



Adsorption of Cadmium and Copper Ions in Aqueous Solution by Using Activated Carbon Resulting from Hulls of Neem

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Authors' contributions

This work was carried out in collaboration between all authors. Author RD designed the study, performed the statistical analysis, wrote the protocol and wrote the first draft of the manuscript. Authors MH and NLCF managed the analyses of the study. Authors SFG, GB and BBL managed the literature searches. All authors read and approved the final manuscript.

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ABSTRACT

The present work aimed at the adsorption of cadmium (Cd^{2+}) and Copper (Cu^{2+}) ions from an aqueous solution by activated carbon prepared from hulls of Neem by a simple process of pyrolysis preceded by a chemical impregnation in zinc chloride. The process was optimized on the basis of the analysis of the index of iodine of carbon prepared according to the parameters of the preparation. Prepared activated carbon was characterized by Fourier transform infra-red spectroscopy (FTIR), and by determination of the iodine number and the carbonization yield. To implement the adsorption capacity of activated carbon, of the contact time, adsorbent mass, and

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initial concentration of the adsorbate. The adsorption equilibrium is quickly reached at the end of 10 min and 15 min for Cd^{2+} and Cu^{2+} ions respectively. The maximum adsorbed quantities are of 8.382 mg/g and 52.356 mg/g for Cd^{2+} and Cu^{2+} ions respectively. The pseudo-second order models describe the kinetics adsorption of Cd^{2+} and Cu^{2+} ions. The use of zinc chloride, as chemical agent, favors the adsorption of molecule of very small sizes. The isotherms of adsorption of Cu^{2+} and Cd^{2+} on the prepared activated carbon are of type L and are compatible with the model of Langmuir. This study revealed that activated carbon obtained from *Neem* shells is a good adsorbent for the elimination of Cd^{2+} and Cu^{2+} ions from aqueous solution. This study aims to contribute to clean up the waters.

Keywords: Hulls of neem; activated carbon; adsorption; Cu^{2+} ; Cd^{2+} ; isotherms.

1. INTRODUCTION

The world interest carried in the environmental protection of solid waste led by various activities and human transformations, arose the attention of researchers to find the ways to value this waste [1]. Develop activated carbons from the plant waste is interesting from the economic point of view because it is from simple transformations that is made a direct application of these materials [2]. Activated carbons are titled expensive materials, they can be obtained by carbonization and precursor's activation already containing an important part of carbon and a low percentage in inorganic material [1]. Their manufacturing can be made of two ways: either by physical activation, or by chemical activation. Various studies were dedicated to the production and to the characterization of activated carbon resulting from materials of various vegetable sources: hulls of Balanites [3], granulate of Moringa [4], skiffs of Kola [5], skiffs of palms [6], swipe of corn [7], pits of date [8], shell of almond [9], sawdust of rattan [10]. It is in this perspective that joins this work, the purpose of which is to value hulls of neem in activated carbon. Indeed hulls of neem are materials resulting from extraction of oil. In Cameroon we find the neem in Regions of the North and the Far-North, where its oil is very used for its antiseptic, anti-inflammatory, antibacterial or immune-stimulant properties. What lets glimpse multiple possibilities as for it is not used hulls which can be valued as raw material in the preparation of activated carbon. These materials were valued in activated carbon by using zinc chloride as activating agent [8]. The objective of this work is to obtain an activated carbon with hulls of neem collected in Djamboutou, Region of the North Cameroon and to estimate its capacities of adsorption on two heavy metals in aqueous solution: Cu^{2+} and Cd^{2+} .

2. MATERIALS AND METHODS

2.1 Collection and Pre-treatment of the Raw Material

Hulls of neem used in this work were collected in the locality of Djamboutou, Region of the North Cameroon. They are abundantly washed in distilled water and then dried at room temperature during a week. They are then crushed and sieved to retain several particles of sizes between 1.25 to 2.5 mm.

2.2 Chemical Activation by Zinc Chloride

The reserved particles (20 g) are soaked in two solutions of zinc chloride (ZnCl_2) with a weight ratio of 1/1 and 1/2 respectively. The impregnation is made in a steam room for a temperature of 120°C for a while, about 10 minutes. The soaked particles are kept in flasks, hermetically closed until the trials of carbonization. The carbonization is realized in an oven preheated in an adequate temperature for a minimum of 2 hours before the beginning of the experience to obtain a temperature of state of balance. The carbonization is made for a temperature of 500°C during 1 hour. Obtained carbons are cooled at room temperature in a desiccator. To eliminate the possible residues of carbonization, activated carbons are washed several times in distilled water until the test in the silver nitrate turns negative. Carbons so washed are dried in the steam room in 110°C during 24 hours, cooled in a desiccator, and then crushed to obtain activated powder carbons. The obtained powder is kept shielded from air in flasks hermetically closed until the essays of characterization and adsorption.

2.3 Determination of the Index of Iodine

The index of iodine was determined by using the experimental protocol proposed by ASTM

(American Society for Testing and Materials). The method appears as follows:

1 g of dry activated carbon is put in touch with 10 mL of a solution of hydrochloric acid to 5 % in a clean and dry conical flask of 250 mL. We shake then we boil during 30 seconds, approximately. Introduce into the mixture 100 mL of a titled solution of iodine of concentration N_1 equal to 0.10 N. Block immediately and shake during approximately 30 seconds. To filter quickly the mixture on a filter paper sheet. A volume of 50 mL of the filtrate is titled by a solution of thiosulfate of sodium of concentration N_2 equal to 0.10 N. Starch solution was used as indicator of the end of dosage. We note the volume V (mL) of the solution of thiosulfate sodium used. We determine the quantity (X') of iodine adsorbed by the relation:

$$X'(\text{meq}) = 100 N_1 - \frac{(110 N_2 V)}{50} \quad (1)$$

With N_1 the normality of the solution of iodine, N_2 the normality of the solution of thiosulfate of sodium, V the volume of the solution of thiosulfate of sodium used for the dosage.

The index of iodine of the activated carbon is then calculated by using the following equation:

$$\text{Index of iodine} = \frac{X}{m} A \quad (2)$$

With A correction factor obtained after the calculation of the normality of the residual filtrate, m the mass (g) of activated charcoal, X the quantity mg from adsorbed iodine. X is given by:

$$X(\text{mg}) = 126.93 X'(\text{meq}) \quad (3)$$

2.4 Infra-Red Spectroscopy

For the qualitative determination of surface functional groups, an Infra-red spectrometer was used with a good resolution. The basic line is made with a pastille of KBr and the measures are made on powders.

2.5 Adsorption of Cu^{2+} and Cd^{2+} Ions in Aqueous Solution

The adsorption of Cu^{2+} and Cd^{2+} ions takes place at room temperature where 0.1 g is introduced into 20 mL of solution. After agitation, the mixture is filtered by means of a filter paper and the obtained filtrate is measured by UV-visible spectrophotometer in a wavelength of 810 nm for

Cu^{2+} and by volumetry for Cd^{2+} . The quantity adsorbed by unit mass of adsorbing (q_e) in the balance and the percentage of adsorption ($\%E$), are given by the relations (4) and (5) below:

$$q_e = \frac{(C_0 - C_e)}{m} \quad (4)$$

$$\%E = \frac{(C_0 - C_e)}{C_0} 100 \quad (5)$$

With C_0 the initial concentration of the adsorbate (mg.L^{-1}), C_e the concentration of the adsorbate in the balance (mg.L^{-1}), V the volume of the solution containing the adsorbate (mL) and m the mass of the activated carbon used (g).

2.6 Modeling of Adsorption in Aqueous Middle

To study the power adsorbing of activated carbon, the models of Langmuir and Freundlich were applied because of their simplicity and their easy interpretation.

2.6.1 Langmuir isotherm model

The isotherm is represented by the following equation:

$$q_e = \frac{q_m K_L C_e}{1 + K_L C_e} \quad (6)$$

Where, C_e is the equilibrium concentration of the remaining solute in the solution (mg.L^{-1}), q_e is the amount of the solute adsorbate per mass unit of adsorbent at equilibrium (mg.g^{-1}), q_m is the amount of adsorbate per mass unit of adsorbent unit at complete monolayer coverage (mg.g^{-1}) and K_L (L.mg^{-1}) is a Langmuir constant. The Langmuir isotherm equation can be rewritten as follows:

$$\frac{C_e}{q_e} = \frac{1}{K_L q_m} + \frac{C_e}{q_m} \quad (7)$$

The q_m and K_L values were calculated from the slopes ($1/q_m$) and intercepts ($1/q_m K_L$) of linear plots of C_e/q_e vs. C_e .

2.6.2 Freundlich isotherm model

Freundlich isotherm model can be used to describe the non-ideal adsorption of heterogeneous system and reversible adsorption. The isotherm is expressed by the following equation:

$$q_e = K_F C_e^{1/n_F} \quad (8)$$

Where, K_F (mg.g^{-1}) and n_F are Freundlich adsorption isotherm constants. Expression (8) can be linearized to give the following equation:

$$\ln(q_e) = \ln(K_F) + 1/n_F \ln(C_e) \quad (9)$$

The K_F and n_F values are determined from the intercept and slope of linear plots of $\ln(q_e)$ vs. $\ln(C_e)$.

3. RESULTS AND DISCUSSION

3.1 Characteristics of the Prepared Activated Carbon

3.1.1 Infra-red spectroscopy

Fig. 1 and Fig. 2 gives the records of spectrum to the frequencies of the various functional groups on the surface of the activated carbon before and after adsorption.

The analysis of the Infra-red spectrum of activated carbon before and after adsorption

allows to identify three major signals (Fig. 1 and Fig. 2):

- Peaks between 1563 and 1543 cm^{-1} correspond to the connections C=C of the aromatic pit.
- The signals 882 and 870 cm^{-1} correspond to the connections C – H of the benzene.
- Peaks corresponding to the frequencies of strain in 1074 cm^{-1} and 1038 cm^{-1} in both cases make the link for the connections S=O of the sulfone [4].

The results of the Infra-red shows that on the surface of activated carbon before and after adsorption, there are two main functions: benzene and sulfone.

3.1.2 Influence of the operating parameters on the index of iodine

The index of iodine gives an evaluation of the specific surface of an activated carbon, and is usually used to measure the porosity of the pores

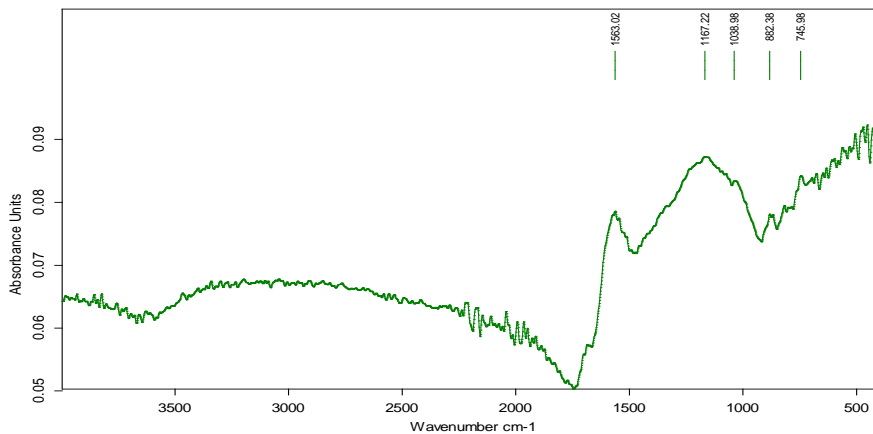


Fig. 1. Infra-red spectrum of the activated carbon before adsorption

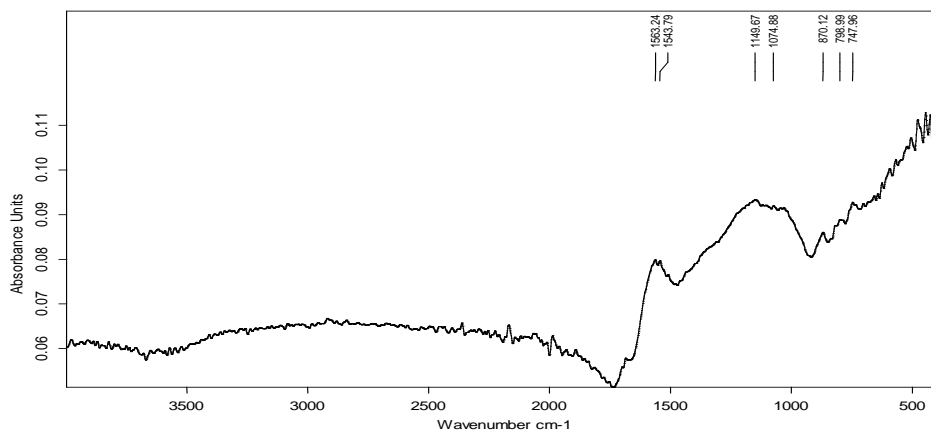


Fig. 2. Infra-red spectrum of the activated carbon after adsorption

of diameters bigger than 1.0 nm. So the index of iodine is used to estimate the capacity of adsorption of the activated carbon [1]. Fig. 3 gives the variation of the quantity of iodine adsorbed per gram of activated carbon used according to the ratio of impregnation and the concentration of the activating agent. The analysis of the results reveals that, the porosity of the activated carbon increases with the concentration of the activating agent and decreases when the volume of the latter increases. This could give some explanation by the fact that when the concentration of $ZnCl_2$ increases, chemical agents penetrate easily inside carbon fibre and this insertion contributes to the development of the porosity, where from a growth of the index of iodine. As for the rate of impregnation, more the proportion in activating agent grows, more it has decrease of the fixed quantity of iodine there. This gives some explanation by the fact that beyond the rate 1/1, there would be the beginning of saturation of pores to the detriment of the development of the porosity.

3.2 Adsorption of the Ions Cu^{2+} and Cd^{2+} on Activated Carbon

3.2.1 Adsorption kinetics

For every ion, 50 mg of activated carbon was introduced into reactors containing volumes of 20 mL of solution of Cu^{2+} at 1800 mg/L or of Cd^{2+} at 225 mg/L. Mixtures are carried in agitation with magnetic agitators SBS A-01, series C, constant

during progressive durations varying between 5 and 40 minutes.

The influence of the time of agitation on the adsorption of both ions on the activated carbon is represented on Fig. 4. The obtained curves allow to determine the time required to reach the balance enter adsorbent and every ion. The balance time is reached at the end of 10 minutes for Cd^{2+} and 15 minutes for Cu^{2+} . However, the adsorption kinetics, fast during the first minutes of reaction, can be interpreted by the fact that in the beginning of adsorption, number of the active sites available on the surface of the activated carbon, is much more important than that of the sites remaining after [11]. Similar results were obtained by Mounir et al. [12], on the adsorption of the arsenic (III) on pits of dates valued in activated carbon.

3.2.2 Kinetic models

Several kinetic models were used to examine the controlling mechanism of the adsorption processes on the experimental data obtained. The experimental data was fitted with pseudo-first order differential equation as follows:

$$\log(q_e - q_t) = \log q_e - \frac{k_1 t}{2.303} \quad (10)$$

Where, q_e and q_t are the amounts of heavy metal ions adsorbed ($mg.g^{-1}$) at equilibrium and time, t (min), respectively. k_1 is the rate constant of first order adsorption (min^{-1}). The first order rate, k_1 , can be obtained from the slope of the plot of $\log(q_e - q_t)$ vs. time.

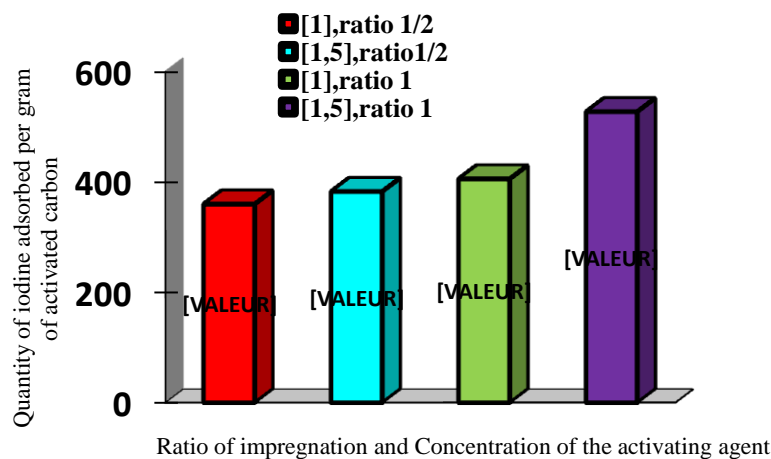


Fig. 3. Diagram of the influence of the operating parameters on the index of iodine

A pseudo-second order equation can be expressed in the form of:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad (11)$$

Where, k_2 is the rate constant of the second order adsorption (min^{-1}).

A good agreement of the experimental data with the second order kinetic model was obtained Fig. 5. The correlation coefficients, R^2 are greater than 0.98, which suggests a strong relationship

between the parameters and also explains that the process follows pseudo-second order kinetics. The adsorption process involves transport of the solute molecules from bulk solution to the surfaces of activated carbon followed by intra-particle diffusion/transport process [13].

3.2.3 Influence of mass

The study of the influence of mass of activated carbon was matched at the balances times for

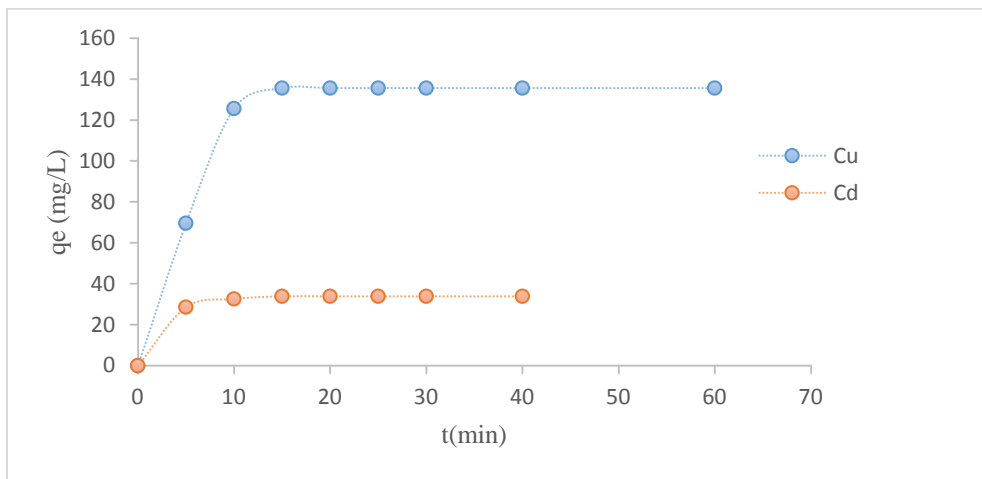
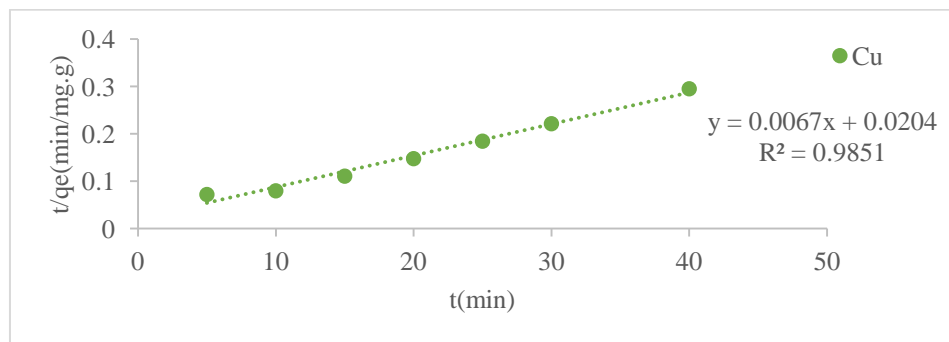
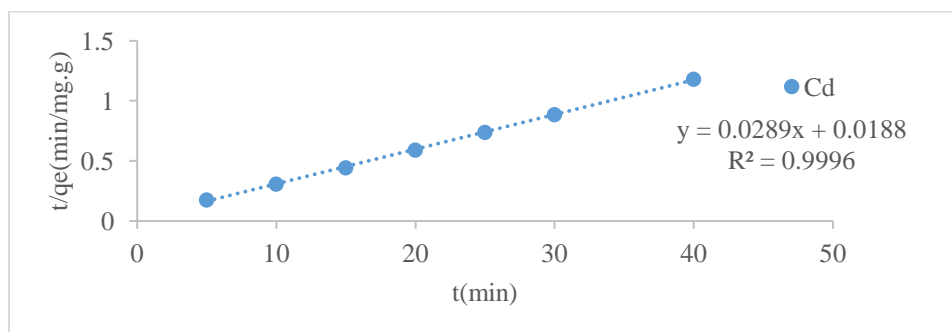


Fig. 4. Kinetic study for heavy metal ions uptake by activated carbon



(a)



(b)

Fig. 5. Plots for second order model for heavy ions uptake by activated carbon

both ions, in respective the concentrations of copper and cadmium at 1800 and 225 mg.L⁻¹ respectively. Fig. 6 show the results obtained for a variation of mass between 0.05 g and 0.4 g.

It reveals from Fig. 6 that, the adsorbed quantity decreases with increase of the mass. This can give explanation that in values of high masses, there is conglomeration of the particles of adsorbent so forming plops or aggregates. The formation of aggregates is governed by strengths of electrostatic attraction between particles and material that access to the site of adsorption is very difficult because there is increase of the route to be browsed by the pollutant. Similar results were obtained by Lékéné et al. [14] during the study of the adsorption of blue of methyl alcohol on the powder activated carbon and by Abega et al. [15] during the adsorption of Co²⁺ and Ni²⁺ on the activated carbon. Then, the mass of 0.1 g was retained for the continuation of the study.

3.2.4 Study of the adsorption isotherms of Cu²⁺ and Cd²⁺ on the activated carbon

Fig. 7a and Fig. 7b represents respectively the adsorption isotherms of the Cu²⁺ and Cd²⁺ ions on the activated carbon resulting from hulls of neem. The adsorption isotherms of these two bivalent ions on the activated carbon illustrated by Fig. 7 and Fig. 8 Shows that curves are well similar to the isotherm of type L, according to the classification proposed by Giles et al. [16]; characteristic type of microporous adsorbent. This type L is met when we attend a decrease of the available sites of adsorption, when the concentration of the solution increases. This phenomenon occurs when the strengths of attraction between the adsorbates are low, as meant Akra et al. [17] and Avom et al. [18]. The adsorption of Cu²⁺ and Cd²⁺ ions seems to be of the monomolecular type; the carbon being saturated during the filling of the monolayer.

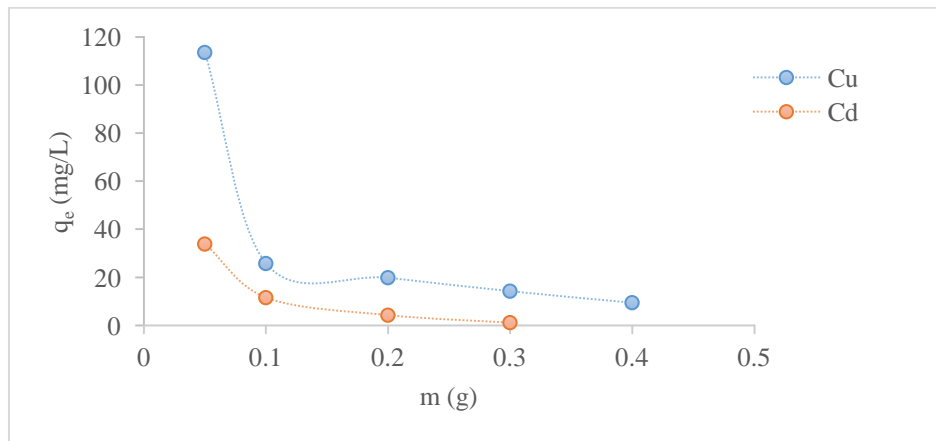


Fig. 6. Influence of the mass of activated carbon on the adsorption of heavy metal ions

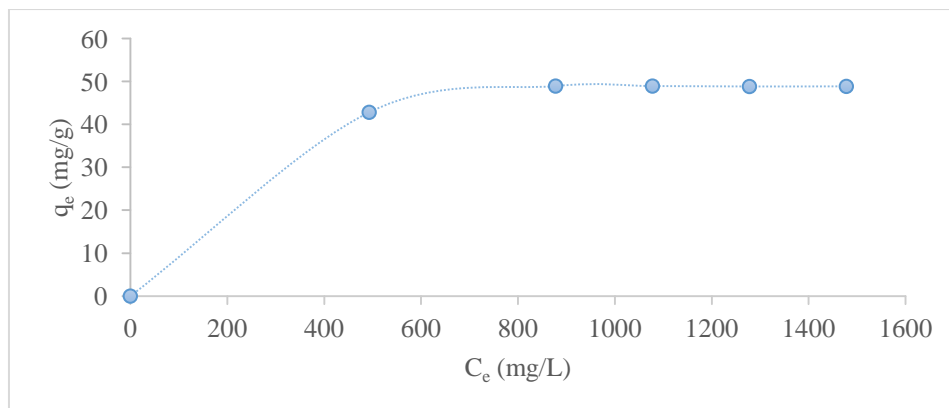


Fig. 7a. Adsorption isotherm of Cu²⁺ on the activated carbon

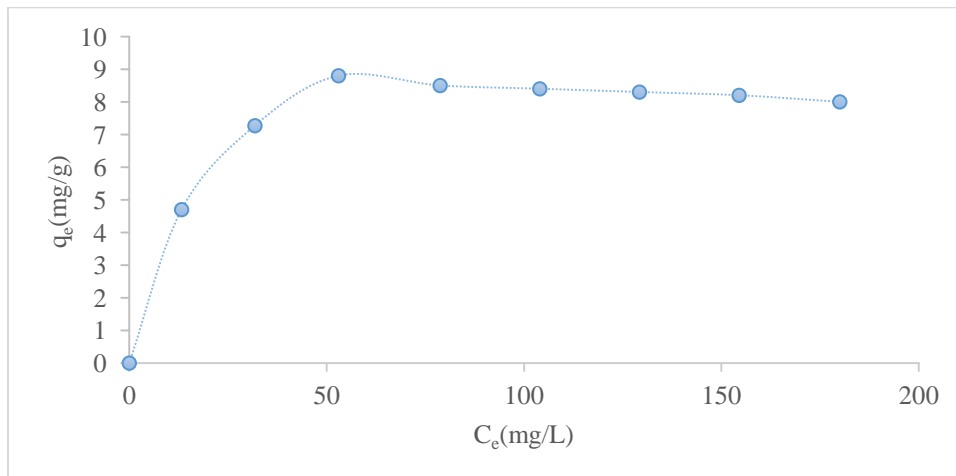


Fig. 7b. Adsorption isotherm of Cd²⁺ on the activated carbon

Fig. 7. Adsorption isotherm of Cu²⁺ and Cd²⁺ on the activated carbon

Table 1. Constants of Langmuir and Freundlich isotherms of adsorption of Cu²⁺ and Cd²⁺ ions on the activated carbon

		Cu ²⁺	Cd ²⁺
Langmuir	R ²	0.997	0.994
	K _L (L.mg ⁻¹)	11.32	10.44
	q _m (mg.g ⁻¹)	52.356	8.382
Freundlich	R ²	0.788	0.628
	K _F	3.665	1.726
	n _F	7.880	5.485

3.2.5 Modeling of the adsorption in aqueous middle according to Langmuir and Freundlich

Parameters values of K_L and q_m calculated from Langmuir model, parameters of Freundlich K_F and n_F, as well as the various coefficients of correlation of both models, are presented in Table 1. In this Table, the coefficients of correlation of the model of Langmuir get closer to the unit for both ions. Adsorption of Cu²⁺ and Cd²⁺ on the carbon resulting from hulls of neem can be described in a satisfactory way by the model of Langmuir. The high values of K_L and q_m, guarantee a good affinity and a good adsorption for this carbon. The coefficients correlation of the model of Freundlich are very low than the unit for both metallic ions. Consequently the model of Freundlich does not describe in a satisfactory way the adsorption of Cu²⁺ and Cd²⁺ on the synthesized activated carbon.

The Langmuir R_L separation factor is high for any adsorption phenomenon studied, which

translates to a reversible adsorption, implying a physisorption. The values 1 / n less than 1, it is unfavorable and could give Van Der Waals-type interactions leading to a physisorption [19]. This proves that the adsorption mechanism of the pollutants on our material is more guided by the affinity between the adsorbate-adsorbant and not the surface of the latter.

4. CONCLUSION

The objective of this work was the study of the Cd²⁺ and Cu²⁺ ions adsorption from aqueous solution on the activated carbon of the hulls of neem. The prepared activated carbon was characterized by FTIR spectroscopy, the index of iodine and the Burn-off. The analysis of the parameters such as the agitation time, mass adsorbent, pH of the solution and initial concentration of the adsorbate made it possible to determine the optimum conditions for adsorption. The study of the adsorption isotherms and the kinetic models made it possible to describe the mechanism of adsorption brought into play. The best conditions were established, with respect to pH and contact time, to saturate the available sites located on the activated carbon. This work revealed a fast attack of the balance of adsorption at the end of 10 min for the Cd²⁺ ions and of 15 min for the Cu²⁺ ions. The mechanism of adsorption brought into play showed the adsorption of the Cd²⁺ and Cu²⁺ ions follows the Langmuir isotherm. The kinetics adsorption revealed that pseudo-first order models better describe the adsorption of the Cd²⁺ and Cu²⁺ ions. This study showed maximum adsorbed quantities of 8.382 mg/g for

the Cd²⁺ ions and of 52.356 mg/g for the Cu²⁺ ions. *Neem* is a multipurpose plant with many attributes and considerable potential, functional groups that are considerably different to those of activated materials, which may lead to greater adsorption potential of activated samples. From this study, it is clear that the hulls of *Neem* can be used in manufacturing of activated carbon which is used in the process of water treatment, oil bleaching and filtration of food products.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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