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RESEARCH ARTICLE

DYE REMOVAL FROM AQUEOUS SOLUTION BY BASE ACTIVATED CORN COBS POWDER FROM ADAMAWA REGION OF CAMEROON

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ABSTRACT

The study concerns the adsorption of methylene blue which is an organic dye by the corn cobs activated at the base powder (BACCP). The analysis of the time parameter allowed us to determine the adsorption equilibrium time which is 15min. The maximum adsorption capacity obtained is 11.904 mg / g at a normal pH of 6.5 and at the temperature of 25 °C. Experimental data are analyzed by the Freundlich and Temkin isotherms. The kinetic model of pseudo second order and Elovich were applied with the correlation coefficients of R² is more than 0.99. The reactions are made according to the models of pseudo second order and Elovich suggesting the hypothesis that the mechanism of the reactions are done in three phases:

- The diffusion of the MB towards the surface of the adsorbent;
- The diffusion of the MB into pores ;
- The interaction between the adsorbate molecules (MB) and the surface of the adsorbent.

And that adsorption sites increase exponentially with adsorption, which implies multilayer adsorption. Thermodynamic parameters have shown that MB adsorption on corn cobs is spontaneous ($\Delta G < 0$), endothermic ($\Delta H > 0$) and physical in nature ($\Delta H > 50$ KJ/mol) (Djidel, 2011) in the temperature range of 298K and 338K.

INTRODUCTION

Water is used in many fields including agriculture, households, and industries. Many industries such as leather, textiles, use a lot of dyes. Water, after its use, is often loaded with pollutants such as heavy metals and dyes whose consequences are numerous on the environment. It is these consequences on the environment that have led to the improvement of existing depollution techniques and the development of new pollution control. Among these depollution techniques, there is electrolysis, flotation, precipitation, ion exchange, liquid-liquid extraction, membrane filtration. However, these processes are expensive and lead to the generation of large amounts of sludge or drift (Robinson et al., 2001). Adsorption is one of the most popular techniques for removing such pollutants because of its great ability to purify contaminated water. The activated carbon previously used as adsorbent remains very expensive in terms of costs and requires a regeneration which is a limiting factor. It is therefore in the light of this that research directed towards treatment processes using less expensive and widely available natural materials such as clay (Adjia et al., 2014), corn cobs (El-Hendawy et al., 2001 ; Cao et al., 2006), eggshells (Khelifi et al., 2016), palm waste (Hazourli et al., 2007).

Few studies in our opinion have used corncobs in the depollution of wastewater. The present study consists in activating the Ngaoundere-Cameroon corncobs with sodium hydroxide before its use in the depollution of water loaded with organic pollutants like Methylene Blue. To this end, we will prepare a synthetic solution of Methylene Blue in the laboratory and carry out the clean-up tests to follow the behavior of this new material in the elimination of this pollutant.

MATERIALS AND METHODS

Preparation of the bioadsorbent: The corn cobs are collected locally in maize fields at Ngaoundere Cameroon, dried and then crushed and sieved to obtain the powder of corn cobs smaller than 300 μ m, washed with distilled water and dried in an oven at 105 ° C put in soda base 0,1 N for 24 hours. Corn cobs powder and soda were used in proportion of 1/5 (weight of corn cobs powder/volume of soda) (Khormaei et al., 2007). In this way, the base activated corn cobs powder (BACCP) is obtained ready for use.

Preparation of the mother solution of Methylene Blue: We Weighed 1000mg of solid Methylene Blue which is dissolved

in 1 liter of distilled water. The resulting MB solution is 1000 mg / L.

Adsorption in batch mode: The adsorption in batch mode was done using a magnetic stirrer at a constant rate of 250tr/min on which was deposited a 250 mL beaker containing our BM solution of concentration 10mg / L at a volume of 20mL to which 100mg of corn cobs powder was added for predefined times. The corn cobs powder is separated from the solution using whatmann filter paper and the optical density is read from a spectrophotometer to determine the residual concentration using the calibration line.

RESULTS AND DISCUSSION

The influence of the contact time: We studied contact time using the initial concentration of 10mg / L, the MB volume of 20mL and a corn powder mass of 100mg. No pH adjustment of the solution. The result obtained gave us Figure 1 below. We got the contact time of 15min, needed to reach balance. Time from which we observe the stability of the curve.

Influence of the dose of the adsorbent: To study the influence of the dose of the adsorbent, the initial concentration of 10mg / L, a MB volume of 20 ml was used and the mass of corn cobs powder was varied from 100mg to 600mg in steps of 100mg keeping the pH of the natural solution without adjustment. Figure 2 below gives us the result of this experiment. The adsorption is maximum at the mass of 100mg which in the continuation of the work is considered as being the optimal mass. The efficiency of the elimination decreases when the mass of the adsorbent increases, this same tendency is also observed in the works of (Mira et al., 2016) when they eliminated Methylene Blue by three types of unactivated eucalyptus powder of different grain size. This behavior can be explained by the fact that, as long as the amount of adsorbent added to the dye solution is low, the dye cations can easily access the adsorption sites. The addition of adsorbent makes it possible to increase the number of adsorption sites, but the dye cations have more difficulty in approaching these sites because of the bulk. A large amount of adsorbent creates agglomerations of particles, resulting in a reduction in the total adsorption surface area and, consequently, a decrease in the amount of adsorbate per unit mass of adsorbent (Asmaa et al., 2010).

Influence of initial concentration: To study the influence of the initial concentration we varied the initial concentration from 10mg / L to 60mg / L in steps of 10mg / L, a volume of 20mL and a corn cobs powder mass of 100mg keeping the pH of the natural solution without adjustment. The result of this experiment is given in Figure 3 below. We observe that the curve has two levels: the first level when the curve increase, which results in the increasing of the efficiency of the elimination situated at the concentration less than 10 mg/L and the second step which shows a general decrease of the curve which results in the decrease of the efficiency of the elimination situated at the concentration more than 10 mg/L. We conclude that, the efficiency of the elimination is the function of the concentration of the solution. When the pollutant concentration increases, the efficiency of the removal decreases because of the fact that, at low concentrations, the ratio of the initial number of MB to the available adsorption surface is low, therefore the removal rate becomes

concentration independent, and at high concentrations saturation of the available adsorption sites is observed, and thus the adsorption becomes concentration dependent. Adsorption rates decrease when the dye concentration increases, this is an increase in competition at the adsorption sites, whereas competition decreases at active sites of the adsorbent at low concentrations (Dawood and Sen, 2012). The best performance is obtained at the concentration of 10 mg / L.

Influence of initial Ph: The initial pH of the colored solutions is a very important parameter to control the adsorption process (Tavlieva et al., 2013), it has an effect on the adsorbed quantity. It can change:

- The surface charge of the adsorbent ;
- The degree of ionization of the adsorbate ;
- The degree of dissociation of the functional groups of the active sites of the adsorbent (Nandi and Sunil, 2017).

To study the influence of the initial pH we used the initial concentration of 10mg / L, a volume of 20mL and a corn cobs powder mass of 100mg by varying the pH of the solution from 2 to 12 by adjusting the pH of the solution, using a solution of soda and sulfuric acid of normality 0,1N. The result of this experiment is given in Figure 4 below. To study the influence of the initial pH we used the initial concentration of 10mg / L, a volume of 20 mL and a corn cobs powder mass of 100mg by adjusting the pH of the solution from 2 to 12 using a solution of soda and sulfuric acid of normality 0,1N. The variation of the MB adsorption rate as a function of the pH is graphically represented in figure 4 below.

There is an increasing trend of the curve showing three levels. The first between $\text{pH} < 2$, the curve grows faster with the increase in pH, the second where $2 < \text{pH} < 8$, the curve grows less faster with the increase in pH and the third where $8 < \text{pH} < 12$, the growth of the curve is less sensitive with the increase in pH. The result shows that the removal efficiency increases and reaches 99,02% with pH increasing. The maximum elimination is reached at $\text{pH} = 12$. This increase in MB adsorption with increasing of pH is noted in the work of (Saritha et al., 2015).

Influence of temperature: The influence of temperature on the adsorption of MB was studied using beakers containing 20 mL of solutions each, immersed in a thermostatically controlled water bath to preserve the desired constant temperature, the values of the temperatures studied were 25; 35; 45, 55 and 65 ° C respectively. The whole is agitated during the contact times which is 15min and the filtra is analyzed spectrophotometrically. The result of this study is shown in Figure 5 below. The observation of the figure above, allows us to see that the percentage of removal of MB by corn cobs powder increases with the increase in temperature which explains the endothermic nature of the reaction. In addition, it is observed that an increase in temperature improves the adsorption capacity of the MB by the corn cobs powder. The effect of temperature on the adsorption of cationic dyes has been studied in many studies, most of them have noted a positive influence of temperature on the adsorption capacity (Doğan et al., 2007). Increasing the temperature facilitates diffusion of the adsorbed molecules to the internal pores of the adsorbent particles by decreasing the viscosity of the solution.

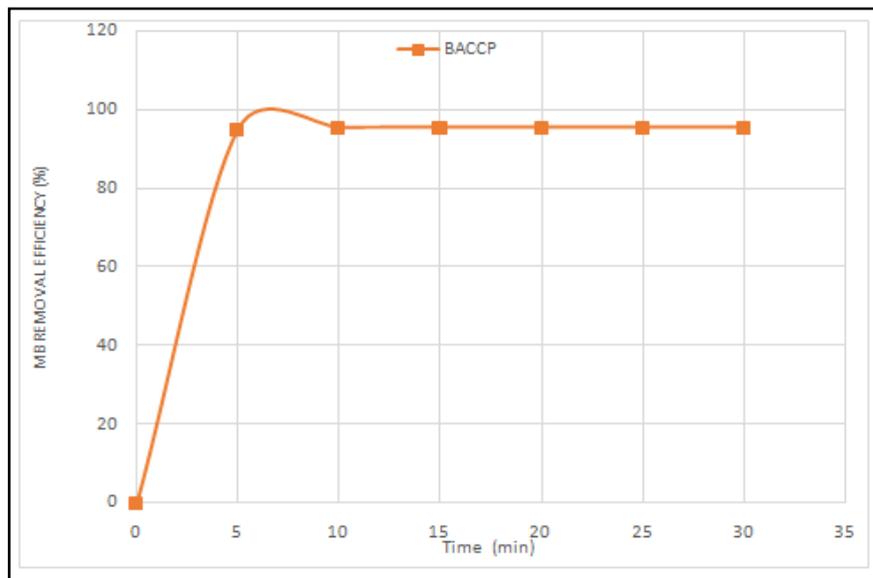


Figure 1. Influence of contact on adsorption of MB by BACCP

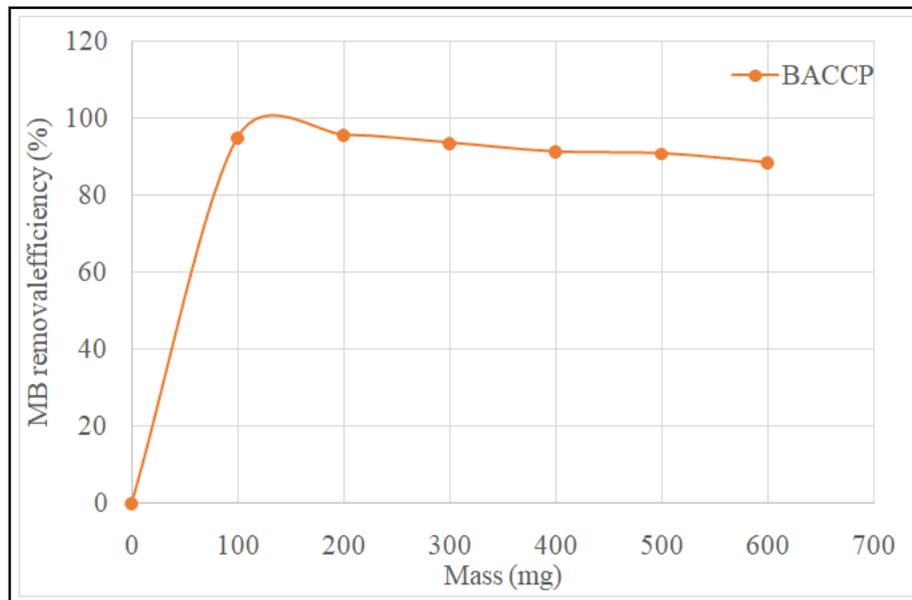


Figure 2. Influence of contact on adsorption of MB by BACCP

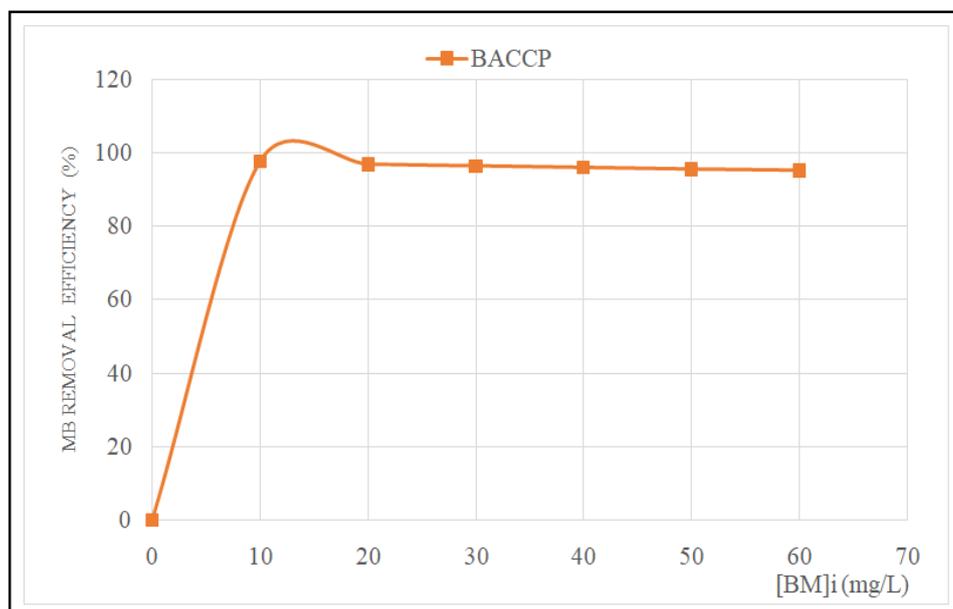


Figure 3. Influence of initial concentration of MB

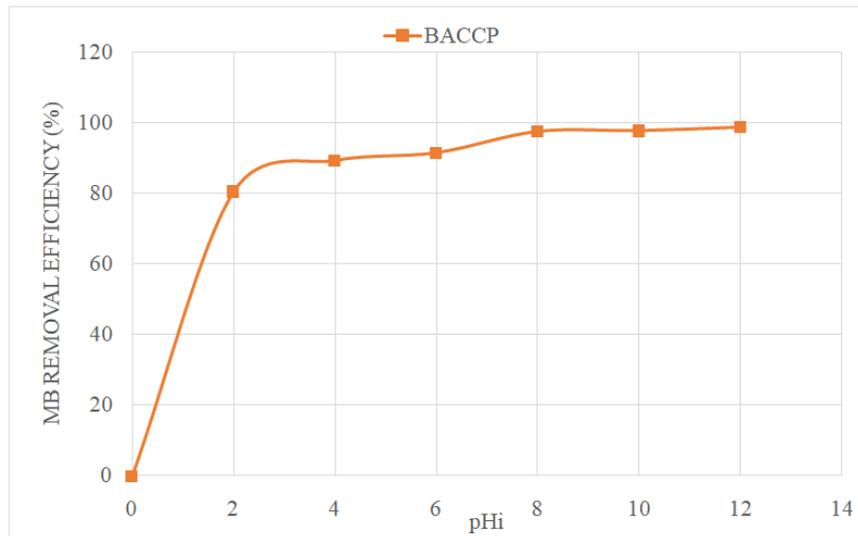


Figure 4. Influence of initial pH on MB adsorption

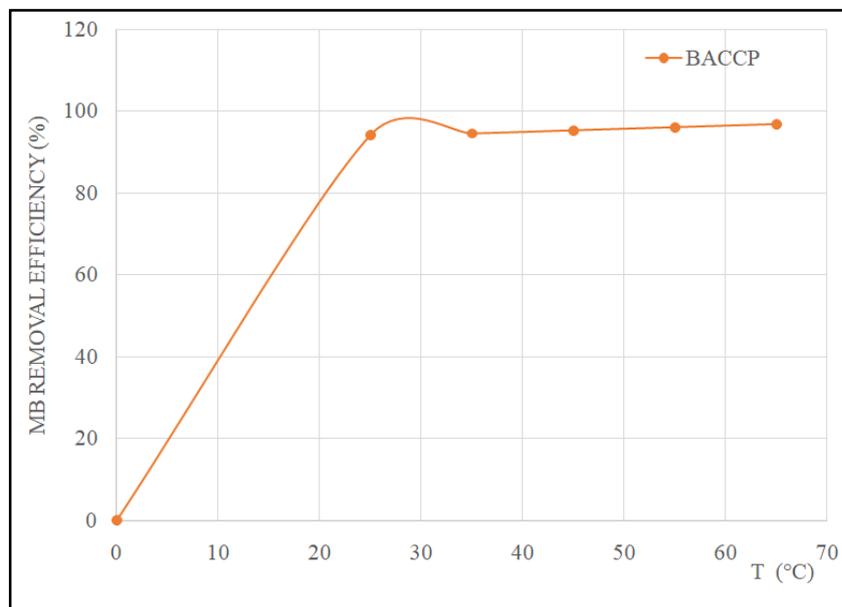


Figure 5. Influence of temperature on MB adsorption

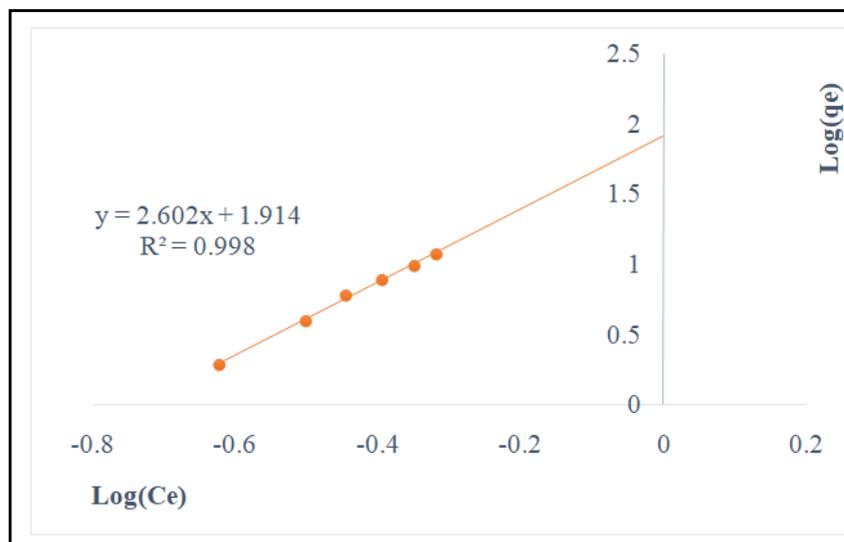


Figure 6. Freundlich Isotherm

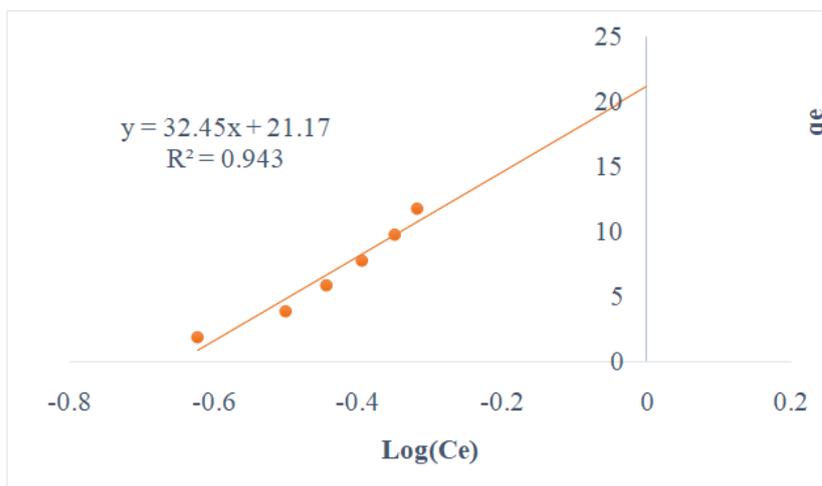


Figure 7. Temkin Isotherm

Table 1. Parameters of the Freundlich and Temkin adsorption isotherm

CORN COBS	Isotherm of Freundlich			Isotherm of Temkin		
	$K_f \text{ mg}^{(1-n)} \cdot \text{L}^n \cdot \text{g}^{-1}$	n	R^2	B	A (L/g)	R^2
BACCP	2,478	10,593	0,682	0,9871	4697,410	0,943

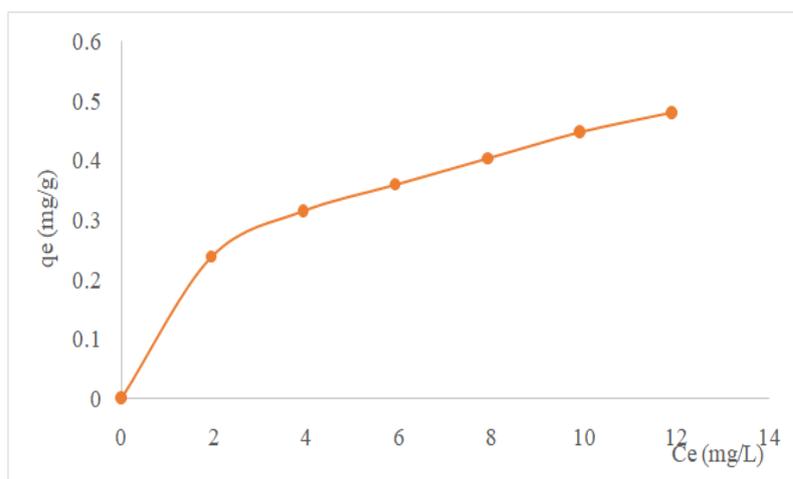


Figure 8. Isotherme d'adsorption du BM par PEMAB.

Adsorption isotherms

The Freundlich isotherm: The Freundlich model (1909) applies to many cases, in particular those of multilayer adsorption with possible interactions between the adsorbed molecules. The linear expression of the Freundlich equation is:

$$\ln q_e = \ln K_f + \frac{1}{n} \ln C_e \tag{1}$$

$K_f \text{ (mg}^{(1-n)} \cdot \text{L}^n \cdot \text{g}^{-1})$ is a constant relative to the adsorption capacity. $C_e \text{ (mg} \cdot \text{L}^{-1})$ is the concentration of BM at equilibrium and q_e is the adsorption capacity $\text{(mg} \cdot \text{g}^{-1})$, (Figure 6).

Temkin Isotherm: Temkin's (1940) model is based on the assumption that the adsorption heat due to interactions with the adsorbate decreases linearly with the rate of recovery during adsorption in the gas phase. It is an application of the Gibbs relation for adsorbents whose surface is considered to be energetically homogeneous.

(Limousin et al., 2007, Gimbert et al., 2008) have proposed using this model in the liquid phase, tracing q_e to $\ln C_e$ according to the following expression:

$$\frac{q_e}{q_{mqx}} = \theta = \frac{RT}{\Delta Q} \ln(K_T \cdot C_e) \tag{2}$$

$R = 8.314 \text{ J} \cdot \text{mol}^{-1} \cdot \text{K}^{-1}$
 T : absolute temperature (in K)
 ΔQ : variation of adsorption energy (in $\text{J} \cdot \text{mol}^{-1}$)
 K_T : Temkin's constant (in $\text{L} \cdot \text{mg}^{-1}$)
 $B_T = (q_m RT) / \Delta Q$ is the slope of the representation of the equation

$$q_e = B_T \ln A + B_T \ln X_e \tag{3}$$

The introduction of the value of q_m (for example from the application of Langmuir) makes it possible to calculate the variation of adsorption energy ΔQ , (Figure 7).

The following table 1 gives a summary of the adsorption isotherm constants of BM by corncobs.

According to the Freundlich isotherm, $n > 1$, then the reaction is favorable. $R^2 > 0.90$ in the case of the Temkin isotherm, this isotherm is used to describe the adsorption phenomena studied. In order to plot the variation of the amount of adsorbed MB per gram of adsorbent (q_e) as a function of the equilibrium concentration (C_e). MB adsorption isotherms were made by the corn cobs at 25 ° C, at normal pH of the solution without adjustment and at equilibrium time of 15 minutes using the following relationship:

$$q_e = \frac{(C_i - C_e).V}{m} \quad (4)$$

Where:

C_i : the initial concentration of the MB solution ;
 C_e : the concentration of the MB solution after adsorption ;
 m : the mass of the corn cobs powder used ;
 V : the volume of the BM solution.

The modeling results obtained gives us the following figure 8. This figure has two stages: a first narrow stage corresponding to a faster adsorption of the dye and the second which corresponds to the slowing down of the adsorption. The adsorption of MB is very strong at low residual concentration, which indicates a high affinity between the MB and the adsorbent. In addition, the curve shows that the amount adsorbed increases rapidly and reaches the maximum adsorption amount of MB which is 11.904 mg / g. It should be remembered that the increase in temperature has improved the adsorption of MB by corn cobs. The curve of figure 8 is in the form L, it corresponds to the low concentrations of solute in water. The isotherm is convex, which suggests a gradual saturation of the solid. It should also be noted that when this tends towards zero, the slope of the isotherm is constant. L forms is the form of the IUPAC classification. Our experimental results have been confronted with the theoretical models of Freundlich and Temkin. We have established the theoretical models on the basis of certain assumptions. The experimental data obtained, we seem to be in agreement with the hypotheses, this allows us to assume that the assumptions made are true for the experiment conducted. This allowed us to obtain information on the adsorption mechanism.

Conclusion

It was for us in this study to remove Methylene Blue from the wastewater using a bioadsorbent obtained from the basic activation of the native corn cobs powder. It is obvious that this study is a plus in the water treatment sector. The Methylene Blue solution studied is obtained by synthesis in the laboratory. We then studied the influences of certain parameters such as the contact time, the influence of the adsorbent dose, the initial concentration, the influence of the initial pH and the temperature that allowed us to analyze the phenomenon of adsorption. We also studied kinetics and thermodynamic parameters. The adsorption equilibrium was reached after 15 minutes. The experimental data are analyzed using the Freundlich and Temkin isotherms. The value of the Freundlich constant n_f obtained is greater than 1, we are in phase of a favorable adsorption. The Temkin model explains this phenomenon better: the adsorption heat due to interactions with the adsorbate decreases linearly with the recovery rate during adsorption.

The kinetic model of Elovich is applicable to this adsorption with $R^2 = 0.99$, which means that the adsorption sites increase exponentially with the adsorption, leading to a multilayer adsorption. The pH change showed that the adsorption increases with increasing pH and that the maximum adsorption is reached at pH = 12 giving a yield of 98.71%. The adsorption phenomenon observed is endothermic and of a physical nature. Basal activation improved the adsorption capacity of the corn cobs powder.

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