e-ISSN:2347-7857
p-ISSN:2347-7849

Research & Reviews: Journal of Pharmaceutics and Nanotechnology

ADSORPTION OF TARTARIC ACID ONTO BENTONITE. A KINETIC STUDY

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Research Article

Received date: 13/11/2015
Accepted date: 07/12/2015
Published date: 14/12/2015

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Keywords: Adsorption, Bentonite, Clay, Langmuir, Freundlich isotherms.

ABSTRACT

The kinetics of adsorption of Tartaric acid onto bentonite was investigated at 33 ± 2°C using different weight of the clay (0.005, 0.01 and, and 0.1 g). Each weight represents a certain sample size. The order of the process appeared to be 1 with respect to Tartaric acid and 2 with respect to the clay surface area. The rate was found to depend on internal diffusion which produced a decrease in the specific rate of sorption as a function of time. We used spectrophotometer model -2300UV for analytical analysis. This interest stems from the fact that for each specific organic acid molecule, the rate constant of the adsorption is directly related to the nature of the substrate, namely the surface properties of the clay. The batch isotherm studies for the adsorption of tartaric acid onto bentonite was carried out. The initial concentration of Tartaric acid was varied 0.0063 eg.g/l to 0.0356 eg.g/l. From the experimental data, the isotherm parameters of Freundlich and Langmuir were calculated for the sample and compared. There exists a correlation between the physicochemical properties of Bentonite and the adsorption process.

INTRODUCTION

With the rapid increase in global industrial activities, Organic compound pollution has become serious. Among various treatment technologies, montmorillonite is commonly used, due to its relatively high surface area, cation exchange capacity, harmlessness to the environment, and ease of operation [1-3]. The adsorption properties of this clay mineral have intrigued soil scientists for over a century. Most investigators [4-6] have been interested in the total exchange capacity and equilibrium exchange relationships, but few [7-10] have studied the rate of anion exchange on clay mineral.

The management of these involves the establishment of specific methods. Among these we find the chemical precipitation [11], electrodeposition [12], the liquid-liquid extraction [13], coagulation-flocculation [14], reverse osmosis [15], and adsorption on activated carbon [16].

Nevertheless, the presence of organic substances such as carboxylic acids can disrupt the operation of biological treatment processes. It is therefore important to remove the prior and optionally if the value obtained with a high degree of purity [17].

The adsorption has become a well-established separation technique for removing pollutants. The removal of organic dyes by adsorption on agricultural residues has recently become a topic of considerable interest [18].

The adsorption properties of bentonite have intrigued scientists for more than a century. More investigators [1-3] have studied the ability of total trade and exchange relations of balance, but some [4-7] studied the exchange rate of cations on clay minerals. One reason for this was the overall conclusion [8] that the cation exchange clays occur quite rapidly, within 1-3 min. The reaction kinetics may suggest the mechanistic details of the adsorption process; they are also human rights importance for a complete understanding of interfacial phenomena at the solid-liquid interface. The main objective of this research is to determine the adsorption process of tartaric acid. This allows us to predict and control the behavior of tartaric acid on the surface of the clay. For this purpose, besides the adsorption equilibrium experiments that were made, the kinetic behavior also appears to be of great interest. This interest stems from the fact that for each specific organic acid molecule, the rate constant of the adsorption is directly related to the nature of the substrate, namely the surface properties of the clay.

This study was conducted to determine an appropriate expression that describes the adsorption of tartaric acid on the clay surface to define a predictive model of the kinetics of adsorption of organic acids by the bentonite.

MATERIALS AND METHODS

Adsorbent: The sample of bentonite used in the context of our work comes from the French laboratory name PROLABO 12 located on the street cracked 75011 Paris.

The specific surface area is 65 m^2/g. The clay used has the following chemical composition: 54.55% SiO_2. 20.12% Al_2O_3. 2.23% Fe_2O_3. 0.21% TiO_2. 1.48% FeO. 0.95% CaO. 1.99% MgO. 0.01% MnO. 2.48% Na_2O. 0.31% K_2O. 16.04% H_2O.

Tartaric acid: The tartaric acid crystals white (99%) is supplied by Merck. Acid 2,3-dihydroxybutanedioïque or d-alpha acid, beta-dihydroxysuccinic. From a crystalline appearance and a white color with fusion point ranging from 168-170°C. It is odorless and its relative density is 1.6 g/cm^3.

Analytical method: Sample analysis was performed before and after adsorption by UV-visible spectrophotometry (Spectrophotometer model -2300UV) to the absorption wavelength of Tartaric acid (490 nm). We used the vanadic reagent for the colorimetric assay.

It is necessary to perform a calibration line with a series of standard solutions obtained by diluting the stock solution of Tartaric acid.

Process Kinetic

Several models were tested: pseudo-first order, pseudo- second order [4] and intraparticle diffusion model [5]. The model of the pseudo first order is based on the adsorption capacity of the solid phase; it is generally applicable for the first 30 minutes of the adsorption phenomenon. For the pseudo-first order relationship Largergren [6] based on the amount adsorbed, the first equation is established speed to describe the kinetics of adsorption in a liquid-solid system **(Table 1 and 2).** The application of the model Blanchard [8] allows us to define the pseudo second-order reaction in a sorption process.

Table 1. Kinetic parameters Largergen model (mads=0,1g).

Mass of Bentonite = 0.1g		
Co(eg.g/L)	**Kads(s^{-1})**	**R^2**
0.021	0.0314	0.0884
0.0292	0.0244	0.9142
0.0356	0.0092	0.9874

Table 2. Kinetic parameters Largegen model (mads=0.01g).

Mass of Bentonite = 0.01g		
Co(eq.g /L)	**Kads(s^{-1})**	**R^2**
0.021	0.0029	0.9719
0.0292	0.0016	0.9200
0.0356	0.0016	0.9414

We introduce a mass of bentonite ranging from 0.01 g to 0.1 g from sample previously placed in an oven at a temperature of 110°C for 24 hours in a flask. We added 10 ml of tartaric acid solution of a concentration ranging from 0.0210 eq.g/l to 0.0356 eq.g/l along with stirring at a speed of 500 rpm and varying the contact time between tartaric acid and bentonite. After a time seems to be of great interest. This interest stems from the fact that for each specific organic acid molecule, the rate constant of the adsorption is directly related to the nature of the substrate, namely the surface properties of the clay.

This research was conducted to determine an appropriate expression that describes the adsorption of tartaric acid on the surface of the clay to define a predictive model of the kinetics of adsorption of organic acids by the bentonite [8].

RESULTS AND DISCUSSION

The order of the reaction was carried out on three different solutions concentrations 0.0210, 0.0292 and 0.0356 eq.g/l.

Pseudo first order

The relationship Largergren [6] based on the amount adsorbed, the first equation is established speed to describe the kinetics of sorption in a liquid-solid system.

The pseudo-first order model is represented by the following relationship:

$$\frac{dq}{dt} = K_{ads} \times (\mathrm{qe} - \mathrm{qt}) \quad (1)$$

The integration of equation (1) for the boundary conditions:

$$qt \ = \ 0 \ à \ t \ = \ 0 \ et \ qt \ = \ qe \ à \ t \ = t_{équilibrs}$$

$$\mathrm{Ln}\frac{qe - qt}{qe} = -K_{ads} \quad (2)$$

qe: Quantity adsorbed at equilibrium (mg/g), qt : amount adsorbed at time t (mg/g), t: time of the adsorption process for our case, it is 0-150 seconds; K_{ads}: sorption rate constant of pseudo-first order (s^{-1}) linearization of the equation gives:

$$Ln\left(qe - qt\right) \ = \ -K_{ads} \times \ t \ + \ Lnqe \quad (3)$$

By applying the equation on our experimental data, we get straight **(Figures 1-3)** slope (K_{ads}) and intercept (lnqe) [7].

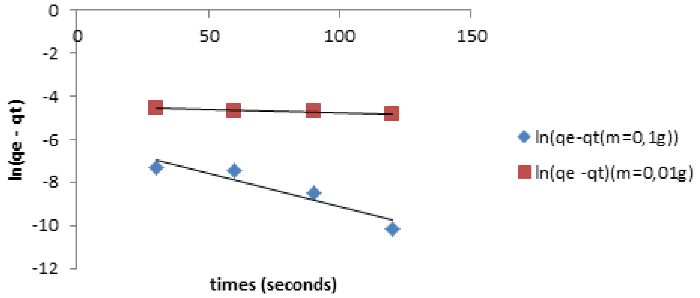

Figure 1. Kinetic sorption of tartaric acid-Largergen model (Co = 0.0210 eg.g/L).

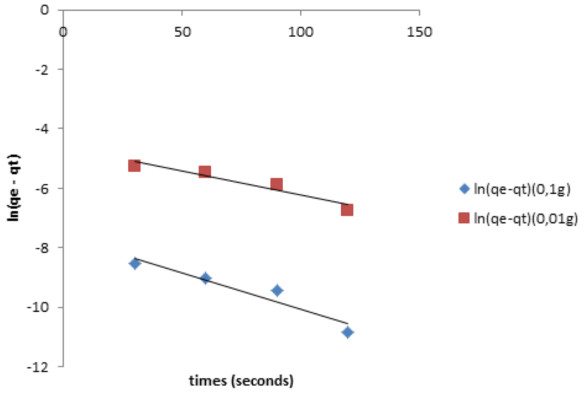

Figure 2. Kinetic sorption of tartaric acid-Largergen model (Co = 0.0292 eg.g/L).

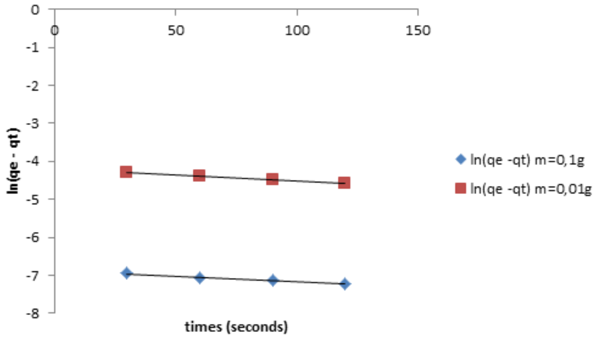

Figure 3. Kinetic sorption of tartaric acid-Largergen model (Co = 0.0356 eg.g/L).

Pseudo second order

The application of the model Blanchard [8] allows us to define the pseudo second-order reaction in a sorption process **(Table 3 and 4).**

Table3. Kinetic parameters of pseudo second order (Weight of Bentonite =0.1g).

Weight of Bentonite = 0.1g		
Co(eq.g/L)	**Kads(s^{-1})**	**R^2**
0.021	9.9823	0.9589
0.0292	82.8708	0.9998
0.0356	26.5811	0.9910

Table 4. Kinetic parameters of pseudo second order (Weight of Bentonite =0.01g).

Weight of Bentonite = 0.01g		
Co(mol/L)	**Kads(s^{-1})**	**R^2**
0.021	0.7967	0.8592
0.0292	0.4253	0.8986
0.0356	0.3523	0.9120

$$\frac{dq}{dt} = K_{ads} \times (qe - qt)^2 \qquad (4)$$

qe: Quantity adsorbed at equilibrium (eq.g/g), qt: amount adsorbed at time t (eq.g /g), t: time of the adsorption process for our case, it is 0-150 seconds; K_{ads}: rate constant of pseudo-first order adsorption (eq.$g^{-1}s^{-1}$).

By integrating the equation (8), and using the boundary conditions, we obtain the following equation

$$\frac{t}{qt} = \frac{1}{qe} \times t + 1/(\frac{1}{K_{ads} \times q_e^2}) \qquad (5)$$

Wearing $\frac{t}{qt}$ versus time t, we get the straight lines shown in **Figures 4-7** with slopes $\frac{1}{qe}$ and intercepts $\frac{1}{K_{ads} \times q_e^2}$

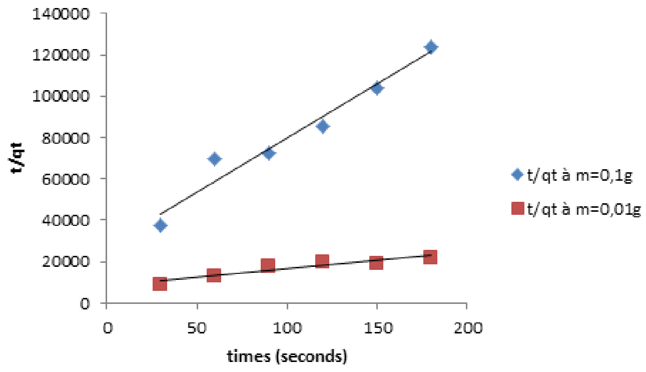

Figure 4. Kinetic sorption of tartaric acid-Blanchard model (Co = 0.021eq.g/L).

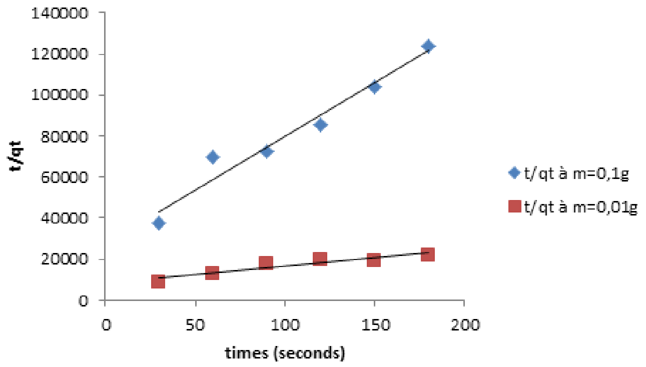

Figure 5. Kinetic sorption of tartaric acid-Blanchard model (Co = 0,0292 eq.gl/L).

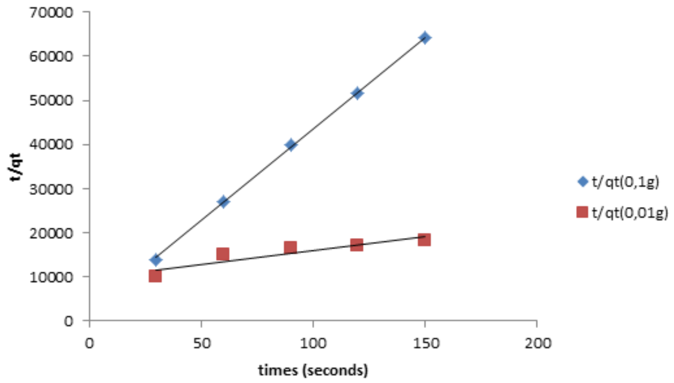

Figure 6. Kinetic sorption of tartaric acid-Blanchard model (Co = 0, 0356 eq.g/L).

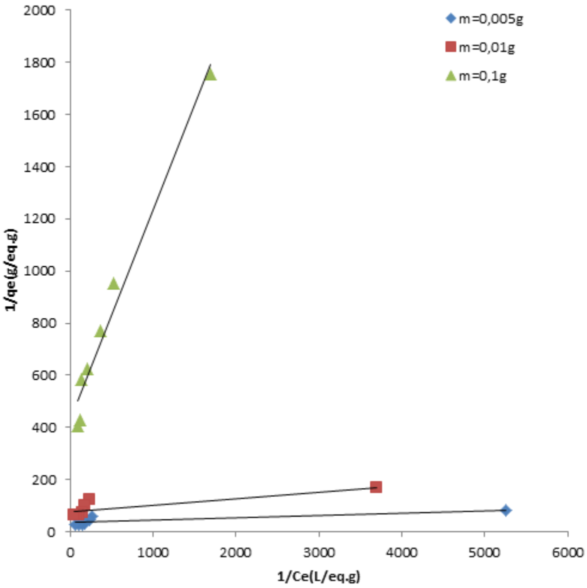

Figure 7. Langmuir model for sorption of tartaric acid for various masses of Bentonite.

Kinetic models that have been proposed to allow better study the mechanisms likely adsorption of tartaric acid on bentonite. The mechanism of adsorption depends on the physical or chemical characteristics of the adsorbent such that the mass transfer process. The adsorption of the tartaric acid mechanism was studied by using the proposed model kinetic Largregen. We calculated the rate constant k at room temperature (33 ± 2°C), the equation of Largregen has been exploited for calculations. K values for each concentration and mass of the adsorbent are calculated and it allowed us to calculate qe and the correlation coefficient R^2. The experimental data are in agreement with the calculated values representing the applicability of the first order model. It is therefore clear that for high concentrations and low mass of adsorbent model first order is more appropriate. However, the 2nd order model is also operated and showing the relationship between $\frac{t}{qt} = f(t).\, qe$ values obtained are in agreement with experimental data. The correlation coefficients are large in the order of 0.9998.This result reflects the fact that the adsorption of the tartaric acid onto bentonite respects the order kinetics more than two to one order of high masses.

The curves expressing the binding kinetics of tartaric acid onto bentonite shows that after 150 sec, equilibrium is reached, the study of the kinetics of adsorption has also shown that the attachment mechanism of the especially tartaric acid could vary depending on the mass of the adsorbent so that the concentration of the adsorbate. In particular, the phenomena of complexation and ion exchange surface may extend competitively in the dissemination phase of the organic compound between the layers of clay [19, 20].

STUDY OF ADSORPTION

We introduce a mass of bentonite ranging from 0.005 g to 0.1 g of bentonite previously placed in an oven at a temperature of 110°C for 24 h. We add 10 ml of tartaric acid solution of a concentration ranging from 0.0063 eg.g/l to 0.0356 eg.g/l. We stir with a speed of rotation of 500 rpm by varying the contact time between tartaric acid and bentonite. After a stirring time given, we filter and measure the absorbance by spectrophotometer (model -2300UV).

Co is the initial concentration of tartaric acid, the quantity qe of equilibrium adsorbed per unit mass of adsorbent acid is given by the relation acid:

$$qe = (Co - Ce)\frac{v}{m_{adsorbant}} \tag{6}$$

Where V is the volume of the solution of tartaric acid introduced into the flask.

m is the mass of adsorbent used in a test.

It is adsorbed to the equilibrium concentration.

Application of the Freundlich model: determination of the constants 1/n and k_f

The Freundlich model equation is presented as follows [2]:

$$qe = \frac{x}{m} = K_F \times C_e^{\frac{1}{n}} \tag{7}$$

This and where qe have the same definitions as above, $\frac{1}{n}$ and K_F Freundlich constants, indicative of the intensity and the adsorption capacity.

The linearized Freundlich relationship is as follows:

$$Lnqe = \frac{1}{n} LnCe + LnK_F \tag{8}$$

The route curves Lnqe = f (LnCe) gives us straight lines shown in **Figures 8.** These lines have to $\frac{1}{n}$ slope intercept LnK$_F$. So we get constant $\frac{1}{n}$ and K$_F$ whose values are reported in **Table 5.**

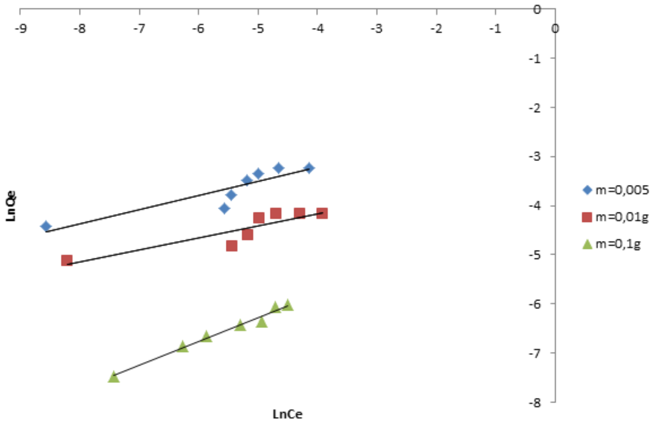

Figure 8. Freundlich model for the sorption of tartaric acid for various masses.

Table 5. Langmuir Parameters.

	Mass of the bentonite (g)		
Parameters	**0.1**	**0.01**	**0.005**
qmax(eq.g./g)	0.00233	0.01282	0.02959
K$_L$(1/g)	531.5331	3182.5714	3519.3819
R^L	0.058	0.014	0.012
R^2	0.9737	0.7005	0.7284

Application of the Langmuir model: determination of the constants k$_L$ and q$_{max}$

The equation of the Langmuir adsorption model is given as follows [3]:

$$qe = \frac{K_L \times C_e \times q_{\max}}{1 + K_L \times C_e} \tag{9}$$

The linearized equation Langmuir equation:

$$\frac{1}{qe} = \frac{1}{K_L \times q_{\max}} \times \frac{1}{C_e} + \frac{1}{q_{\max}} \tag{10}$$

The route curves $\frac{1}{qe} = \frac{1}{Ce}$ gives us straight lines shown in **Figures 7.** These lines have the slope $\frac{1}{K_L \times q_{max}}$ and For ordered $\frac{1}{q_{max}}$. So we get constant $\frac{1}{n}$ and KF whose values are reported in **Table 6.**

Table 6. Freundlich Parameters.

	Mass of the bentonite (g)		
Parameters	**0.1**	**0.01**	**0.005**
1/n	0.453	0.228	0.280
K$_F$ (eq.g/g)	0.0173	0.042	0.14
R^2	0.9825	0.7999	0.8016

Figure 8 shows the sorption isotherm of tartaric acid using the bentonite to the Langmuir model calculates constants Langmuir absorbed tartaric acid, that is to say, complete absorption of the adsorbate by the available sites in intensity and energy absorption are obtained by the y-intercepts and the calculation of the slope explaining isotherm equation. The lines obtained allow us to state that the absorption of tartaric acid on bentonite is in agreement with the Langmuir model (R^2 = 0,9737). Qmax vary from 0.012824 to 0.029598 for adsorbent masses ranging from 0.005 to 0.1g.

The essential feature of the isotherm is expressed by a constant called separation factor or (R$_L$) balance parameter is defined by the equation:

$$R_L = \frac{1}{1 + K_L \times C_m} \tag{11}$$

The R_L value indicates what type of Langmuir isotherm she obeyed [2]:

$R_L = 0$ irreversible Langmuir isotherm,

$0 < R_L < 1$ favorable,

$R_L = 1$ linear,

$R_L > 1$ unfavorable.

Table 5 shows that for the different masses of adsorbents used to vary R_L 0012-0058 show that although adsorption onto bentonite tartaric obeys the Langmuir isotherm acid.

Application of the Langmuir model : determination of the constants kL and qmax

This model assumes that the adsorption is carried out in a homogeneous manner at the adsorptive surface and that the adsorption sites all have the same affinity for the adsorbate molecules, that is to say of the same energy adsorption. The results obtained with this model are shown in **Figure 7.** Langmuir constants are reported in **Table 5.**

Adsorption tartaric acid on bentonite according to the linear model is shown by Freundlich **(Figure 8).** The relationship between the adsorbed amount at equilibrium of tartaric acid and the concentration of this acid at equilibrium is given by the equation denoted (7). For different masses used, the values of $\frac{1}{n}$ range from 0228-0453 and R^2 range from 0.7999 to 0.9825. The values of n are greater than 1, this clearly indicates that the Freundlich adsorption supports, and also shows that the surface of the bentonite is of a heterogeneous nature. This is more interesting when the clay mass is large this can be explained by the availability of active sites during adsorption process [21, 22].

Influence of the contact time on the percentage adsorption

The reaction yields sorption of tartaric acid onto bentonite based on time are shown in **Figures 7-9.** In view of the results, we find that the percentage adsorbed increases with stirring time to reach an equilibrium time (150 sec) in which this percentage is changing almost. This can be explained by the availability of active sites are available that before and after the adsorption there saturation of these sites, allowing to reach the adsorption equilibrium. Based on **Figures** R = f (t) , we find that this adsorption is fast at the beginning and after that becomes slow this can be explained by Arrinnghieri [23] that the whole adsorption process can be divided into two steps:

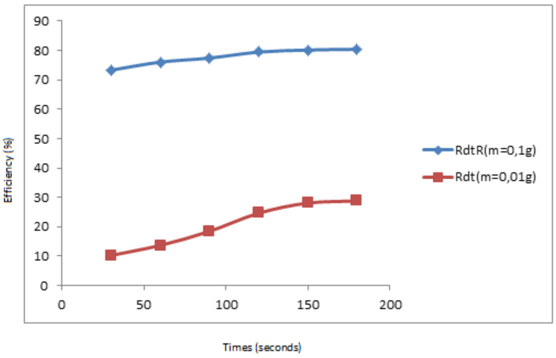

Figure 9. Effect of contact time on the adsorption efficiency of tartaric acid (Co = 0.021eq.g / L).

- Rapid initial adsorption followed by a slow adsorption. This step could be fast Arrinnghieri rating according to the rapid diffusion of ions from the solution to the outer surface of the bentonite.
- The step of slow diffusion of ions in the pores of the adsorbent. In the case of our study, the operating conditions allow us to assert this claim in view of the fact of maintaining the adsorption constant speed.

Influence of the amount of adsorbent

According to the results, we find that the percentage adsorbed varies in an increasing manner with the amount of adsorbent which can be explained by the fact that over the adsorbent surface is; the greater the availability of adsorption is favorable. It is also apparent in view of the results obtained that the order of reaction rate varies with the amount of adsorbent. We find adsorbent for masses low reaction tends more towards order 1, order 2 and otherwise.

We can generally say that the 2nd order equations are used to describe the chemisorption of gases, ionic exchanges and observed on the surfaces. In our case, the tartaric acid is a polyfunctional molecule containing the carboxyl hydroxyl groups, which can interact in the chemical bonds may be established between the surface of the bentonite, and ion exchange of the charge on the surface.

The advantage of this method lies in the fact that it is not always necessary to know the adsorbed at equilibrium capacity.

DETERMINATION OF LIMITING STEPS

External transport

When the adsorption process is controlled by the external carriage (resistance due to the boundary layer), the plot of the logarithm of the residual concentration versus time should be linear [24]. The correlation coefficients **(Tables 7 and 8)** obtained are our references to illustrate this mechanism.

Table 7. Internal transport and external transport parameters for a 0.01g bentonite mass.

	Paramètres de Weber et al.		Paramètres de transport externe	
Co (eq.g/L)	Ordonnée à l'origine	R^2	K_w(eq.g/g.s)	R^2
0.021	0.000	0.934	0.001	0.854
0.0292	0.001	0.975	0.001	0.962
0.0356	0.001	0.980	0.001	0.971

Table 8. Parameters of the internal transport and external transport for 0.1 g of bentonite.

	Paramètres de Weber et al.		Paramètres de transport externe	
Co (eq.g/L)	Ordonnée à l'origine	R^2	K_w(eq.g/g.s)	R^2
0.021	0.000	0.903	0.005	0.892
0.0292	0.002	0.969	0.002	0.924
0.0356	0.001	0.946	0.001	0.962

The drawing of straight, the logarithm of the residual concentration versus time InCt = f (t) actually gives us straight **(Figure 10 and 11).** We find that:

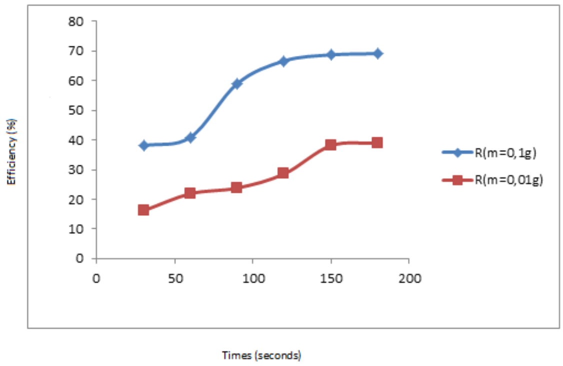

Figure 10. Effect of contact time on the adsorption efficiency of tartaric acid (Co = 0.0292 eq.g / L).

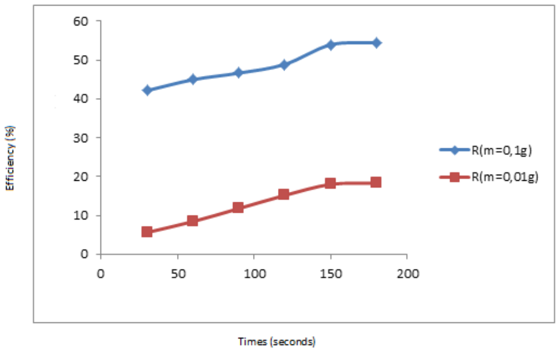

Figure 11. Effect of contact time on the adsorption efficiency of the tartaric acid (Co = 0.0356 eq.g / L).

- For The mass equal to 0.01 g bentonite, only the coefficients of two of the three concentrations are above 0.95. The correlation coefficient is 0.854 for the concentration of 0.021eq.g/l. We can say in agreement with Lomauro in the case of model validation that this value is acceptable. We can therefore conclude that for this mass, external transport is a step that seems to control the speed of the overall sorption process of tartaric acid in aqueous solution with bentonite [25].
- For 0.1 g of bentonite, we have only one correlation coefficient is greater than 0.95. We believe that operation errors are the cause of this value. We could have had as in the previous two favorable cases out of three. Once again we can say that the external transport is a step that seems to control the speed of the overall process tartaric acid sorption in aqueous solution by bentonite.

In general, it appears that on the external transport appears to be a rate controlling step in the overall sorption process of tartaric acid in aqueous solution by bentonite. The intra-particle diffusion model is used to determine the limiting phenomenon the adsorption mechanism. The expression of this model is:

$$qt = K_i \times \mathbf{t}^{0.5} + C \tag{12}$$

K_i is the intra-particle diffusion constant (eq.g g^{-1} s^{-1}) and C is a constant (g. g^{-1}). The model obtained may submit a multilinearity corresponding to existence of several successive steps in the adsorption. The first step, very short, is a limitation of the adsorption by the external diffusion. The second step corresponds to the progressive adsorption of solute, the phenomenon being then limited by intra-particle diffusion. The third step corresponds to obtaining a state of equilibrium: the solute concentration is very low, thus slowing the diffusion phenomenon [25].

Weber et al. [26] reported that if the intra-particle diffusion is involved in the sorption process, carrying the sorbed amount depending on the square root of time, we have to get right. This step is limiting if the line passes through the origin.

Where these lines do not pass through the origin, it indicates that the pore diffusion is not the only mechanism limiting the sorption kinetics. It appears that other mechanisms are involved [27, 28].

According to **Figures 12 and 13**, we see that two straight lines drawn on three of the issues of the function qt= f ($t^{1/2}$) do not pass through the origin, which shows that the distribution of tartaric acid in pores is not the only mechanism limiting the sorption kinetics. Surely there are other mechanisms involved.

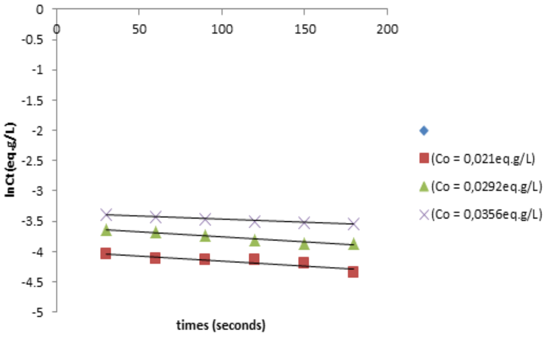

Figure 12. LnCt variation as a function of time t (external transport) to a mass of 0.01 g of adsorbent.

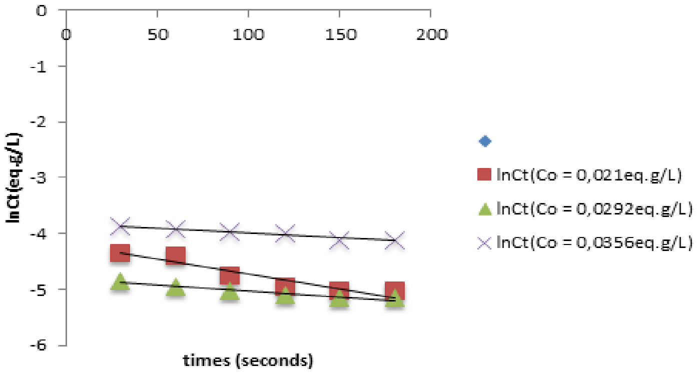

Figure 13. LnCt variation in function of time t (external transport) to a mass of 0.1 g of adsorbent.

CONCLUSION

In the present study, bentonite clay was selected as a local, cheap and readily available adsorbent for the removal of Tartaric acid from the aqueous solutions. Adsorption and Kinetic study was studied by batch adsorption experiments.

Natural bentonite used is of montmorillonite nature. The results revealed that the study of the kinetics of adsorption has also shown that the attachment mechanism of the especially tartaric acid could vary depending on the mass of the adsorbent so that the concentration of the adsorbate.

The adsorption kinetic studies showed that the removal of Tataric acid is a rapid process and the adsorption process obeys the pseudo-second order model, indicating cationic dye has a very strong affinity on the bentonite surface. It was found that the experimental isotherm data can be fitted well to the Langmuir equilibrium isotherm model.

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