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Adsorption of Copper Ions (Cu++) in Aqueous Solution Using Activated Carbon and Biosorbent from Indian Jujube (*Ziziphus mauritiana***) Seed Hulls**

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Authors' contributions

This work was carried out in collaboration among all authors. All authors read and approved the final manuscript.

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ABSTRACT

The present work aimed at mitigating the level of copper ions (Cu++) by adsorption in aqueous solution using activated carbon powder (AC) and biosorbent (BS) from the Indian jujube seed hulls. The AC and BS were prepared from the Indian jujube seed hulls and characterized using standard procedures. In addition, the adsorption effects of Indian jujube seed hulls through the AC and BS were carried out using jar test experiment (batch mode) at different pH (1, 2, 3, and 4), ionic strengths (100-600 mg/L) and stirring speed (120rpm). Therefore, it was found that the adsorption time at equilibrium were 10 and 5 minutes respectively for the AC and the BS. Furthermore, it was found that the reduction of copper ions were 99.40% and 73.08% for aqueous solutions of 100 ppm and 500 ppm respectively at the equilibrium state. It was also revealed that when the mass of the AC or the BS increases, the quantities of ions adsorbed per gram decrease. The maximum pH of adsorption for the AC was found to be pH=1, while it was found to be pH=4 for the BS. The Freundlich model indicated that the adsorption of copper ions by the Indian jujube is linear while the Temkin and Dubinin-Kagana-Radushkevich models described

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the adsorption as a physical reaction. It was finally observed that the adsorption of copper ions by the AC and the BS from Indian jujube seed hulls influenced by the addition of some concentration of NaCl.

Keywords: Ziziphus mauritiana seed hulls; activated carbon; adsorption; Cu++; isotherms.

1. INTRODUCTION

Copper ion (Cu++) exists in natural waters and is an essential micronutrient for human nutrition and wellbeing. It is involved in the growth and physiological maintenance of human structures like bones when taken in recommended concentrations. However, high level of copper ion (Cu++) in groundwater is a worldwide problem [1]. The World Health Organization (WHO) has set the concentration of 2mg/L as the maximum limit for copper ion (Cu++) concentration in potable water [2]. Concentration higher than this value taken intentionally can lead to death and several types of damage (kidney, liver) in severe cases [3]. Many methods have been developed for copper ion (Cu++) removal from water such as adsorption [4-5], ion exchange**,** and precipitation [6]. Among these methods, adsorption is a widely used method for the elimination of copper (II) ion which depends on ions (adsorbate) in fluid diffusing to the surface of a solid (adsorbent) when they are bound to the solid surface or are held there by weak intermolecular forces [7]. The adsorption method is recognized as the most efficient, promising, and widely used fundamental approach in water and wastewater treatment processes [8]. Different adsorbents such as waste sugar beet pulp [9], low cost activated carbon [10], natural and acid-activated clays [3], modified activated carbon [11], modified sand [12], and cashew nut shell [13], were tested to find out efficient and economically viable elimination of copper (II) ions. Among these adsorbents, activated carbons are widely used; this is attributed to their well-developed internal porosity. Iarge surface area and multiples functions at the surface which are active sites [14-15]. Activated carbons can be produced from any carbon containing organic materials, but the structure of the obtained carbon is closely linked to the structure of the raw material. However, the best raw materials for the preparation of the good activated carbon are materials having a high content of carbon and inexpensive [16-17]. As a result, wastes of lignocellulosic origin [18-20] are used, to obtain the low-cost active carbon. In the present investigation, the activated carbon was prepared using lignocellulosic waste of z*izuphus* *mauritiana* hulls [21]. The main objective is to study the removal efficiency of biosorbent and activated carbon of *zizuphus mauritiana* hulls as a potential adsorbent for copper ion (Cu++) removal under different physicochemical conditions.

2. MATERIALS AND METHODS

2.1 Materials

Ziziphus mauritiana seed materials were collected from Mayo-Oulo, a village in the northern part of Cameroon. The Biosorbent powder was obtained after scrubbing, drying, crushing and sieving the seed hulls with standardized stainless sieves and the fraction less than 80μ m was collected. All other reagents used in the present study were of analytical grade. A stock solution of copper ion (Cu++) 1000 mg/L was prepared by dissolving appropriate quantity of sulfate copper (Sigma-Aldrich, USA) in distilled water.

2.2 Preparation of Activated Carbon

The preparation of activated carbon was carried out by taking 50 g of biosorbent powder which were mixed in the weight ratio 1:1 with phosphoric acid 0.1 M, after 24 hours the mixture was then subjected to carbonization using a Nabertherm furnace, German (30-3000°C). The heating started at room temperature with a temperature increment rate of 10°C/min up to a final temperature of 400°C. The residence time of the samples at the final stabilized temperature of 400°C was 2 hours. After heat treatment, the activated carbon was cooled down to room temperature.

2.3 Characterization of the Activated Carbon

2.3.1 Determination of lodine number (Q_{12}) **of the activated carbon**

A sample of 0.2 g of activated carbon powder was placed into a 100mL beaker and 20 mL of 0.02 N solution of iodine (I_2) was pipetted and added. The mixture was stirred for a period of about 4 to 5 min and then filtered through a filter paper. An aliquot portion (10 mL) of the filtered solution was titrated with 0.1 N solution of sodium thiosulfate ($Na₂S₂O₃$). The Iodine number, Q_{12} was calculated using the formula below:

$$
Q_{I_2} = \left(C_0 - \frac{c_{th} \times V_{th}}{2V_{I_2}}\right) \left(\frac{M_{I_2} \times V_{ads}}{m_{ca}}\right)
$$
 (1)

Where,

 C_0 : Initial concentration of I_2 ;

 C_{th} : Concentration of sodium thiosulfate solution $(Na₂S₂O₃)$;

 V_{th} : Volume of $\text{Na}_2\text{S}_2\text{O}_3$ solution used to reach the end point;

 V_{I_2} : Volume of I_2 solution;

 M_{I_2} : Molar Mass of I_2 ;

 V_{ads} : Volume of adsorption;

 m_{ca} : Mass of activated carbon.

2.3.2 Determination of methylene blue number (Q_{BM}) of the activated carbon

2.3.2.1 Preparation of standard methylene blue solution

1.2 g of methylene blue crystals was measured and put into a 1 L volumetric flask. The volume was made up to the mark using distilled water and the solution was allowed to stand for 24 hours at room temperature. The solution was then tested with 0.25% acetic acid solution (5 mL for 1 L of solution). The concentration of the solution was determined by colorimetric method using a UV- spectrophotometer calibrated at $\lambda = 620$ nm.

2.3.2.2 Determination of methylene blue number (QBM)

100 g of activated carbon powder and 100 mL of the methylene blue standard solution were added into a 250 mL beaker. The mixture was then stirred for 20min using a magnetic stirrer and filtered through a filter paper. After filtration, the concentration of the residual solution of methylene blue was determined using a UV spectrophotometer calibrated at $\lambda = 662$ nm wave length.

The methylene blue number Q_{BM} was calculated using the following formula (2):

$$
Q_{BM} = V \times M \left(\frac{c_i - c_r}{m_{ca}} \right) \tag{2}
$$

Where,

V : Volume of the methylene blue solution;

M : Molar mass of methylene blue (295,5 g/mol);

 C_i : Initial mass the methylene blue solution;

 C_r : Concentration of the residual solution methylene blue solution;

 m_{ca} : Mass of activated carbon used.

2.4 Batch Adsorption Studies

Adsorption experiments were carried out on a jar test (batch mode) at different pH (1, 2, 3 and 4) ionic strengths (100-600 mg/L) and stirring speed (120rpm). In a typical run, beakers (50mL) were filled with 20 mL of copper ion (Cu++) solution of predetermined concentration, with pH adjusted to the desired value with 0.1 M NaOH or 0.1M HCl solution. The ionic strength of the solution was also adjusted by adding NaCl solution. A predetermined amount of adsorbent was added to the beaker and the resulting suspension was stirred at a fixed speed. After a given time, the stirring stopped, the suspension was filtered on whatman paper N°1. The residual copper ion (Cu++) concentration in the aqueous solution was measured by lecture
on UV-visible spectrophotometer on UV-visible spectrophotometer (spectrophotometer PRIM Light RS232, France). Isotherm experiments were carried out at $pH = 4$, by varying copper ion (Cu++) concentration ranging from 100 to 500mg/L. The amount of copper ion (Cu++) adsorbed (Qe) was calculated as the change in the aqueous phase concentration from initial value according to equation (1).

3. RESULTS AND DISCUSSION

3.1 Characteristics of Activated Carbon

The Table 1 presents values of Iodine index, Methylene blue (MB) index and the pH as characteristics of the activated carbon from the hulls of *zizuphus mauritiana*. These values showed that our activated carbon powder is primarily made up of meso and macro pores.

Table 1. Characteristics of the activated carbon from the hulls of *Zizuphus Mauritiana*

Parameters	Values
lodine Index	418.77 mg/g
MB Index	402.84 mg/g
рH	6.5

3.2 Adsorption in Aqueous Solution

3.2.1 Effect of contact time

The adsorption of copper ion (Cu++) in aqueous solution of initial concentration 500 mg/L with neutral pH is carried out on 0.05 g of activated carbon (AC) and biosorbent (BS) for increasing times of agitation give the results represented in Fig. 1. From these curves, it comes out that the adsorption of copper ion (Cu++) respecting the activated carbon and biosorbent is fast during the first 5 minutes to reach the balance which is 10min for the AC and 5min for BS with adsorbed quantities maximum which are 34.16 mg/g and 121.84 mg/g respectively. The fast speed of adsorption of the first phase (fast phase) can be explained by the fact that at the beginning of the phenomenon, the sites of adsorption are free on the surface of our adsorbent materials. Once that the copper ion (Cu++) are fixed, they block the pores, thus slowing down the speed of adsorption, there is apparition of a plate of saturation, from where the second phase (slow phase). This trend agrees with the report of other investigators [22].

3.2.2 Effect of adsorbent dose

The studies are carried out with a mass alternative from 0.010 g to 0.100 g; a time of agitation of 10 min and 5 min respectively for the AC and BS; a volume of 20 mL for 500 mg/L of concentration. The Fig. 2 is noticed, that the quantities of pollutants adsorbed decrease with the increase of the mass of adsorbent and tend to be stabilized for great values of masses. This can be explained by the fact that, the increase of mass involves a growth of the electrostatic interactions between the particles of the adsorbent which leads to the desorption of the copper ion (Cu++) of the sites of the adsorbents, thus led an agglomeration of the particles of the adsorbent forming aggregates; there is reduction in the surface of contact by the fact that active sites of the activated carbon (AC) or biosorbent (BS) become masked. Consequently, there is increase in the way of diffusion of the known as ions with respect to the active sites. These results agree with those observed by other authors [23]. The mass of 0.01 g of activated carbon and Biosorbent were retained for the continuation of our work for the two adsorbents; because the quantity adsorbed are maximum with this value.

3.2.3 Effect of pH on copper ion (Cu++) adsorption by AC and BS

This study is carried out starting from the solutions of copper ion (Cu++) of initial concentration of 500 mg/L; with a mass of 0.01 g of the AC or BS; a volume of 20 mL and with times of contact. The effect of pH was studied in the range from 1.0 to 4.0 and the results are represented by Fig. 3. It arises that the adsorbed quantity of the copper ion (Cu++) increases with the pH for BS and decrease for AC, this increase can be due to the fact that the surface of BS would be negatively in charge with values of weak acid pH, which supports the adsorption of the copper ion (Cu++). On the other hand, for values of weak acid pH, the surface of activated carbon would be charged positively and thus

Fig. 1. Effect of contact time on sorbent-sorbate interaction time

Fig. 2. Effect of AC or BS dose for the uptake of copper ion (Cu++)

likely to push back the cations of copper ion (Cu++). In addition, lower adsorption of copper ion (Cu++) at acidic pH on AC might be due to the presence of excess $H⁺$ ions competing with cations for the available adsorption sites. Similar results were observed by other authors [11]. Furthermore, it is noted that the adsorbed quantity is maximum with pH equal to 4.0 for BS and with pH equal to 1.0 for AC. This can be explained by the fact that in weak acid zone, the load of surface of the the biosorbent is negative and the one of activated carbon is positive. Similar results were observed by other authors [5].

3.2.4 Effect of ionic strength on copper ion (Cu++) adsorption by AC and BS

The Fig. 4 shows that, the adsorption of copper ion (Cu++) decreases then increases gradually with the addition of the small quantities of NaCl with AC, while for BS the adsorbed quantities increase and stay constant with the addition of small quantities of NaCl salt. The NaCl addition

Fig. 3. Effect of pH on copper ion (Cu++) adsorption by AC and BS

led to the increase in the adsorption of metal. The addition of salt supports the process of bringing together association of the particles of the BS and the AC, therefore the formation of a new porosity and new sites of surface where the ions and the aggregates of copper ion (Cu++) would be trapped [24]. Several intermolecular forces were suggested to explain this aggregation, these forces include: the forces van der Waals [25]; forces of dipole-ion and the forces dipole-dipole which occur between the molecules of the copper ion (Cu++) in solution. Consequently, the rise in the capacity of the adsorption of the copper ion (Cu++) under these conditions can be allotted to the aggregation of these last armatures by the action of the NaCl ions, in other words, the copper ions (Cu++) are bound increasing the extent of the adsorption on the surface of BS and decreasing of AC.

3.3 Kinetic Study

The kinetic study was carried out in an agitated vessel as described earlier by changing the various operating conditions. The pseudo-firstorder, pseudo-second-order, and intraparticle diffusion models were used to test the adsorption kinetics data to investigate the mechanism of adsorption. The pseudo-first-order rate expression of Lagergren is given as [26].

$$
\ln (Q_e - Q_t) = \ln Q_e - K_1 t \tag{3}
$$

where Q_t (mg/g) is the amount of adsorbed copper (II) ion at time t, k_1 (min⁻¹) is the rate constant of the first-order reaction, and Q_{ℓ} is the equilibrium sorption uptake derived from extrapolation of experimental data at time $t =$ infinity. A straight line of $ln(Qe - Qt)$ versus t suggests the applicability of this kinetic model (Fig. 5). Qe and k_1 (Table 2) were determined from the intercept and slope of the plot, respectively.

The pseudo-second-order kinetic model is integrated and linearized form has been used:

$$
\frac{t}{Q_t} = \frac{1}{K_2 Q_e^2} + \frac{t}{Q_e}
$$
 (4)

The plot of t/q_t versus t (Fig. 6) gave a straight line showing that second-order kinetic model was applicable and Q_t and k_2 (Table 2) were determined from the slope and intercept of the plot, respectively. From Table 2, the values of correlation coefficients obtained for the pseudofirst-order reaction were not appropriate to describe the adsorption of copper (II) ion on AC or BS. On the contrary, the values of correlation coefficients for pseudo-second-order model were all greater than 0.95 and the adsorption capacities calculated by the model are close to those determined by experiments. Hence, the data was well correlated by the pseudo-secondorder model as shown in Fig. 7. This fact suggested that the rate of copper ion (Cu++) adsorption onto AC and BS is dependent on the availability of adsorption sites rather than the adsorbate concentration in solution [27].

Fig. 4. Effect of ionic strength on copper ion (Cu++) adsorption by AC and BS

Fig. 5. First order kinetic modeling of copper ion (Cu++) adsorption on AC and BS

Fig. 6. Second order kinetic modeling of copper ion (Cu++) adsorption on AC and BS

To evaluate the limiting rate of the copper ion (Cu++) adsorption on AC and BS, the possible contribution of intraparticle diffusion on copper ion (Cu++) adsorption process was explored using Weber-Morris model [28]:

$$
Q_t = k_{int}\sqrt{t} + c \tag{5}
$$

where k_{int} can be calculated from the slope of the plot of q versus $t^{1/2}$. As shown in Fig. 7, the linear portion of plot is not passing through the origin at each of the tested concentrations, which indicates copper ion (Cu++) adsorption on AC and BS is a complex procedure. Both the surface adsorption as well as intraparticle diffusion contributes to the rate determining step [29].

3.4 Adsorption Isotherms

Copper ion (Cu++) adsorption was carried out at pH=4 for BS and pH=1 for AC with stirring speed of 120 rpm. Four isotherms equations have been applied for this study, Langmuir, Freundlich, Temkin and D-R-K. The linear form of the Langmuir equation is given by equation (5).

$$
\frac{1}{\mathbf{Q}_e} = \frac{1}{\mathbf{Q}_m \mathbf{K} \mathbf{C} e} + \frac{1}{\mathbf{Q}_m} \tag{6}
$$

where Qe and Ce are the equilibrium concentrations of copper ion (Cu++) in the adsorbed (mg/g) and liquid phases (mg/L), respectively. Q_m and b are the Langmuir

where Qe and Ce are the equilibrium concentration of copper ion (Cu++) in the adsorbed (mg/g) and liquid phases (mg/L), respectively. k_F and n are the Freundlich constants which are related to adsorption capacity and intensity, respectively. These constants can be calculated from the slope and intercept of the linear plot, with log Q_e versus log

constants which are related to the adsorption capacity and energy of adsorption, respectively, and can be calculated from the intercept and slope of the linear plot, with $1/Qe$ versus Ce . The adsorption equilibrium data was also applied to Freundlich isotherm model. The linear form of the model can be expressed as follows:

$$
\ln Q_e = \ln K_F + \frac{1}{n} \ln C_e \tag{7}
$$

Ce.

Fig. 7. Intraparticle diffusion kinetic modeling of copper ion (Cu++) adsorption on AC and BS

Table 2. Kinetics parameters for adsorption of copper ion (Cu++) on AC and BS

Models	Pseudo-first order			Pseudo-second order			Intraparticle diffusion		
Parameters		⊶⊾	R ²	K٥	Je.	R^2		$\mathsf{K}_{\mathsf{int}}$	R ²
	$(min^{-1}$	mg/g)		(q/min.q)	(mg/g)		$m^2.S^{-1}$	(mq.L.min ^{1/2})	
AC	-0.02	2.40	0.56	-0.13	33.2	0.99	33.015	0.11	0.04
BS	0.223	$1.2x 10^{-9}$	0.08	-0.04	119.04	0.99	1212	-0.147	0.03

Fig. 8. Langmuir adsorption isotherm of copper ion (Cu++) adsorption on AC and BS

Table 3. Isotherms parameters for adsorption of copper ion (Cu++) on AC and BS

Fig. 9. Freundlich adsorption isotherm of copper ion (Cu++) adsorption on AC and BS

To show the ability of different isotherms to correlate with experimental results. the correlate with experimental results, the theoretical plots from each isotherm have been fitted with the experimental data for adsorption of copper ion (Cu++) on AC and BS (Fig. 9). Langmuir and Freundlich parameters and regression coefficients (R^2) are presented in Table 3. From Fig. 9, the adsorption data fitted well with Freundlich isotherm model indicating the heterogeneous distribution of active sites on AC and BS [22]. The value of the correlation coefficient is 1.0, indicating a good mathematical fit. The Langmuir plot model has equal R^2 with the Freundlich model. To predict the adsorption efficiency of the adsorption process, the dimensionless equilibrium parameters were determined by using the following equation:

$$
R = \frac{1}{1 + bc_0} \tag{8}
$$

where C_0 is the initial concentration and b is the Langmuir isotherm constant. The value of R varied from 1.0 to 0.84. These values are less or equal to 1, indicating the favorable adsorption of copper ion (Cu++) on AC and BS [30].

4. CONCLUSION

Adsorption of copper ion (Cu++) onto AC and BS from the *Zizuphus mauritiana* seed hulls collected from the Northern Cameroon has been experimented in this study. It was revealed that the AC and BS are composed of different oxides. Kinetic studies reveal that equilibrium is attained within 10 min for AC and 5 min for BS at

respectively 1 and 4 pH studied. The maximum adsorption concentration of copper ion (Cu++) is 34.16 mg/g for AC and 121.84 mg/g for BS at a stirring speed of 120 rpm. The adsorption followed pseudo-second-order and both the surface adsorption and intraparticle diffusion contributed to the rate determining step. Freundlich isotherm fits the adsorption data adequately. The effect of ionic strength has a significant effect on copper ion (Cu++) adsorption. Therefore, it is important to note that the *Ziziphus mauritiana* seed hulls could be of upmost importance in industrial application for removing copper ion (Cu++) from water despite their very low utilization.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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