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Evaluation of Sorption Capacity of Scrap Tyre in the Removal of Copper (II) Ion from Aqua System

N. A. Oladoja[a]*, **A. Ofomaja**[b] **and E. Ebare**[c]
[a]Department of Chemistry, Adekunle Ajasin University, Nigeria
[b]Department of Chemistry, University of Benin, Nigeria
[c]Department of Polymer Technology, Auchi Polytechnic, Nigeria

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Abstract. The use of scrap-tyre (ST), which was both a waste and pollutant was investigated as a low-cost sorbent to sorbed Cu (II) from aqueous solution. The influence of pH, sorbent dosage, contact time, and initial sorbate concentration on the uptake of Cu (II) by ST were studied. Optimum sorption of Cu (II) by ST was achieved at pH 6. The amount of sorbate sorbed per gram of sorbent decreased with increase in sorbent dosage. Maximum uptake of the Cu (II) was achieved within the first thirty minutes of contact between the ST and the Cu (II). The equilibrium relationship between the concentration of the Cu (II) in the fluid phase and the concentration in the ST particles at a given temperature showed that the sorption mechanism was like adsorption rather than distribution into any phase. Analysis of the results using Langmuir and Freundlich models showed that it conformed to Langmuir equation based on the formation of a monomolecular layer. The adsorption capacity due to monolayer coverage was 12.95 mg/g, while the energy of adsorption was 3.95 dm/mg.

Keywords: scrap tyre, Cu^{2+} ion, adsorption

Introduction

Copper is one of the metals whose density is greater than 5 g/cm^3 or 5 kg/m^3. Its presence, even at low concentration in aqueous medium portends dangers for all forms of the aquatic life or any living organism whose existence is knitted to the aquatic resource. Due to its toxic effects and non-biodegradability, it has emerged as a prominent pollutant. It accumulates in living tissues and may be transferred to human beings through the food chains. The presence of copper and its compounds in the environment could be traced to anthropogenic origin. Copper bearing mining wastes and acid mine drainage discharge significant quantities of dissolved copper in waste water (Ajmal *et al.*, 1998). Other sources of copper bearing wastes include swine waste (Wu *et al.*, 1999), plating baths, fertilizer industry, paints and pigments municipal and storm water run-off (Dean *et al.*, 1972; Tallmange,1965).

The conventional methods available for the attenuation of Cu and its like from solution include lime precipitation, ion exchange, and sorption on activated carbon (Dean *et al.*, 1972), membrane filtration processes and electrolytic methods (Braukman,1990). These methods have been found to be limited since they often involve high capital and operational cost. They may be associated with the generation of secondary waste which may present treatment problems e.g. those involving precipitation processes resulting in generation of large quantities of sludge (Williams and Edyvean, 1997).

The diverse problems emanating from the use of existing methods for the removal of copper from solution, active research has been undertaken on ways to improve upon the existing system or to patent new ones. Arrays of low cost sorbents have been studied and the activated carbon and coal have also been extensively used in metal ion adsorption from aqueous solutions (Macias-Garcia *et al.*,1995; 1993; Petrov *et al.*, 1992; Velenzuela-Calahoro *et al.*, 1990; Ferro-Garcia *et al.*, 1988). The use of biosorbents for metal ion was removed from aqueous effluents (Volesky and Holan, 1995). Some of the biosorbents have been investigated by including agricultural by-products, such as wood, rice-straws, coconut husk and peat-moss (Ho *et al.*, 1994).

An estimated 5 million scrap tyres (ST) existed in Nigeria in 1983 (Ebewele and Dzony, 1990). Out of these, each year about 700 to 850 thousands ST were added into the waste stream. Scrap tyers are recalcitrant to biodegradation and they remain in the environment for a long time to come and constitute a waste disposal problem. The applications of ST is used for the removal of Hg (II) ion from aqueous solution (Knocke and Hemphill, 1981), and for immobilization of Hg (II) ion in contaminated soils. The production of active carbons from tyre wastes has been used by Manchon-Vizuete *et al*. (2005) for the removal of Hg (II) ion from aqueous system.

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*Author for correspondence

In this study scrap tyre (ST) was used as a low-cost sorbent, for the treatment of water contaminated with Cu (II) ions. The choice of scrap tyre as a sorbent was predicted on the report that adsorbents containing sulphur or sulphur bearing groups have a high affinity for heavy metals but a low affinity for light metals (Bailey, *et al.,* 1999). This assertion was corroborated by the work that Hg, Cd and Pb show a high affinity towards sulphur. Notable sulphur bearing compounds investigated include thiols, dithiocarbamates, dithiophosphates and xanthates. Xanthates are most prominent because they are easy to prepare with relatively inexpensive reagents and are highly insoluble (Flynn *et al.,* 1980).

This study was aimed at investigating the potentialities of ST as a low cost sorbent for Cu (II) in aqueous medium. Contact time between sorbent and sorbate, solution pH, sorbent dose and sorbate concentration were optimized by the method of continuous variation following batch adsorption experiments.

Materials and Methods

Sorbent preparation. The sorbent of scrap tyre (ST) for the study was collected from a tyre dealer. It was dismembered. The rubber part was collected and washed thoroughly under running tap and then with distilled water. The washed rubber part was pulverized and sieved through 180 µ sieve and used as such. The physicochemical and textural characteristics of ST, as reported by Manchon-Vizuete *et al*. (2005) are presented in Table 1.

Sorbate preparation. Copper stock solution was prepared using $CuSO_4.7H_2O$ (BDH) salt in distilled water obtained by demineralizing water through a Barnstead demineraliser and then distilled in an all-glass apparatus. The different concentrations of copper solution needed for the experiments were prepared from the copper stock solution. The pH of the solution was adjusted using dilute H_2SO_4 and NaOH.

Batch adsorption studies. A typical batch adsorption experiment was conducted by contacting a known weight of the adsorbent with concentration of the adsorbate adjusted to the appropriate pH, in a thermostatic shaker/water bath agitated at a rate of 100 rpm for a known period of time. At the end of the study period, the adsorbent was removed by filtration and the equilibrium concentration was determined by atomic absorption spectrophotometer (AAS) (Buck Model 200A). Copper uptake by the adsorbent was calculated by the equation below:

$$q_t = \frac{V(C_o - C_f)}{m} \qquad (1)$$

Where:

q = the amount of adsorbed per gram (g) of adsorbent (mg/g).
C = the initial concentration of adsorbate.
m = the mass of adsorbent (mg).

The equilibrium time for maximum adsorption was determined at 30 min interval for 300 min. The effect of pH on the adsorption phenomenon was studied between pH 2 and 6. The effect of sorbent dose was studied between 0.05 g-0.35 g at 0.5 g intervals and sorbate concentration was studied between Cu (II) ion concentration of 98.85 and 125.77 mg/l. Each study was carried out in triplicate. The mean of the results was found and reported herein for brevity.

Results and Discussion

The physico-chemical and textural characteristics of the ST as reported by Manchon-Vizuete *et al.* (2005) show, the scrap tyre (ST) is a highly carbonaceous material. The values of the elemental composition (wt %) of ST reported, similar to the values determined and reported by Miguel *et al.* (2003) (C = 84.5%; H = 6.5%; N = 0.2%). The high carbon content of ST reported, to account for the choice of ST as a precursor for the production of active carbon. The low sulphur content was ascribed to the fact that sulphur was possibly not amenable to the method of analysis used (i.e. LECO micro analyzer made up of both VTF-900 and CHNS-932 determination units with suitable detector).

The textural characteristics reported show that ST is practically a non porous material ($V_T = 0.08$ cm^3/g). The extremely low porosity of the ST is made up of almost exclusively high pores i.e. mesopores and macropores. Owing, to the low value of W_o in the ST, the specific surface area ($S_{BET} = 0.0$ m^2/g) was very low.

The sorption capacities of the adsorbent (ST) under varying operational condition are presented in Fig.1-4. These results show the ability of ST to adsorb Cu (II) ion from aqueous solution. The propensity of sulphur bearing surfaces for metal ion could be ascribed to the ability of sulphur to coordinate metal ions. This is possible because sulphur is an anionic monodentate ligand. Irrespective of the forms in which it is present on a surface (e.g. thiocyanate, isothiocyanate, sulphate, and sulphite e.t.c) it is a susceptible site for binding metal ion.

Aside the curatives (i.e. sulphur), a number of ingredients are added to the base polymer (i.e. natural rubber), in tyre compounding, to enhance its service life. Other main ingredients added come under the following classification: filler, accelerator, activator and protective agents. Some of these ingredients also contain functional groups that are known to seques-

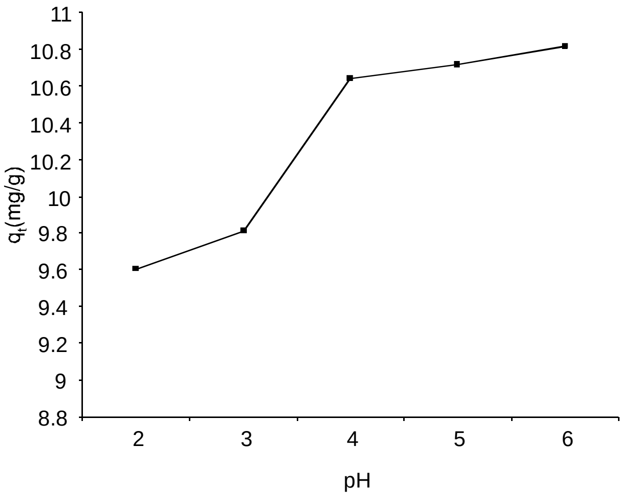

Fig. 1. Effect of pH on the uptake of Cu (II) ion by ST

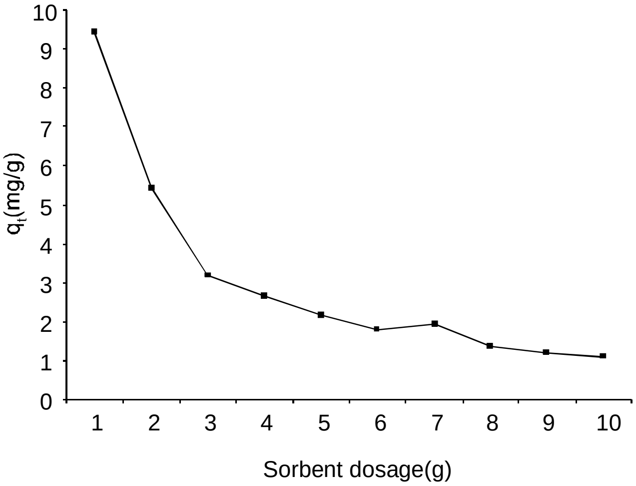

Fig. 2. Effect of sorbent dosage on the uptake of Cu (II) ion by ST

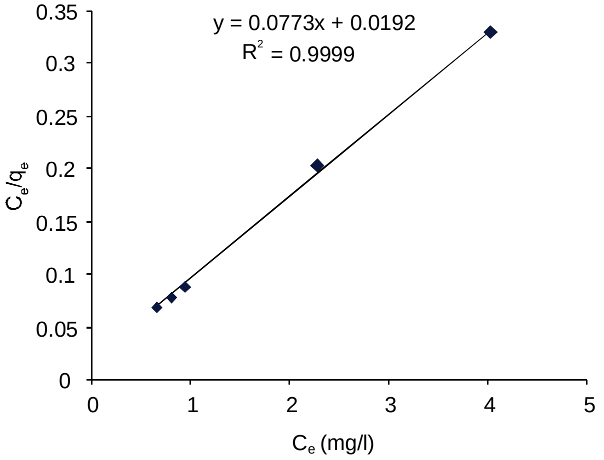

Fig. 3. Langmuir isotherm plot for the sorption of Cu (II) ion by ST

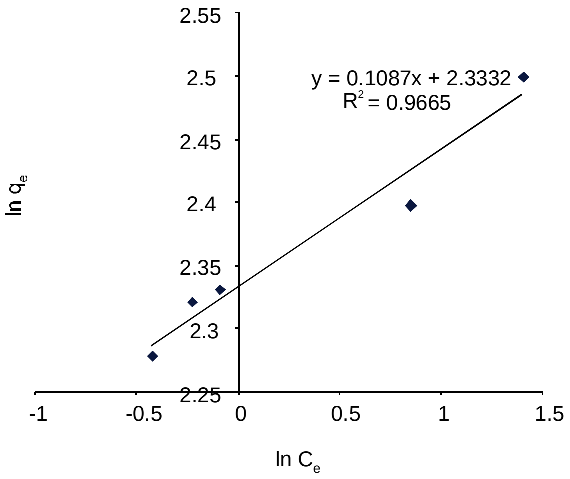

Fig. 4. Freundlich isotherm plot for the sorption of Cu (II) ion by ST

trate metal ions. Carbon black, the filler used in tyre compounding and has a large surface area and possesses surface activity that favours the attraction of metal ions. The chemical nature of the carbon black particle surface as elucidated by Blow and Hepburn (1982), show that carbon black consists of 90-99% elemental carbon. The principal functional groups present on the carbon surface are phenolic, ketonic and carboxylic together with lactones. It may also contain small amount of sulphur depending upon the nature of hydrocarbon used for the preparation. A large percentage of the accelerator and the protective agents used in tyre compounding contain amine, phenolic and sulphur functional groups. Amine and phenolic groups are also monodentate ligands that can form a coordinate compound with metal ions. Despite the presence of these ingredients in ST, the presence of sulphur could have enhanced their metal ion sorption capacity. Gomez-Serano *et al.* (1998) has proven that the presence of sulphur on activated carbon considerably enhances its heavy metal ion binding capacity.

Effect of pH on uptake of Cu (II) ion by ST. The results of the removal of the metal ion as a function of pH are presented in Fig.1. The study was carried out at the initial concentration of 108.42 mg/l of Cu (II) ion and at pH ranging between 2 to 6 and a sorbent dosage of 0.5 g/50 ml of sorbate. The pH of the solution was not allowed to exceed pH 7.0 as it has been reported by other workers that at pH > 6.5, Cu (II) ion precipitate out of solution as the metal hydroxides (Volesky and Holan, 1995). The adsorption of the Cu (II) ion on the scrap rubber increases with increase in pH of the solution. This study revealed that optimum uptake of Cu (II) ion by scrap rubber is achieved at pH 6, where an average of 10.81 mg/g of Cu (II) ion was sorbed by the sorbent. The adsorption of Cu (II) ion increased gradually between pH 2 and 3 and rapidly between pH 3 and 4 and finally became gradual again between pH 4 and 6.

The effect of pH on metal ion uptake by surfaces has been studied and the mechanisms have been explained by several authors. The heavy metal cations are completely released under circumstances of extreme acidic conditions thereby resulting in low sorption capacity of sorbent at low pH values. Seco *et al.* (1997) employed surface complex formation model (SCF) to account for the effect of pH on the adsorption of Cu (II) onto activated carbon. According to the SCF theory, the increase in the metal ion removal as the pH increases, can be explained on the basis of a decrease in competition between proton and metal species for the surface sites and by the decrease in positive surface charge, which results in a lower columbic repulsion of the sorbing metal.

Greenwood and Earnshaw (1984) also posit that the ionic mobility of H_3O^+ in water is abnormally high as compared to most other ions. Consequent upon the high mobility of H_3O^+ ion, they would get to the surface of the ST and get adsorbed faster than the Cu (II) ion.

Effect of sorbent dosage. The effect of changing the sorbent dosage(0.5g - 5g) in a fixed volume and fixed concentration of sorbate solution is shown in Fig.2. The amount of sorbate sorbed by the sorbent (i.e. mg/g of sorbent) reduced with increase in the sorbent dosage, while a reverse trend (i.e. an increase in value from 86.91% - 99.67% when the sorbent dosage was increased from 0.5 g-5.0 g) was observed in the extent(i.e. percentage) of adsorption. Increasing the dosage of the ST and keeping the Cu (II) ion concentration constant, makes a large number of sites available for a fixed concentration of sorbate, hence the reduction in the value of q_t. It can be observed from sorbent dosage of 1.5 g and above the amount of sorbate removed from solution is almost constant. Higher value of Cu (II) ion uptake was observed for sorbent dosage 0.5g and 1.0 g. Ho and McKay (2000), observed similar trend when they studied the uptake of Cu (II) on sphagnum moss peat. They observed that the Cu (II) uptake sorbed at any contact time was higher as the dosage of the peat moss decreased.

Effect of contact time. The results obtained (results not shown for brevity) from the effect of contact time on the uptake of Cu (II) ion on ST, at pH 6, sorbent dosage 0.5g and initial concentration of 108.42 mg/l, showed that the maximum uptake on the sorbent was attained within the first 30 min of this study. No visible change in the sorption capacity of the ST was observed, even when the contact time between the sorbent and the sorbate was increased to 300 min. Owing to the results obtained from this study equilibrium time was set at 60 min for all the experiments conducted thereafter.

Adsorption isotherm. Adsorption isotherm is the equilibrium relationship between the concentration in the fluid phase and the concentration in the adsorbent particle at a given temperature (McCabe *et al.*, 1993). The amount of Cu (II) sorbed increased with the increase in equilibrium concentration of Cu (II). The amount of Cu (II) uptake became almost constant when about 10.44 mg/g of Cu (II) was removed from solution. The curvature (the plot is not shown for brevity) indicates that the optimum capacity of the sorbent had been reached and the sorption mechanism was adsorption rather than distribution into any phase.

Langmuir and Freundlich model were used to analyze the adsorption behavior and to determine the Cu (II) ion adsorption capacity of the ST, the adsorption of Cu (II) at pH 6 on ST followed both isotherms. However, the Langmuir equation was better obeyed by the sorption process than the Freundlich isotherm as it was evident in the values of regression coefficient (R^2 = 0.999 and 0.9665 respectively. Table 2). The Langmuir equation was expressed as (Esma *et al.*, 1998; Diamadopoulos *et al.*, 1993; Panday *et al.*, 1985).

$$q_t = \frac{bQ^oC_e}{1+bC_e} \qquad (2)$$

Where, the Langmuir parameters, Q^o and, b, relating to monolayer adsorption capacity and energy of adsorption respectively. The values for the two parameters can be obtained from the slope and intercept of the plot of C_e/q_e Vs C_e.

The equation can be modified to give:

$$C_e/q_e = (1/Q^o)C_e + 1/Q^ob \qquad (3)$$

Premised on this relationship:

$Q^o = 1/\text{slope}$

while,

$b = 1/\text{intercept} \times \text{slope}$

The linearized Langmuir plot according to equation 3 is shown in Fig. 5. The Langmuir parameters were calculated by the aid of equation 4. The adsorption capacity due to monolayer coverage (Q^o) was 12.95 mg/g. The Langmuir parameter (b) related to energy of adsorption was 3.95 dm^3/mg.

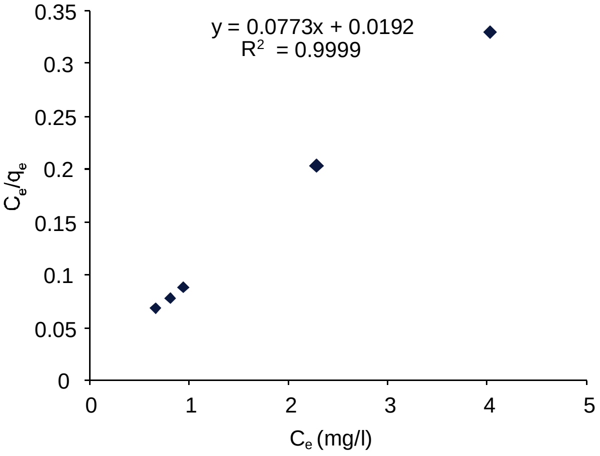

Fig. 5. Langmuir isotherm plot for the sorption of Cu (II) ion by ST

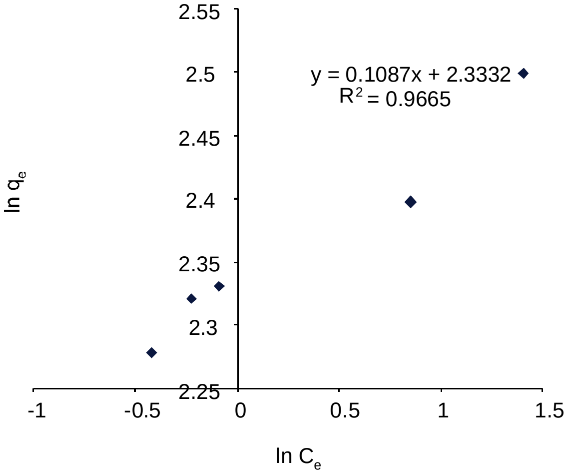

Fig. 6. Freundlich isotherm plot for the sorption of Cu (II) ion by ST

The Freundlich equation is expressed as (Ajmal *et al.*, 1998):

$A_m = K. C_e 1/n$ or $\ln A_m = \ln K_f + 1/n. \ln C_e$ (4)

Where:

A_m = the amount of sorbate adsorbed per specified amount of adsorbent mg/g.

C_e = the equilibrium concentration.

A plot of ln (A_m) Vs ln (C_e) should give a straight line with slope =1/n and intercept = ln K_f. The Freundlich plot according to equation (4) is presented in Fig. 6. The Freundlich constants were obtained from the linear equation and K_f was 10.28 mg/g, while: n was 9.20. The value of, n, obtained was greater than unity (1) which indicate good adsorption of Cu (II) ion onto ST

Table 1. Physicochemical (wt %) and *textural characteristics of ST (Manchon-Vizuete *et al.*, 2005)

Carbon	83.52
Hydrogen	7.28
Nitrogen	0.33
Sulphur	0.02
Oxygen	3.08
$S_{BET}(M^2/g)$	0.0
$V_o(cm^2/g)$	0.001
$V_{me}(cm^3/g)$	0.04
$V_{ma}(cm^3/g)$	0.04
$V_t(cm^3/g)$	0.08

* = S_{BET} (specific surface area); W_o = (micropore volume); = V_{me} (mesopore volume); V_{ma} = (macropore volume); = V_t (total pore volume)

Table 2. Sorption isotherm model constants and coefficient of determinations for sorption of Cu (II) ion by ST

Langmuir isotherm model		Freundlich isotherm model	
Q_o(mg/g)	12.95	K_f(mg/g)	10.28
b (dm^3/g)	3.95	n (mg/g)	9.20
R^2	0.999	R^2	0.967

Table 3. Sorption of the Langmuir constants of sorbent for Cu (II) ion by ST with other sorbents

Sorbent	Q_o(mg/g)	Source
Oil palm fibers	2.0	Low *et al.* (1993)
Aspergillus orysae	6.9	Huang *et al.* (1998)
Treated *A. nigar*	10.10	Raw *et al.* (1993)
Sago waste	12.42	Quek *et al.* (1998)
Scrap type	12.95	Present study
Sphagnum moss peat	16.40	Ho *et al.* (1994)
Anaerobic sludge	49.00	Gould and Geneteli (1978)

The favourable nature of adsorption can be expressed in terms of a dimensionless separation factor of equilibrium parameter, which is defined by:

$R_L = 1/1+bC_o$

Where, 'b' is the Langmuir constant and Co, is the initial concentration of the adsorbate in solution (McKay *et al.*, 1982). All the RL values for Cu (II) ion sorption were less than unity (they range between 2.0×10^{-3} and 2.5×10^{-3}). This indicates favourable adsorption.

A comparison of Langmuir adsorption capacity of ST for Cu (II) with some sorbents earlier investigated by other workers (Table 3) showed that ST has greater capacity for copper than many of these materials.

Conclusion

i) The uptake of Cu (II) ion by ST increased with pH. Optimum uptake of Cu (II) was achieved at pH 6.

ii) The amount of sorbate sorbed by the scrap tyre (i.e. mg/g of sorbent) reduced with increase in the dose of the ST. The highest value of Cu (II) uptake was achieved at sorbent dosage of 0.5 g.

iii) Maximum uptake of Cu (II) was achieved within the first 30 min of contact between the sorbent and the sorbate.

iv) The results obtained from the adsorption isotherm showed that the sorption mechanism is like adsorption rather than distribution into any phase.

v) The adsorption isotherm model of Cu (II) from aqueous solution onto scrap tyre conforms to a Langmuir equation based on the formation of a monomolecular layer.

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