



# **3<sup>RD</sup> BIENNIAL ENGINEERING CONFERENCE**

**SCHOOL OF ENGINEERING AND ENGINEERING TECHNOLOGY  
FEDERAL UNIVERSITY OF TECHNOLOGY, MINNA.**

## **BIENNIAL ENGINEERING CONFERENCE BOOK OF PROCEEDINGS**

**T H E M E :  
CHALLENGES IN ENERGY SUPPLY AND INFRASTRUCTURAL  
DEVELOPMENT IN DEVELOPING COUNTRIES**



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## Adsorption of Copper (II) Ions from Waste Polluted Effluents using Calcium-Alginate Modified Shea Butter Husk

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**Abstract:** Shea Butter Husks entrapped in calcium-alginate was effectively used to adsorb Cu (II) ions from an aqueous solution. Instrumental analysis such as FT-IR, XRD and SEM were used to characterise the sorbent. The functional groups responsible for metallic biosorption were majorly -OH, and -COOH. Equilibrium sorption experiment on the time variant of Cu (II) uptake at different concentrations was studied. Percentage removal of Cu increased with an increase in initial pH of 2 and optimum pH of 5 was found to be best before precipitation of the hydroxides. The results obtained for  $\Delta G^\circ$ ,  $\Delta H^\circ$  and  $\Delta S^\circ$  revealed that the adsorption was spontaneous, endothermic and occurs in an orderly manner. Isotherm models like the Langmuir, Freundlich, and Tempkin, were investigated and all were in good agreement with the experimental data. The Equilibrium data best fits the Langmuir isotherm with the correlation coefficient at unity. The results indicated that calcium alginate modified shea butter husks can serve as an adsorbent for the uptake of copper (II) ions from polluted waste streams.

**Keywords:** Shea butter husk, Calcium alginate, Copper (II) ions, Equilibrium Study

### 1. Introduction

Heavy metals removal from wastewater is a subject of great interest in a developing Country like Nigeria. Their increase has been caused by growth in the small and medium scale enterprises. Copper is one of the heavy metals whose threshold limit has been exceeded through the activities of craftsmen and iron smelters. The recommended in drinking water by World Health Organization (WHO) is 1.5 mg/l. The toxic effect through its non-degradability, bio-accumulation in the tissues poses a serious health hazard. Excessive intake of large doses of Cu.(II) (>1.0 mg/l in drinking water) by human may lead to central nervous system irritation, possible necrotic changes in the liver, and kidney (Zhang *et al.*, 2011).

Conventional methods for heavy metal removal are precipitation, coagulation, reduction, ion exchange, evaporation, and membrane processes. These methods have several disadvantages such as less effective removal of metal ion, high reagent

requirements, high process costs, the generation of toxic sludge, and the problem of the safe disposal of the material. The Adsorption process has distinct advantages over conventional methods due to the availability of the waste matter, selectivity nature of the adsorbent, efficiency, ease of operation, and cost effective.

Shea Butter husk is a natural agricultural waste obtained from the Shea butter tree after de-pulping of the shea fruits. It is found in large concentration of approximately 350,000 metric tonnes per annum in Niger state, of Nigeria (Suleiman, 2004). Zanu *et al.*, (2012) in his article on using the sheanut cake as a feed for cockerals reported that the chemical analysis as carried out by Atuahene *et al.*, (1997) were as follows: crude protein 162.4 g kg-1 DM, fat 162.4 g kg-1 DM, fibre 134.0 g kg-1 DM, ash 95.0 g kg-1 DM, nitrogen free extract 42.0 g kg-1 DM and metabolizable energy 7.12 MJ kg-1 DM. Studies have shown that shea butter husks can be used in the removal of heavy metals Eromosele

*et al.*, (1996) for the removal of chromium and copper; Itodo *et al.*, (2010) for the removal of atrazine from polluted effluents. In the developed Countries, pilot plants have been constructed for the applicability of biosorption as a basis for metal sequestering processes. In order to stabilize and enhance the mechanical properties, immobilization is sometimes used (Onate, 2009). Immobilization helps to create a material with the right size, rigidity and porosity necessary for the adsorption operation. However, there is limited or no information on immobilization of the shea butter husk. Grape stalk powder immobilized in calcium alginate has shown high efficiency in Cr (VI) removal (Fiol *et al.*, 2003). Yahya *et al.*, (2012) also immobilized the shea butter cake for the removal of lead. This study therefore seeks to use the shea butter husks by immobilizing it in calcium alginate for the removal of Cu(II) in polluted effluent.

## 2. Methodology

### 2.1 Adsorbent preparation

The Shea fruits gathered from Kataregi farmland in Minna, Nigeria were subjected to the following processes: De-pulping that is the removal of the fleshy mesocarp is followed by drying to facilitate the removal of hard shell or coat covering endoderm containing the oil. (de-husking). The husk is washed and dried in oven at 80 °C until a constant weight was attained (Runping *et al.*, 2008). This is further crushed with a blender (Moulinex) and then sieved with an electric sieve shaker (Octagon 2000, Endecotts England) to obtain a smooth powder of particle size <250µm.

The modified shea butter husk (MSBH) was prepared by dissolving 1 g of sodium alginate into 100 ml of distilled water at a temperature of 65 °C on a constant temperature magnetic stirrer (Fiol *et*

*al.*, 2006). After complete dissolution of the gel and cooling to a temperature of 25 °C, 2 g of shea butter husk was added. The stirrer was set at a speed of 150 rpm to maintain an intense agitation in order to produce an homogeneous mixture of alginate and the powder. Afterward, the solution was added drop wise via a syringe to a solution of 0.1M calcium chloride dihydrate (CaCl<sub>2</sub>.2H<sub>2</sub>O) under low agitation (20rpm). The entrapped shea butter husks in the calcium alginate (CA) beads were cured in the CaCl<sub>2</sub> solution for 24 hr. The hard spherical beads now called modified shea butter husks (MSBH) were filtered and rinsed several times with distilled water to remove excess Ca<sup>2+</sup> ions. The obtained beads were covered with abundant distilled water and stored in the fridge at around 4 °C for further use.

### 2.2 Preparation of metal solutions

Stock solution (1000 mg/L) of copper (II) was prepared by dissolving 2.6847 g of analytical grade copper chloride dihydrate (CuCl<sub>2</sub>.2H<sub>2</sub>O) in 1000 ml of distilled water in a volumetric flask.

### 2.3 Characterisation of adsorbent

Shea butter husk was characterized using instrumental analysis. The functional groups of the raw sorbents and Cu- loaded sorbent were characterized using the Fourier Transform Infrared Spectrophotometer (FT-IR-8400S, Shimadzu Japan). The spectral range varied from 4000cm<sup>-1</sup> to 400cm<sup>-1</sup>. The X-ray diffraction (XRD) analysis was carried out to determine crystallinity of the husk.

### 2.4 Batch Adsorption process

The measurement of metal ions uptake onto shea butter husks was determined by batch adsorption technique using a Multipurpose flask shaker (12F Techmel & Techmel, U.S.A) at a speed of 300



rpm and  $303 \pm 1K$  (Akhtar *et al.*, 2010). For this purpose, 40 beads of the MSBH were added to 50 ml of Cu (II) solution of various concentrations (10, 50, 100 & 250)  $mgL^{-1}$  in 250 ml Erlenmeyer flasks. Experiments were carried out for a contact time of 240 minute to ensure that the system reaches equilibrium (Srivastava *et al.*, 2006). After agitation, gel beads were separated from the metal solution by cellulose filter paper and metal concentration in the liquid phase was analysed using Flame Atomic Absorption /Emission Spectrophotometer Varian Spectra A A 220FS. The metal concentration in the solid,  $q_e$  ( $mg.bead^{-1}$ ) was calculated from the difference between the initial concentration,  $C_i$  and equilibrium  $C_e$  metal concentration in solution ( $mg.L^{-1}$ ). The uptake of the sorbent was calculated according to equation:

$$q_e = (C_i - C_e) \frac{V}{N} \quad (1)$$

Where  $V$  is the solution volume in liter and  $N$  is the number of beads.

To compute the sorption percentage, % $R$

$$\%R = \frac{C_i - C_e}{C_i} \times 100 \quad (2)$$

### 2.5 Initial pH effect on Cu sorption

The effect of pH on adsorption capacity of MSBH biosorbent was studied by adding 40 beads of MSBH in 50 mL solution of 250 mg/L of Cu (II) solution. 0.1MHCl and 0.1M NaOH were used to obtain desired pH. These were contained in a series of conical flasks in the range from pH1 to pH10 and were agitated for 2 h; 200 rpm and at 300 K (Gilbert *et al.*, 2011).

### 2.6 Effect of Temperature

The effect of temperature on the adsorption of Cu (II) unto shea butter husks was studied in a batch adsorption process at 313 K, 323 K, and 333 K, at

pH value of 5 and dosage level of 40 beads. The equilibrium contact time for adsorption was 120 minutes.

## 3. Results and Discussion of Results

### 3.1 Characterisation of the adsorbent

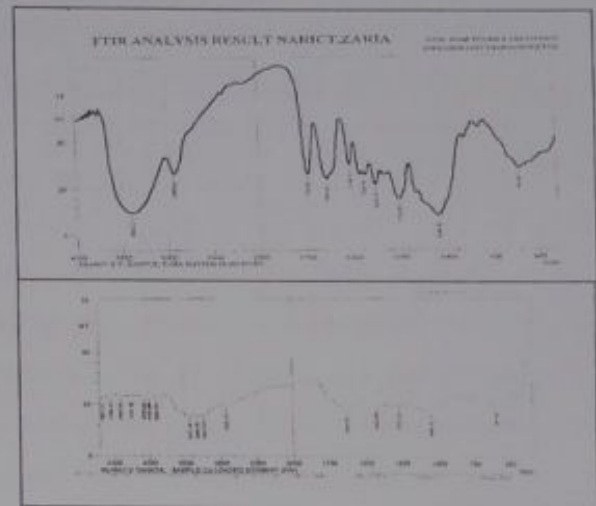


Fig. 1 FTIR spectra of shea butter husk and Cu-loaded modified shea butter husk

Fig. 1 shows the IR-spectrum of raw shea husk and the Cu- laden modified shea butter husk. Wave number at  $3392.90\text{ cm}^{-1}$  on the raw husk indicates the presence of simple-hydroxyl compound and the emergence of several other broad bands at  $3237.63$ ,  $3336.00$ , and  $3431.48$  may be attributed to binding of  $-OH$  group with Cu and the polymeric structure of the modified husk (Coates, 2000). The broad peaks mostly on the Cu-loaded husk indicate adsorption at  $1635.69\text{ cm}^{-1}$  which corresponds approximately to an amide or carboxylate.

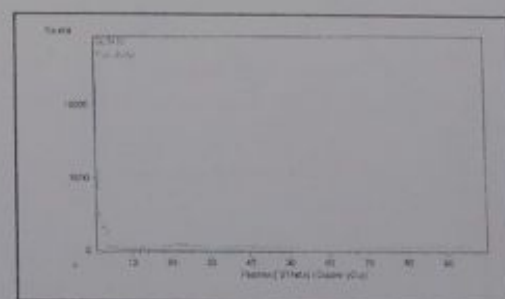


Fig. 2: XRD patterns of shea butter husk

Fig. 2 shows the XRD patterns of the shea butter husk. The non-appearance of any major peaks indicated that the sorbent is amorphous in nature. However a very weak peak was observed at  $22^\circ 2\theta$

diffraction angle and was attributed to cristobalite. (Kumagai *et al.*, 2010).

### 3.2 Effect of pH

pH play a very important role in the extraction of Cu(II) ions from the solution (Rocha *et al.*, 2009). This is noticeable as the initial solution pH was increased from 2 to 3, there was a sharp increase in the amount of Cu (II) adsorbed on the husk from 12.034 mg/g to 24.234 mg/g, and more Cu (II) was removed with increase in pH until it reaches pH of 5. A slight decrease in the capacity was observed as the pH changed from 5 to 6 This can be explained by the fact that at lower pH, H<sup>+</sup> ions formed from the dissociation of the ions in the solution compete with the Cu(II) for the sorptive sites on the adsorbent. When the pH is increased, the concentration of the H<sup>+</sup> decreases, and more Cu(II) ions react with the modified husk, with further increase in the pH the Cu(II) ions existing in the solution would change to Cu(OH)<sub>2</sub> which gradually causes precipitation (Zhang *et al.*, 2011). Authors that have found pH of Cu at 5 include (Benaissa and Eloudchi 2011, Villaesusa *et al.*, 2004).

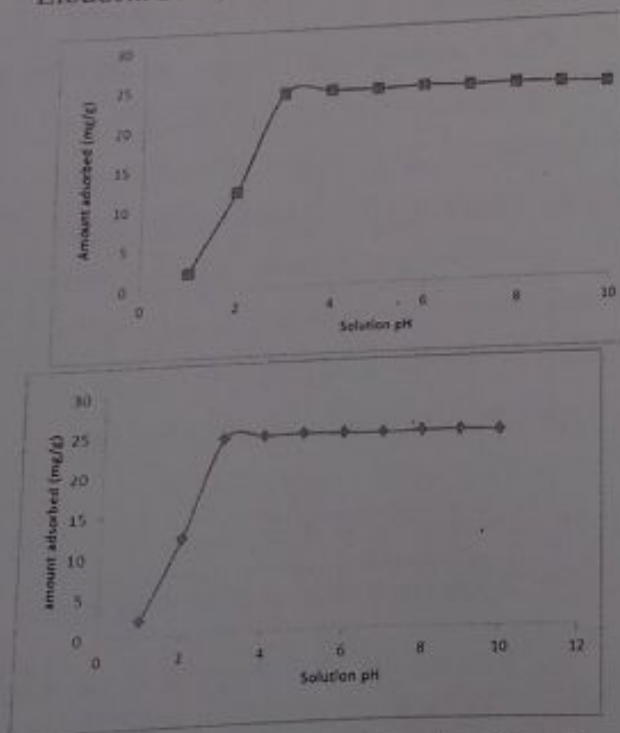


Fig. 3: Effect of pH on 250 mg/l of Cu (II) adsorption on modified husk

### 3.3 Effect of Time and initial metal ion concentration

The adsorption of Cu (II) on MSBH increases with increase in time until at saturation where it remains constant. Maximum adsorption was attained within the first 30 minutes for lower concentration while at higher concentration 120 minutes was the maximum equilibrium time. This was in accordance with previous study of Gilbert *et al.*, 2010; Khorambadi *et al.*, 2011; Rocha *et al.*, 2009 As shown in fig. 4, as the initial concentration was varied from 10 mg/l to 100 mg/l, the amount adsorbed per unit mass of the husks increases from 0.2476 mg/bead which is equivalent to 229.259 mg/g of dry mass (99.1% removal) to 0.9226 mg/bead equivalent to 854.26 mg/g (32.9% removal). This is as a result of the increase in the driving force of the concentration gradient, (Santhi *et al.*, 2010). The rate of percent metal removal is higher at the beginning firstly because of the affinity the sorbates has for the sorbent and also due to the large surface area of the MSBH available for the adsorption of the metals.

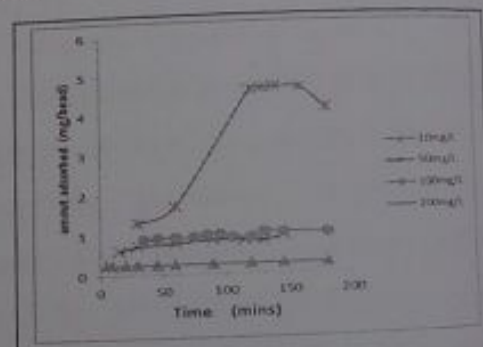


Fig. 4: The amount adsorbed on the Time variant of Cu(II) adsorption at various initial concentration

### 3.4 Evaluation of Thermodynamic Parameter

At 313 K the percentage removal of Cu on MSBH was 93.6% while at 333 K it was 94.7% Afterward further, increment in temperature was significant in the percentage removal of Cu