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Adsorption Study of the Removal of Copper (II) Ions using Activated Carbon Based Canarium Schweinfurthii Shells Impregnated with $ZnCl_2$

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ABSTRACT

The adsorption of Cu^{2+} ions on activated carbon based canarium schweinfurthii impregnated with $ZnCl_2$ was studied. The shells of canarium schweinfurthii were impregnated with $ZnCl_2$ at varying $ZnCl_2$ concentrations, temperature, residence time while keeping the heating rate fixed at 10 °C / min and the ratio of impregnation of 1:1. The activated carbon with the highest surface area in term of iodine number of 860,817 mg/g, the highest methylene blue of 741,6 mg/g and 74,66 % of yield of carbon was obtained at 650 °C, 60 % $ZnCl_2$ and 30min. From the batch adsorption studies, the equilibrium time was found to be 40 min. Analysis of equilibrium isotherm models revealed a good correlation of the experimental data with the Tempkin (R^2 = 0.909) model. This confirms a high affinity of the activated carbon for Cu^{2+} ions on the heterogeneous surface. The value of energy obtained from the Tempkin model was 60,606 J/mol and the presence of pics between 487 cm^{-1} to 871 cm^{-1} indicating that physisorption and chemisorption were taking place during this sorption. The pseudo-second order kinetics(R^2 = 0.999) governs the adsorption of Cu^{2+} ions on this activated carbon.

Keywords: adsorption, physisorption, chemisorptions, activated carbon, isotherm, kinetics.

Introduction

Nowadays, water pollution by dyes, pesticides and heavy metals in particular is an environmental calamity. Indeed heavy metals like copper, zinc, cadmium cobalt nickel are particularly dangerous because they are non-biodegradable, toxic even at low doses, and they can-accumulate in the human body [1]. Copper is an .essential trace element for human metabolism, however, it is toxic at concentrations above 2mg/L [2]. Ingestion of large doses irritate the mucous membranes, damage the hair and can cause necrotic changes in the liver and kidneys [3].Copper is present in wastewaters coming from energy industries (such as PILCAM in Cameroon), the production of certain pesticides and in the manufacture of contraceptives in medicine. [2]

Faced to this problem it is urgent to reduce their quantity, in order to minimize their impact on the environment. Several methods such as reverse osmosis, electrolysis, ion exchange, membrane separation, sedimentation, coagulation, flocculation and adsorption have emerged for the removal of pollutants from waters. Adsorption is most commonly used because its simplicity, economical and easier to operate [3].Its exploitation requires the use of adsorbent on which pollutants are capable to be attached on their surface. Many adsorbents such as are zeolites, silica gel, clay and activated carbon are being used. Activated carbon is the most effective adsorbent used in industrial water treatment because of it large surface area which makes it a powerful adsorbent [4, 5]. It is usually produced in powder form or as grains, depending on the type of pollutant to be adsorbed.

In this work, activated carbon was prepared from the *canarium schweinfurthii* shells, a readily available equatorial and tropical fruit. This carbon will be tested for its ability to remove Cu^{2+} ions from aqueous solution. Equilibrium and kinetics adsorption studies were carried out to certify, without risk of error, that this carbon can be used for such purposes in the future. Kana J. R and al (2014) have done the Growth performance and carcass characteristics of broiler chickens fed diets supplemented with graded levels of charcoal from maize cob or seed of *Canarium schweinfurthii* England not give any information about the characteristics about this biochar. A.S. Olawale and all have done the Thermal Activation of *Canarium Schweinfhurthi* Nutshell and obtained the specific surface of 41 1.99m^2 /g. Bassey and al. have done Adsorption Isotherm, Kinetics and Thermodynamics Study of Cr(VI) ions onto Modified Activated Carbon from endocarp of *Canarium schweinfurthii* and not give any information about the characteristic. Additionally, as far as our knowledge there are no much works dealing with preparation of activated by chemical activation of *canarium schweinfurthii*.

Materials and method

Preparation of adsorbent and adsorbate

From the results of TGA analysis on the biomass, we varied the carbonization temperature from 500-800°C. The residence time was varied from 45 to 120 min, the concentration of $ZnCl_2$ (45 to 60%), and at constant impregnation ratio of 0.6. A volume of 10 mL of $ZnCl_2$at desired concentration was mixed uniformly with 10g of *canariun schweinfurthii.*, shell and placed in an oven (brand) for 24 hours. After carbonization, the impregnated char was washed with distilled water until disappearance of precipitates of silver chlorine. The activated carbon obtained was dried, crushed and sieved using an 80 µm diameter sieve.

Characterization

Thermogravimetric Analysis (TGA) was carried out on the biomass using a LINSEIS STA PT-1000 instrument. The measure of the specific surface area of the activated carbon was obtained through iodine number and

methylene blue adsorption [6,7].The pH of zero point charge was determined by plotting the initial pH as a function of the final pH of a mixture of NaCl and adsorbent. [5, 8,9]. Fourier Transform IR was carried using BRUKER Alpha-P using ethanol as solvent, and I/PSEM2, EDX microscope.

Adsorption studies

Batch adsorption studies were carried out to study the adsorption of Cu^{2+}ions on the activated carbon. The pH of the Cu $^{2+}$ions solution was adjusted using 0.1 M HCL and 0.1 M NaOH.

A certain mass of the adsorbent is added to 20 ml of copper solution and stirred for a predetermined time at room temperature. After equilibrium, the solution was filtered using a Whatman No 1 filter paper and the residual concentration of copper determined using a Techmel & Techmel. USA S 23A UV spectrophotometer. The amount adsorbed (Q_e) was calculated using Equation 1,

$$Q_e = \frac{(C_o - C_e)}{m} \times V \quad (1)$$

where, C_o and C_e is the initial and equilibrium concentration of adsorbent (mg/l), V is the volume of the solution (L), and m the mass of adsorbent.

The influences of the parameters such as pH of the solution, the concentration of the adsorbate, the mass of adsorbent and equilibrium time were studied. Kinetic studies were conducted by varying the time between 10-120 mins with 0.025 g of adsorbent and 20mL of adsorbate.

Results and Discussion

Characterization of adsorbent

SEM-EDX

Figure 1shows the surface morphology of the ACZ as determined by SEM analysis, with the presence of pores on the surface of the ACZ compared to the biomass. An accumulation of zinc ions on the activated carbon surface resulting from the complexation of zinc with -C=O surface groups is observed. The presence of Zn^{2+} and Cl^- ions on the activated carbon surface will be further confirmed by FTIR results, confirming the modification of the activated carbon by $ZnCl_2$ [4]

Figure 1: SEM of the biomass (a) and the ACZ(b)

The Iodine Number

Iodine number determination gave an idea on the specific surface area. For the ACZ, the values of 860,817 mg/g and 741, 6 mg/g. are the iodine and methylene blue numbers respectively.

FTIR

FTIR spectra of the biomass (a), $ZnCl_2$activatedcarbon (b) and $ZnCl_2$ activated carbon after adsorption of Cu^{2+} (c) are in the Figure **(2).**The band at 3352.685 $cm^{-1}$of the biomass is characteristic of -OH vibration of water. This band is shifted to 2996.823 cm^{-1} for the activated carbon due to the effect carbonization. The weak band at 744.451cm^{-1}ischaracteristic of a halogen containing compounds and can be attributed to the C–X stretch of chlorine heterocyclic molecules. The bands from 1000 to 1300 cm^{-1} are attributed to the C-O-C-aromatic vibration. The bands between 2912-2998 cm^{-1} represent -CH_2asymmetric stretching vibration and confirm the presence of methylene groups. The band at 1624 cm^{-1} confirms the presence of ethers. The figures 2a, 2b and 2c are the FTIR of biomass, activated carbon AC-$ZnCl_2$ before and after adsorption respectively.

Batch adsorption studies

Effect of initial pH Cu^{2+} adsorption

The influence of pH on the adsorption of Cu^{2+}ions using the $ZnCl_2$ activated carbon was studied by varying the pH from 2.23 to 5. The amount adsorbed increases with pH up to a maximum of 36.4 mg / g at a pH of 4 (Figure 3). The pH of zero-point charge of ACZ was found to be 7,1, hence, the surface is positively charged at pH lower than 7, and negatively charged at pH greater than 7,1. The Cu $^{2+}$ ions which are positively charged can easily establish the bonds with the **-C = O,** and**-COO** surface groups on the ACZ, thus the high adsorption of Cu^{2+}ions at pH less than 5 [10, 11,12,13].. This trend can also be explained by the contribution of Cl^- attachment on the adsorbent which changes the nature of the surface thus promoting the affinity with the Cu $^{2+}$ ions [14, 15, 16]. Others studies have reported that the maximum adsorption of Cu^{2+}ions occurred at low pH (Q = 88.8mg/g for pH = 4) [12,13,17,18], and that Cu^{2+}ions precipitates at pH greater than 4. The major mechanisms responsible for metal uptake in this range may be attributed to the ion exchange and electronic

attraction. Through adsorption may give best results at pH values greater than 7,1 because of the affinity and absence of repulsion effect. To avoid precipitation, the pH equal to 5 was used

Effect of contact time

The contact time was varied from 10 to 120 min, at constant concentration of 600 mg/L. This variation is represented in Figure 4 below. The equilibrium time was reached after 30 min with 52.4 mg/g of Cu^{2+}ions adsorbed.

Effect of the adsorbent dose

The mass of adsorbent was varied between 0.025 to 0.1gat a constant initial concentration of 600 mg/L at the equilibrium time of 30 min and pH of 4. The quantity of Cu $^{2+}$ions adsorbed as a function of the adsorbent dose is given on Figure 5 below. The quantity adsorbed seems to decrease with an increase in the adsorbent dose. This may be due to the aggregation on the adsorption sites as the amount of adsorbent increase [19,20,21,22]. The maximum quantity of Cu^{2+}ions adsorbed at these conditions was 88,4 $mg.g^{-1}$ with 0.025g of adsorbent.

Effect of the initial concentration Cu^{2+}ions

The quantity of Cu^{2+}ions adsorbed increased with increasing Cu^{2+} ion concentration **(Figure 6**) and the maximum adsorption capacity obtained was 108,4 mg/g recorded at Co=1800 mg/L.

Adsorption kinetics

Pseudo-first order model

The integrated form of the pseudo-first order model (equation 2) with boundary conditions (t=0 to t=t and Qt=0 to Qt =Qt) was applied.

$$Ln(Q_e-Q_t) = lnQ_e - K_1t \quad (2)$$

where, Qe and Qt are the adsorption capacity at the equilibrium and time t respectively (mg/g), K is the rate constant of pseudo-first order equation (min^{-1}). This equation is verified if the plots of ln (Qe-Qt) as function of time give a straight line. K_1 is deduced from slope and Qt from the vertical intercept. The Figure 7 below describes the pseudo-first order model.

Pseudo-second order model

The linear form of the pseudo second order adsorption kinetic rate equation is expressed as:

$$t/Q_t = 1/K_2Qe^2 + t/Q_e \quad (3)$$

Where K_2 is the rate constant of the pseudo second order adsorption. The Figure 8 below describes the pseudo-second order model.

The correlation coefficient (R^2) of the pseudo second order model is closest to unity, implying a significant agreement of the experimental data to this model. Hence, the physisorption. At the same time, the R^2value(R= 0,934) of the pseudo-first-order cannot be neglected. It is sufficiently close to unity indicating that chemisorption and physisorption takes place concomitantly [21]. This conclusion is consistent to the results of isotherms and IR obtained above.

Adsorption isotherms studies

The linearized form of the Langmuir, Freundlich and Tempkin isotherm models are given by the equation 4, equation 5 and equation 6 respectively:

$$C_e/Q_e = 1/Q_{max}K + Ce/Q_{max} \quad (4)$$

$$lnQ_e = lnK_F + lnCe \quad (5)$$

$$Q_e = BlnK_T + BlnC_e \quad (6)$$

where Qe is the quantity adsorbed at equilibrium (mg/g), Q_{max} is the maximum adsorption capacity corresponding to complete monolayer coverage, K_L is the Langmuir constant related to the energy of adsorption (L/mg). K_F is the Freundlich constant, 1/n the heterogeneity factor which is related to the capacity of the adsorption. C_e the concentration at the equilibrium of the adsorbat (mg/g). B=RT/bT is the adsorption energy constant (J/mol)

The isotherm studies were carried out by varying the Cu^{2+} ions concentration from 600 to 1800 mg/Lat room temperature. The Figure 8 (a),(b) and (c)describes the Langmuir, Freundlich and Tempkin isotherms.

The isotherm characteristic helps to determine whether the adsorption is favorable or not. The low value of K_L and R^2 described that the Langmuir model is not suitable to explain the adsorption of Cu^{2+} onto ACZ. The Freundlich constants *n* ranging 1 and 10 determine that the adsorption is favorable or not [22]. On the Table 2 above, n=1,75 which is greater than 1. And it implies that the activated carbon surface is heterogeneous. The value of R^2 low than 0,9 restricts the use of this model to well describe the adsorption of the Cu^{2+} ions adsorption onto this sample. The value of Tempkin isotherm constant (B) constant is positive and R^2= 0,999 implies that the interactions between adsorbent-adsorbate is gravitational. The presence of –CO and –COO- on the ACZ surface explains well these interactions. [23,24]

Conclusion

Activated carbon impregnated with $ZnCl_2$ has been used as adsorbent in this work. The adsorption of Cu^{2+} onto ACZ depends on of the pH and the initial concentration. The pseudo- second order and first order models explained that the adsorption mechanism is both chemisorption and physisorption and takes place concomitantly. Tempkin isotherm described well the adsorption of Cu^{2+}ions; hence, we can conclude that the ACZ is good for the adsorption of Cu^{2}+.The major mechanisms responsible for the Cu^{2+} ions uptake in this range may be ion exchange and electronic attraction.

Acknowledgement

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TABLES & FIGURES Section

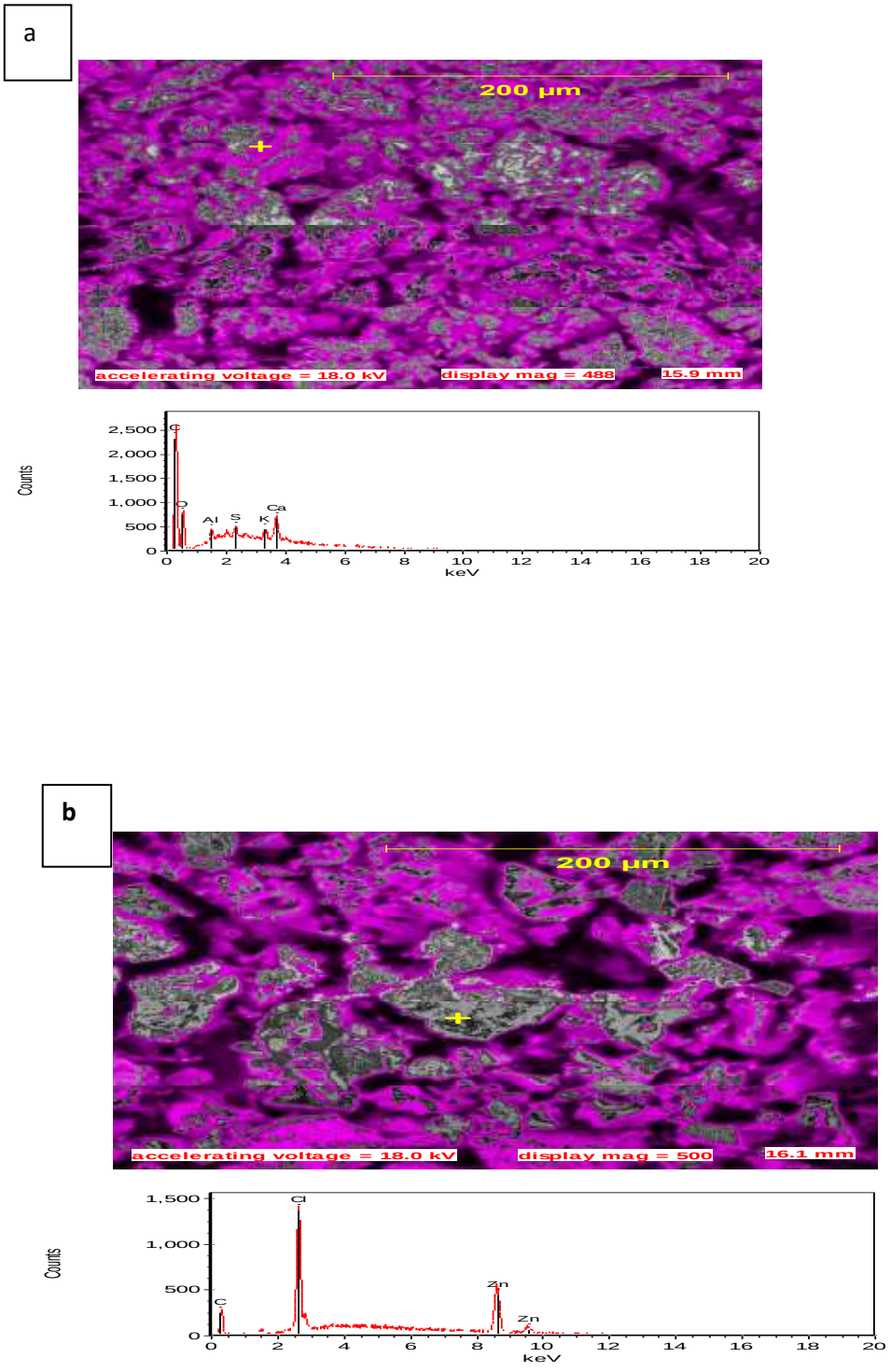
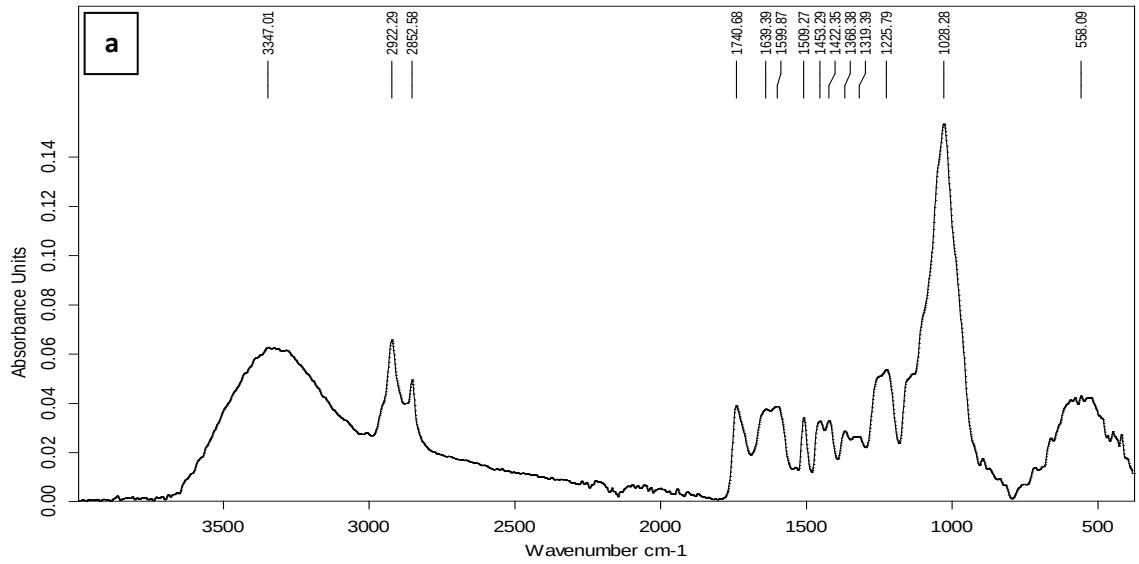


Figure 1: SEM of the biomass (a) and the ACZ (b)



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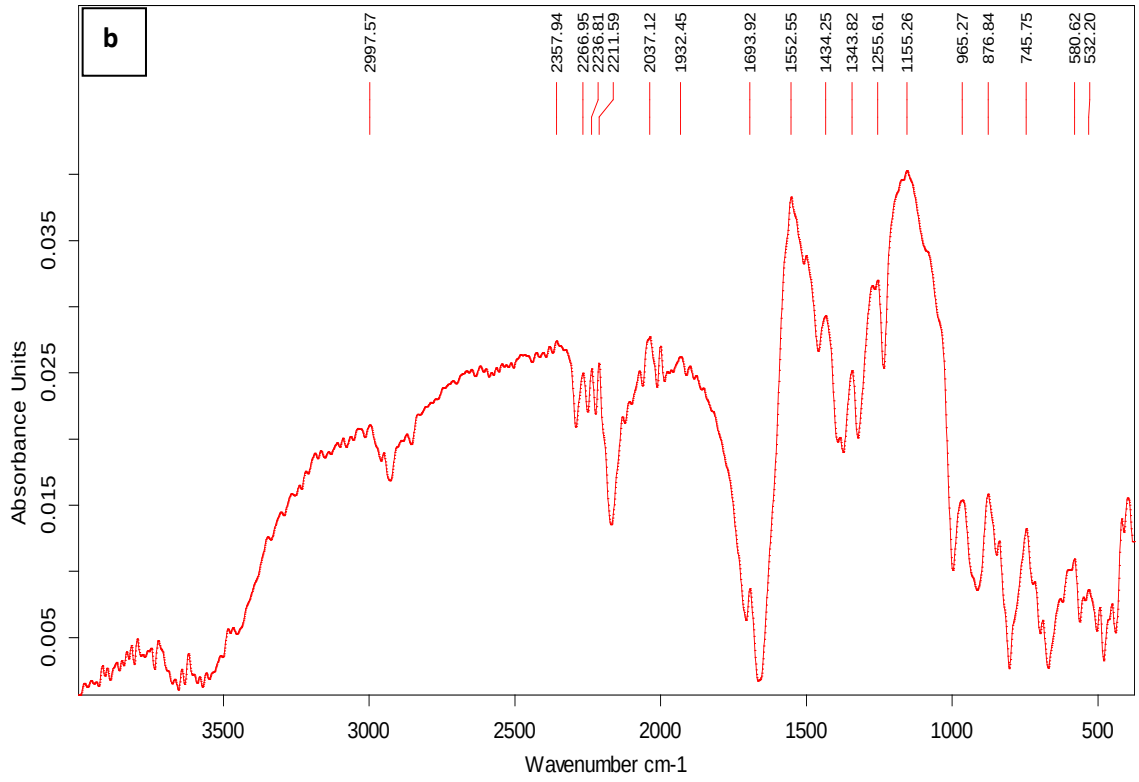

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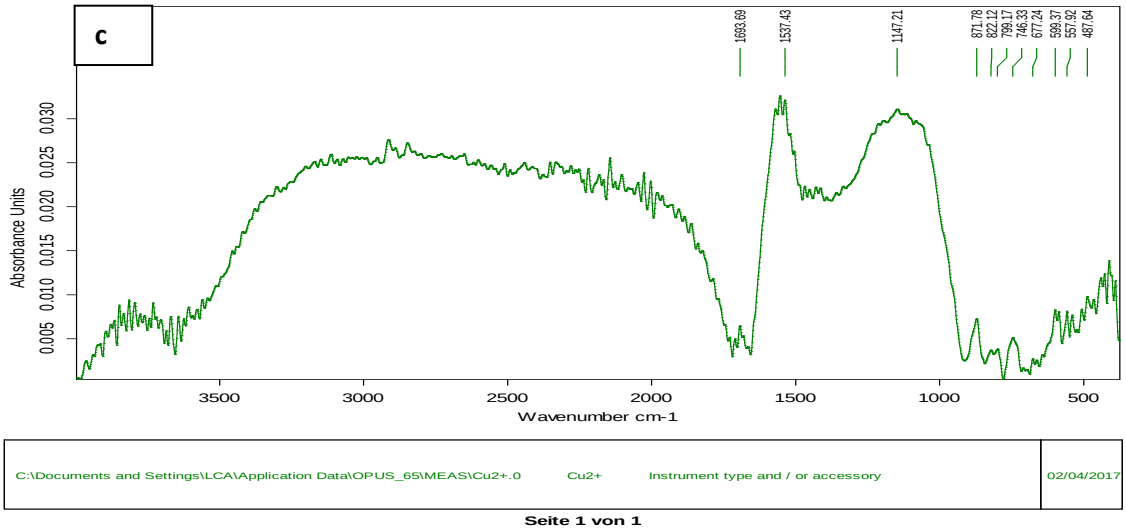


Figure 2: FTIR spectra of biomass (a), ACZ (b) and ACZ after adsorption (c) of Cu^{2+} ions

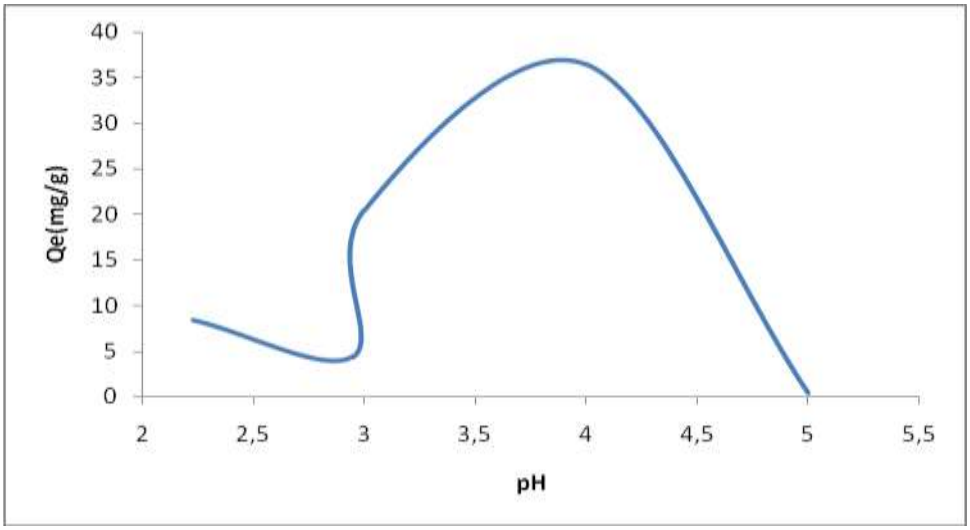


Figure 3: Quantity adsorbed as a function of pH

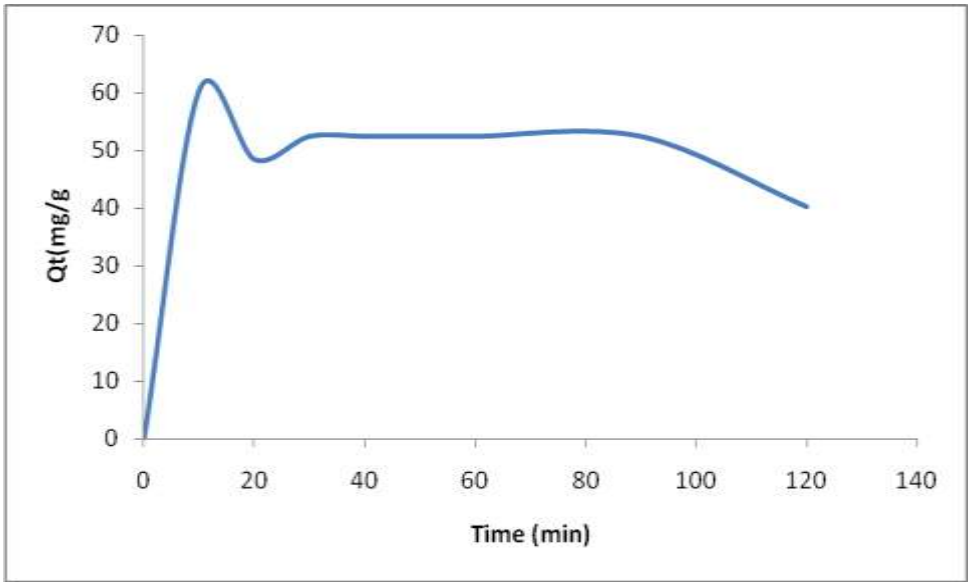


Figure4: Variation of contact time of the adsorption of Cu^{2+}ACZ

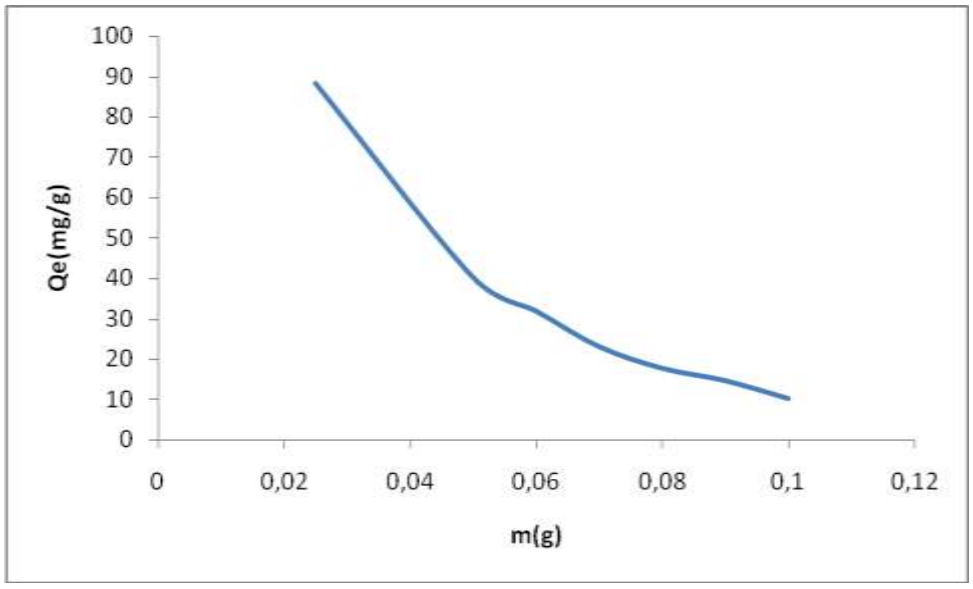


Figure 5: Effect of adsorbent dose

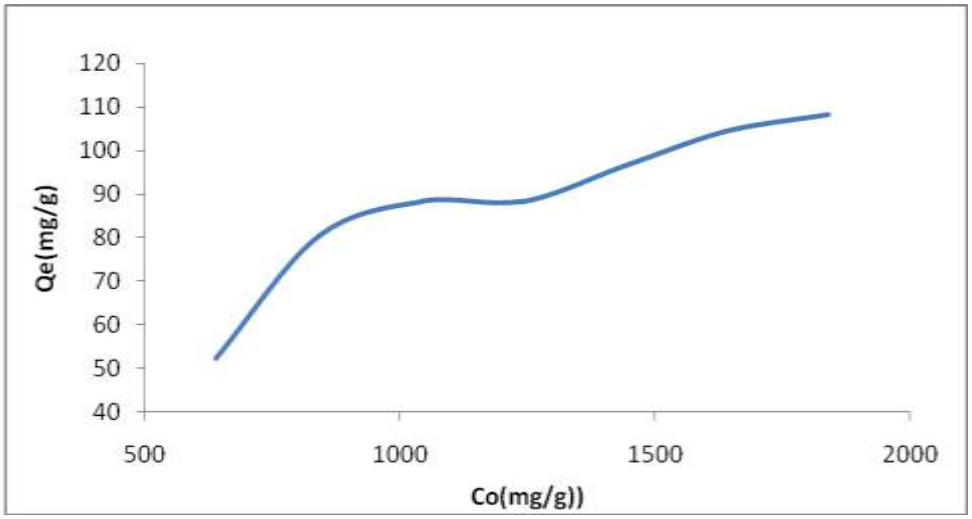


Figure6: Effect of initial concentration of Cu^{2+}ions

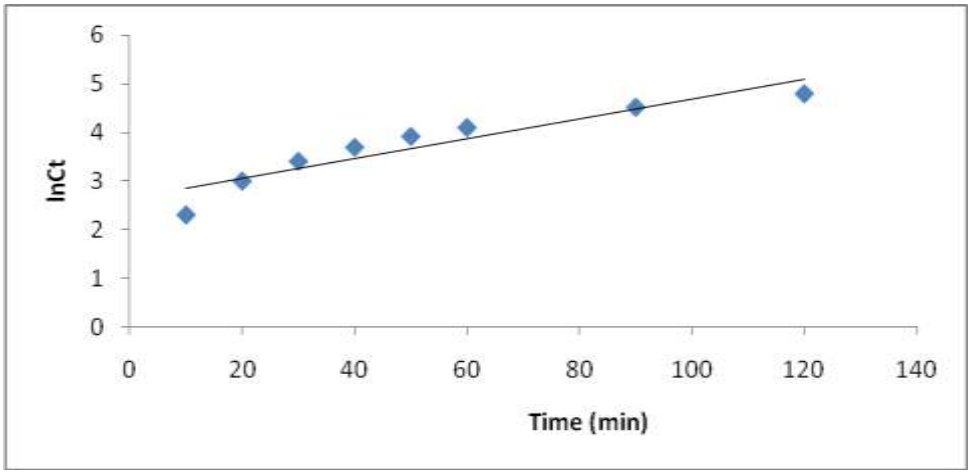


Figure 7:Pseudo-first order kinetics adsorption model of Cu^{2+}ions onto ACZ

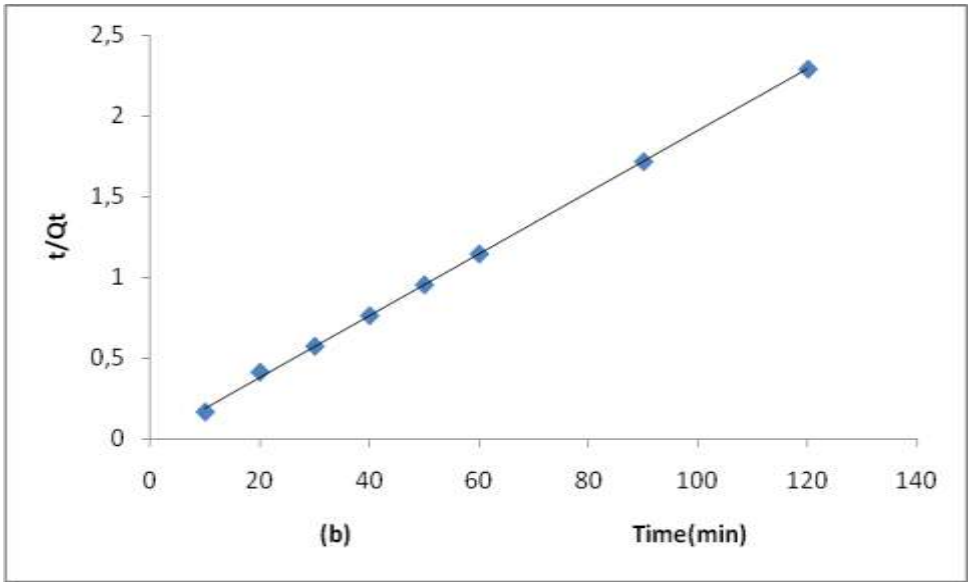
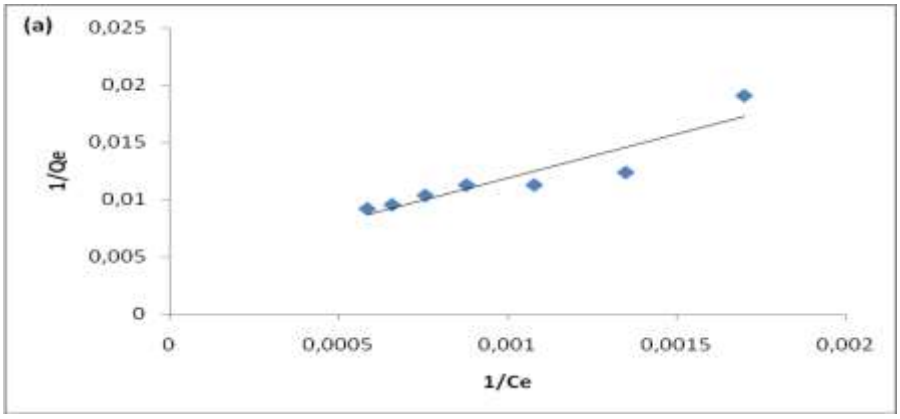
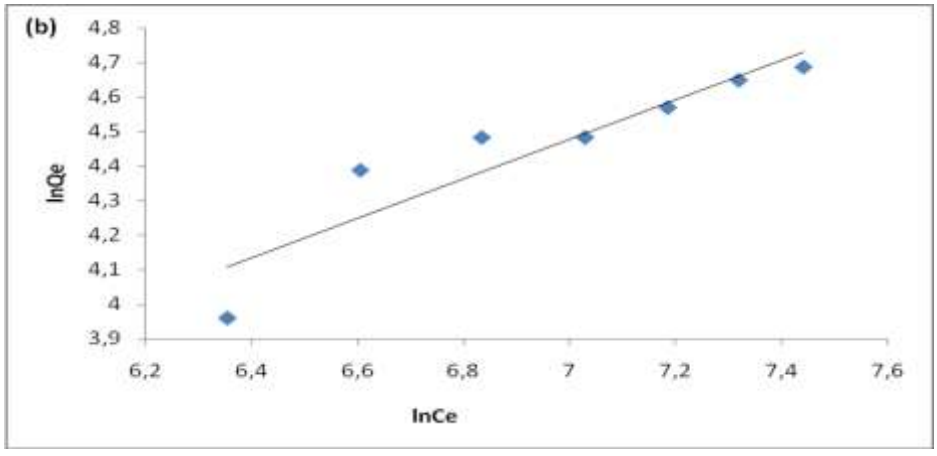


Figure 8: Pseudo-second order kinetics adsorption model of Cu^{2+}onto ACZ





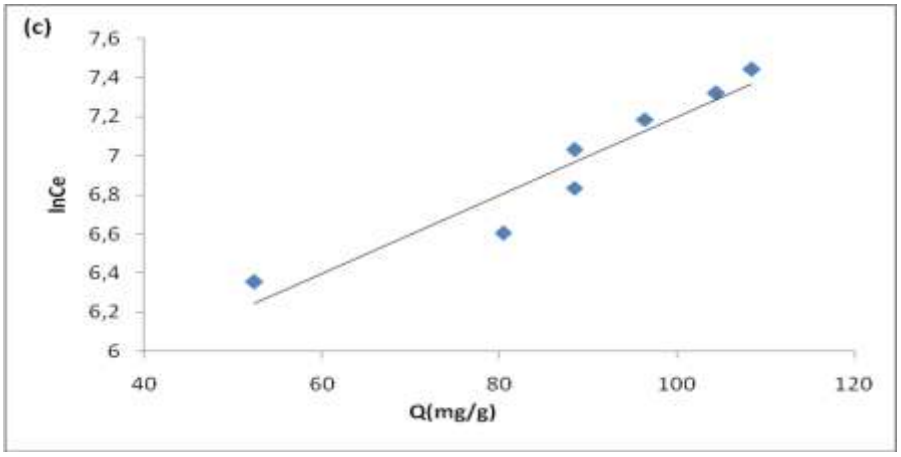


Figure 8:: Langmuir isotherm (a), Freundlich isotherm (b) and Tempkin isotherm (c) of Cu^{2+}onto ACZ

Table 1: **Kinetics constants for the adsorption of Cu^{2+} on ACZ**

Pseudo-first-order			**Pseudo- second order**		
$K_1(min^{-1})$	Q_e(mg/g)	R^2	K_2(g.min-1.m-1)	Q_e(mg/g)	R^2
0,020	13,970	0,934	52,63	-	0,999

Table 2: **Isotherm parameter for the adsorption of Cu^{2+} on ACZ**

Langmuir isotherm constant			Freundlich isotherm constant			Tempkin isotherm constant	
Q(mg/g)	$K_{Lx}10^{-4}$	R^2	K_F	1/n	R^2	B	R^2
250.000	0,523	0,850	2,282	0,572	0,851	0,020	0,909