorption

The effect of TCP concentration on adsorption capacity of modified clay was studied by carrying out a series of adsorption experiments with a constant mass of adsorbent (70 mg) and at different initial concentrations of TCP. As can be seen from Figure 6, the percentage adsorption is maximal for low TCP concentrations. This value decreases with the increase in the initial concentration of the pollutant but remains high (97.88%) for 50 mg/L. This phenomenon is due to the saturation of the adsorption site.

Influence of contact time

Results of the adsorption kinetics of TCP by the modified clay are represented as a percentage of adsorption depending on the stirring time, Figure 7. According to the plot, adsorption of TCP occurred very quickly from the beginning of the experiments within 15–20-min probably this is due to the availability of the surface sites of the organoclay for adsorption of molecule TCP and the maximum adsorption was reached within 60 min with an adsorption rate of 99.46%. At the beginning, the TCP ions were adsorbed by the exterior surface of adsorbent, the adsorption rate was fast. When the adsorption of the exterior surface reached saturation, the TCP ions entered into the pores of adsorbent and were adsorbed by the interior surface of the particles (Hameed, 2007).

Influence of pH

Figure 8 shows a variation in the adsorption rate at pH values varying between 2 and 12. This variation is related to the chemical composition of the adsorbates. We note that the TCP adsorption rate is significantly higher for low pHs and particularly for pH = 4 (99.51%) where the pH is below the TCP pKa (TCP pKa is 6.21). This result is comparable to those obtained by other authors (Hameed, 2007; Zaghouane-Boudiaf et al., 2014). At acid pH, dispersion interactions predominate, whereas at basic pH the high concentration of chlorophenol in the ionic state favors electrostatic repulsion between the species present, thereby decreasing the rate of adsorption of TCP.

Influence of temperature

As seen from Figure 9, the fixation of TCP by the modified bentonite decreases with increasing temperature which indicates that the adsorption of the TCP on Crude-B-BDTA is an exothermic process. Similar results have been found in the case of adsorption of chlorophenols on organophilic clays (Al-Asheh et al., 2003; Su et al., 2011; Zaghouane-Boudiaf and Boutahala, 2011; Zhou et al., 2011). As temperature increases, the solubility of TCP increases and its adsorption



Fig. 4: IR spectrum of BDTAC



Fig. 5: Mass influence of crude-B-BDTA on TCP adsorption; Volume of solution = 50 mL; Concentration = 100 mg/L; Contact time = 120 min; Stirring speed = 180 rpm; PH = 4; Temperature = 19°C



Fig. 6: Influence of TCP concentration on crude-B-BDTA; Volume of solution = 50 mL; Mass of adsorbent = 70 mg; Time = 120 min; Stirring speed = 180 rpm; PH = 4; Temperature= 19° C.

decreases this is due to a weakening of the attractive forces between TCP and adsorbent sites (Hameed, 2007).

Kinetic studies

The effect of contact time on the adsorption rate of TCP on modified bentonite sample is shown in Figure 10. The shapes of the curve show different steps on the kinetic uptake. The Figure showed rapid adsorption of TCP within the first 20 min indicating high affinity between the TCP molecules and the surface of modified bentonite and reached equilibrium after 60 min. The rapid adsorption at the initial contact time can be attributed to the abundant availability of active sites on the adsorbent surfaces. Afterwards with the gradual occupancy of these sites, the adsorption decreased.

To better understand and analyze these results, the constants of sorption and intraparticle diffusion were determined in terms of the pseudo-first-order, pseudo-second-order, and intraparticle diffusion models. The conformity between experimental data and the model-predicted values was expressed by the correlation coefficient R_2 .

Pseudo-first-order model

The rate constant of adsorption can be determined from the following pseudo-first-order model given by Lagergren, as shown in Eq. 2 (Lagergren, 1898).

$$\frac{\mathrm{d}q_{\mathrm{t}}}{\mathrm{d}\mathrm{t}} = \mathrm{K}_{1}(\mathrm{q}_{\mathrm{e}} - \mathrm{q}_{\mathrm{t}}) \tag{2}$$

 K_1 : is the pseudo-first-order rate constant (min⁻¹); q_e : and q_t are the amounts of TCP adsorbed at equilibrium and at time t in mg/g, respectively, t : contact time (min).

The integration of Eq. (2) with the initial condition, $q_t = 0$ at t = 0 leads to, as shown in Eq. 3,

$$\ln(q_e - q_t) = \ln q_e - K_1 t \tag{3}$$

If the pseudo-first-order kinetics is applicable, a plot of ln ($q_e - q_t$) versus t should provide a straight line from which k_1 and predicted qe can be determined from the slope and intercept of the plot, respectively.

Pseudo-second-order model

A pseudo-second order rate expression based on the sorption capacity is proportional to the number of active sites occupied on the sorbent and its equation can be written, as shown in Eq. 4 (Ho and McKay, 1998),

$$\frac{\mathrm{d}q_{\mathrm{t}}}{\mathrm{d}\mathrm{t}} = \mathrm{K}_{2}(\mathrm{q}_{\mathrm{e}} - \mathrm{q}_{\mathrm{t}})^{2} \tag{4}$$

Where, k_2 is the rate constant for pseudo-second order adsorption (g/mg·min), q_e is the amount of solute adsorbed at equilibrium (mg/g) and q_t is the amount of solute adsorbed at any time t (mg/g). By integrating Eq. (4) for the boundary conditions t = 0 to t = t and q_t = 0 to q_t = q_t its equation can be written, as shown in Eq. 5.

$$\frac{t}{q_{t}} = \frac{1}{K_{2}.q_{e}^{2}} + \frac{1}{q_{e}}t$$
(5)

By plotting t/q_t versus t, qe and k_2 can be determined from slope and intercept. The linear plot of $ln(q_e - q_t)$ versus t (Fig. 11) and t/q_t versus t (Fig. 12) give the values of the rate constant of the pseudo-first-order (k_1), the pseudo-second-

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Table 1: Kinetic models parameters obtained in adsorption of 2,4,6-TCP onto crude-B-BDTA							
Pseudo-first-ordre model			Pseudo-second-ord				
K1(1/min)	q _e (mg/g)	R ²	K ₂ (g/mg min)	q _e (mg/g)	R ²		
0.035	4 070	0.93	0.060	24 509	0 99		
01000	1107.0	0198	01000	211009	0199		

Table 2: Intraparticle diffusion model: Constants for adsorption of TCP onto crude-B-BDTA Intraparticle diffusion

Etape 1			Etape 2			Etape 3		
K i1 (mg/g.min ^{1/2})	C ₁	R ²	K _{i2} (mg/g.min ^{1/2})	C ₂	R ²	K _{i3} (mg/g.min ^{1/2})	C ₃	R ²
0.035	33.033	0.99	0.699	30.059	0.98	0	35.523	-

Table 3: Langmuir and Freundlich isotherm constant and values of parameters for adsorption of TCP on the organoclay

Freundlich			Langmuir		
K _F	1/n	R ²	q _m (mg/g)	K _L (L/mg)	R ²
 3.24	0.61	0.96	35	0.006	0.99

order (k_2) respectively and correlation coefficient R_2 . The results reported in Table 1 shows that correlation coefficient value for the pseudo-second order kinetic model was about 0.99, indicating the applicability of the model to describe the adsorption process which is similar to the result reported by (Hameed, 2007; Khenifi et al., 2009; Koyuncu, 2008; Zaghouane-Boudiaf et al., 2014).

Intraparticle diffusion model

The model of intraparticle diffusion was suggested by Weber and Morris (Weber and Morris, 1962). This model is frequently used to describe the rate determining step of the sorption process occurring on porous adsorbents which is theoretically calculated using the equation, as shown in Eq. 6,

$$q_t = K_i \cdot t^{1/2} + C$$
 (6)

Where, K_i is the rate constant for intraparticle diffusion $(mg/g \cdot min^{1/2})$ and obtained from the slope of the straight line of q_t versus $t^{1/2}$, C is a constant.

The adsorbate species are most probably transported from the bulk of the solution into the solid phase through intraparticle diffusion transport process, which is often the rate-limiting step in many adsorption processes (McKay, 1983). According to intraparticle diffusion model, several mechanisms are involved and the adsorption process can be characterized into three steps: external surface adsorption, intraparticle diffusion which is the rate-limiting step and the final equilibrium which is very fast. From the plots q_t versus $t^{1/2}$ (Fig. 13) the regression was linear, but the plot did not pass through the origin, suggesting that adsorption involved intraparticle diffusion, but that was not the only rate-controlling step. Other kinetic models may control the adsorption rate.

Adsorption isotherms

To understand the adsorption isotherm for the mechanism of adsorption system several mathematical models can be used. In this study the equilibrium data were modeled with the Langmuir and Freundlich models to investigate the sorption mechanisms of TCP compound.

Freundlich isotherm

Freundlich suggested an empirical expression to describe the adsorption theory (Freundlich, 1909). The model is based on the assumption that the adsorbent surface is heterogeneous and consists of different classes of adsorption sites. The adsorption takes place at the heterogeneous surfaces.

The most commonly used form is the logarithmic plot of variations of q_e as a function of C_e , as shown in Eq. 7.

$$\log q_e = \log K_F + 1/n \log C_e \tag{7}$$

n is Freundlich constant related to the adsorption intensity of the adsorbent, and Ce is the concentration of solute in the solution at equilibrium (mg/L). K_F is Freundlich constant related to the adsorption capacity. C_e is often expressed in mg/L and q_e in mg/g, the unit of K_F is mg ⁽¹⁻ⁿ⁾.lⁿ/g.

$$q_e = \frac{(C_i - C_e)}{m} . V \tag{8}$$

C_i: initial concentration (mg/L) C_e: concentration at equilibrium (mg/L) V: volume of the solution (L) m: mass of the adsorbent (g)

Langmuir isotherm

The second most commonly used model is Langmuir (Langmuir, 1918), it described the relation between the adsorption of adsorbate and the surface of the adsorbent. The adsorbate is strongly attracted to the surface and it is assumed that a monolayer adsorption is involved. It is then assumed that once an adsorbate molecule occupies a site, no further adsorption can take place at that site. Therefore, a saturation value is reached beyond where no further adsorption can take place. The Langmuir model can be expressed as shown in Eq. 9,

$$\frac{1}{q_{e}} = \frac{1}{C_{e}} \cdot \frac{1}{q_{m}K_{L}} + \frac{1}{q_{m}}$$
(9)

 K_L is the Langmuir equilibrium constant, q_e and q_m are expressed in mg/g and C_e in mg/L and the constant K_L are expressed in L/mg. The adsorption isotherms are evaluated by the graphic plots of the relation $1/q_e = f (1/C_e)$. The adsorption equilibrium data for phenol and chlorophenols onto GAC were analyzed by non-linear curve fitting analysis.

Figure 14 shows Langmuir, Freundlich adsorption isotherms by linear analysis; Table 3 summarizes the corresponding isotherm parameters. High R^2 are derived by fitting experimental data into the Langmuir isotherm model ($R^2 = 0.99$) and the Freundlich isotherm model ($R^2 = 0.96$). These suggest that both Langmuir isotherm model and the Freundlich isotherm model can generate a satisfactory fit to the experimental data.

The adsorption equilibrium data for phenol and chlorophenols onto GAC were analyzed by non-linear curve fitting analysis

Desorption study

In this section, we analyzed the process of adsorption and desorption of TCP on modified clay under the following conditions: 70 mg of organoclay was added into 50 mL of TCP solution of 50 mg/L and stirred for 60 min at room temperature. After centrifugation, TCP adsorbed rate is 99.52%. The same solution was then stirred for 60 min at 250 rpm. The level of TCP released in solution is 7.24%,



Fig. 7: Influence of contact time on TCP adsorption by crude-B-BDTA. Volume of solution = 50 mL; Mass of adsorbent = 70 mg; Concentration = 50 mg/L; Stirring speed = 180 rpm; PH = 4; Temperature = 19°C



Fig. 8: Influence of pH on the adsorption of TCP by crude-B-BDTA; Volume of solution = 50 mL; Mass of adsorbent = 70mg; Concentration = 50 mg/L; Stirring speed = 180 rpm; Contact time = 60 min; Temperature = 19 °C



Fig. 9: Influence of temperature on TCP adsorption by crude-B-BDTA. Volume of solution = 50 mL; Mass of adsorbent = 70 mg; Concentration = 50 mg / L; Stirring speed = 180 rpm; Contact time = 60 min; pH = 4



Fig. 10: Kinetic adsorption data for TCP on crude-B-BDTA. Volume of solution = 50 mL; Mass of adsorbent = 70 mg; Concentration = 50 mg/L; Stirring speed = 180 rpm; pH = 4



Fig. 11: Pseudo-first-order plots of TCP adsorption onto crude-B-BDTA



Fig. 12: Pseudo-second order plots of TCP adsorption onto crude-B-BDTA



Fig. 13: Plot of intraparticle diffusion model for the adsorption of TCP on crude-B-BDTA



Fig. 14: Freundlich (above) and Langmuir (below) isotherm models

Whereas 92.28% has been trapped, which confirms the effectiveness of this material.

CONCLUSIONS

The preparation of a new material was carried out with the bentonite of Maghnia modified with the surfactant BDTAC at 2xCEC for the removal of the organic pollutant (TCP) from diffraction, powder infrared water. X-ray and thermogravimetric analysis showed that BDTAC was successfully intercalated in the interlayer space of the bentonite. The DRX characterization results showed the expansion in the basal spacing of the bentonite due to the intercalation of benzyldimethyltetradecylammonium from 14.79Å to 28.23Å. The appearance of new bands on the IR spectrum of the organophilic bentonite confirms the surfactant insertion into the crude bentonite. The adsorption of TCP on BDTAC-modified bentonite showed good removal of the pollutant (99.52%) at a concentration of 50 mg/L, pH 4 and contact time of 60 min. The adsorption process follows the pseudo-second order kinetic model and intra-particle diffusion. The isothermal data for adsorption followed the Freundlich and Langmuir models. The desorption reaction showed that 7.24% of TCP is released, thus confirming the effectiveness of this low cost material as adsorbents for environmental pollutant.

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