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ADSORPTIVE REMOVAL OF LEAD FROM AQUEOUS SOLUTION USING RAW AND MODIFIED SHEA BUTTER CAKE

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ABSTRACT

The adsorption of lead (Pb) from aqueous solution using Raw Shea Butter Cake (RSBC) and Modified Shea Butter Cake (MSBC) was studied. Fourier Transform-Infrared Spectrophotometer (FT-IR) and Scanning Electron Microscope (SEM) were used to characterize the adsorbent. The chemical functional groups responsible for metallic bio-sorption were the -OH, -COOH and -NH₂. Batch adsorption process was used to study the effect of various parameters ranging from the effects of initial metal ion concentration (10-400 mg/L), pH (2-10), and equilibrium time (0-240 minutes). Optimum conditions of initial metal ion concentration = 250 mg/l, pH = 6 and equilibrium time = 5 min for MSBC and 30mins for RSBC were obtained. Adsorption isotherms, namely Langmuir, Freundlich, Temkin, and Dubunin- Radushkevich (D-R) were found to fit the experimental data adequately well. The Langmuir Isotherm gave the best fit with the highest correlation coefficient of 0.9996 for MSBC and maximum sorption capacity of 13.175 mg/bead, while the corresponding values of 0.9998 and 9.328 mg/g were observed for RSBC. The results obtained showed that both the RSBC and MSBC have the potential to serve as adsorbent for the removal of Pb(II) ions in aqueous solutions.

1.0 INTRODUCTION

Nigeria is blessed with agricultural products which generate large tonnes of wastes during food processing and are sometimes toxic. Shea butter cake is one such waste obtained from shea butter processing. The cake has been found to contain tannin, oxalate, phytate and saponin, which have anti nutritional values and thus render it undesirable as feed for livestock^{1,2,3}. The cake has also been reported to have negative impact when disposed of indiscriminately as it has been found to be non-biodegradable and harmful to aquatic animals^{4,5}. Analysis of a whole shea butter fruit shows that about 18% of its total mass is butter and about 12% water, the remaining 70% accounts for the residue and husks⁶. Studies have shown that it has high contents of lignin, cellulose and phenols, which are good binding agents for metal adsorption⁷. The use of bio-sorbents from lignocellulosic agricultural waste has received much attention in the sorption of heavy metals⁸. Due to the presence of these compounds and possibility of their leaching into the aqueous solution, pre-treatment of the lignocellulosic agricultural waste is necessary. Several authors have used different compounds and technique for pre-treatment purposes. The use of polymeric compounds such as polyacrylamide, ethylenediamine, alginate, methanol and oxidizing agents has been investigated.

This study seeks to use the shea butter cake in its raw

form and through entrapment with calcium alginate to treat a lead contaminated effluent. Lead metal was focused upon because of the current risks it poses to some communities that engage in mining the heavy metal. An unprecedented number of children are reported to have died in Zamfara State, Nigeria, due to lead poisoning⁹. This problem is so widespread because the grinding of leaded rocks, in which gold is found, is done inside village compounds. These rocks are collected and crushed by hand, and much of the grinding is done by women inside homes where there are children. Young children are particularly vulnerable for reasons that include their hand-to-mouth activity, which makes them susceptible to nervous systems damage⁹. Exposure to lead can reduce IQ and cause behavioural problems. Acute exposure can result in blindness, certain types of paralysis, seizures, encephalopathy, and death⁹.

The shea butter cake is a waste material, which is low in cost, readily available and efficient in removal of heavy metals¹¹. There is a double benefit to communities producing it: as source of revenue from sales of its butter and also could serve as an alternative material in the treatment of lead contaminated effluents arising from mining operation before they are discarded into the environment and thus affects man and aquatic habitats.

2.0 MATERIALS AND METHODS

2.1 Shea Butter Cake Preparation

Fresh shea butter fruits were collected from Garatu ward in Minna, Niger State, Nigeria between June-August. The collected fruits were sorted and thoroughly washed with tap water to remove soil and dusts, and then further washed with distilled water. This is then par-boiled to enhance de-pulping. Effect of temperature has been found to be negligible as the oil trapped in the seed becomes extracted after moisture content has been completely removed. The fruits were de-pulped and the kernels dried between 40° and 50°C for a reduction of moisture content to less than 30 % of the initial volume¹⁰. The dried kernels were de-husked manually using a metallic rod. The nuts were further dried for another 5 days for complete removal of moisture at the same temperature and milled into smoothness using machine blender (model, Moulinex) and sieved using an electrical sieve shaker (Endecotts England BS 410/1986) to obtain a powder (<250µm particle size). Oil was extracted with n-hexane solvent for 5 hours using Soxhlet apparatus at 60°C. The raffinate obtained after extraction is the Shea Butter Cake which is the material of interest. This is air-dried until a constant weight is obtained and stored in a desiccator for further use.

The modified shea butter cake was prepared by dissolving 1g of sodium alginate into 100ml of distilled water at a temperature of 65°C on a constant temperature magnetic stirrer (Labtech79-1)¹¹. The resulting solution (jelly-like) was allowed to completely dissolve and cool down at room temperature before the addition of 0.5g of shea butter cake. An intense agitation was maintained in order to produce homogeneous mixture of alginate and the powder. The solution was added in drop wise manner via a 20 mL syringe to a solution of 0.1 mol/dm³ calcium chloride dihydrate (CaCl₂·2H₂O) with slow agitation. The entrapped shea butter cake in the calcium alginate (CA) beads was cured in the CaCl₂ solution for 24hr¹¹. Irregular shapes and sizes were removed and, hard spherical beads of approximately 2mm containing 2% (w/v) of shea butter cake now called modified shea butter cake (MSBC) were filtered and rinsed several times with distilled water to remove excess Ca²⁺ ions. The obtained beads were covered with abundant distilled water and stored in a refrigerator at around 4°C for further use.

2.2 Preparation of Metal Solutions

All solutions were prepared using deionized water. Reagents and chemicals used were of analytical grade. Stock solution (1000mg/L) of lead was prepared by dissolving 1.5980g of lead nitrate [Pb(NO₃)₂] in 100ml

of deionized water and dilute to 1 litre in a volumetric flask with deionized water. Metal ion concentration in the range of 10-400mg/l was prepared by serial dilution for use in the experiment.

2.3 Characterisation of Adsorbent

The raw shea butter cake was characterized using the Fourier Transform-Infrared Spectrophotometer (FT-IR-8400S, Shimadzu Japan) in order to determine the chemical functional groups present in the sorbent before and after adsorption of Pb (II). The sample was prepared using KBr discs and the spectral range was varied from 4000cm⁻¹ to 400cm⁻¹. The surface morphology of the adsorbents to determine the porosity was conducted at different magnification with High Resolution Scanning Electron Microscope (EVO MA 10 Carl Zeiss).

2.4 Batch Adsorption Studies

For each experimental run, 50ml aqueous solution of a known concentration was measured in a 250ml conical flask containing either 0.5g RSBC which is equivalent to 40 pieces of the prepared MSBC beads. These were agitated using a Multi-purpose Flask Shaker (TT 12F Techmel&Techmel, U.S.A) at a speed of 200rpm and 300 ±1K. In the case of the RSBC, after agitation, samples were centrifuged at a speed of 5000rpm for 5minutes. The supernatant was filtered using Whatman filter paper (110cm) and analysed for equilibrium concentration of the metal ion (C_e). The MSBC was also filtered after agitation and C_e determined. Effect of contact time from the start of agitation up to 5h was studied on Pb(II) removal. Samples were withdrawn at different time interval and C_e was determined using Atomic Absorption Spectrophotometer (AAS 240FS) until equilibrium was attained (that is when there is no significant difference between the initial and proceeding readings). The effect of initial pH was studied using LabTech pH meter over range of pH = 2-10 by adjusting with either 0.1M HCl or 0.1M NaOH solutions as the need arises. The effect of initial metal ion concentration was studied from 5mg/l to as high as 400 mg/l. Quantification of equilibrium concentration (C_e) of sorbates in metal supernatant solutions was carried out using AAS (model 240FS). The rate of removal of the metal ions from the solutions (R) is calculated from the difference between the known concentrations and the analyzed concentrations from the AAS using Equation (1), while the equilibrium adsorption uptake in the solid phase, (q_e) is calculated using Equation(2)¹²;

$$\%R = \frac{C_i - C_e}{C_i} \times 100 \quad (1)$$

$$q_e = (C_i - C_e) \frac{V}{N} \quad (2)$$

where C_i is the initial metal ion concentration (mg/l), C_e is the equilibrium metal ion concentration (mg/l), V (dm³) is the solution volume and N is the amount of beads or the weight of adsorbent in g for (RSBC).

3.0 Results and Discussion

3.1 Characterization of the Adsorbent

The IR spectra indicate that the RSBC and MSBC possess different surface structure (Fig.1). There are some absorption above 3000/cm which suggests that it is unsaturated (contains C=C). Additional moderate band in the range 1200-1000 and 800-600 implies simple hydroxyl compound. The shifting of the band of the O-H (Phenol) at 1374.33/cm for RSBC to 1430.26 of the loaded sample could be attributed to O-H (phenol) bonding. A shift in the peak at 1615 for RSBC to 1637.62 of the loaded sorbent may be attributed to the stretching of C=O corresponding to carbonyls, olefinic C=C stretching frequencies of hemicelluloses, lignin and amino groups. This was more prominent in the loaded sorbent indicating higher sorption due to structural modification¹³. The sharp peak observed at 1741.78 for RSBC was assigned to C-O bond of carboxylic and its complete disappearance on the Pb-loaded sorbent inferred that the lead metal bonded to the RSBC through interaction with active groups OH, COOH¹⁴. Figure 2 shows the SEM images of the RSBC and the presence of the Pb (II) after adsorption. The

micrographs indicated that the surface was irregular, porous in nature and has many micropores and hollow cavities. Figure 1: FT-IR spectra : (a) RSBC; & (b) Pb-loaded MSBC. The sorbed Pb can be vividly seen as it is well entrapped within the pores.



Figure 2: SEM of (a) Shea butter cake (RSBC) (b) sorbed Pb in the cake

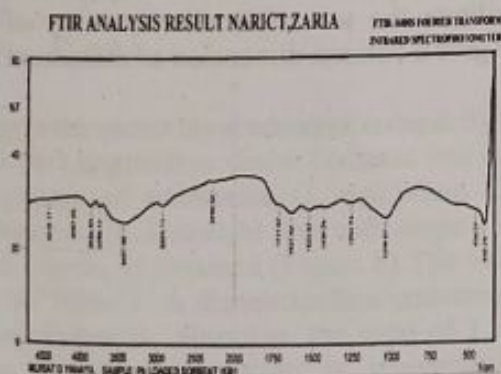
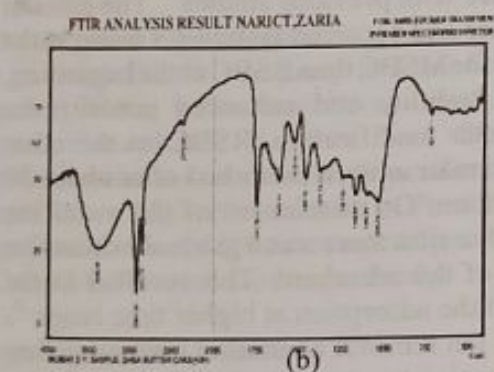


Figure 1: FT-IR spectra: (a) RSBC (b) Pb-loaded MSBC

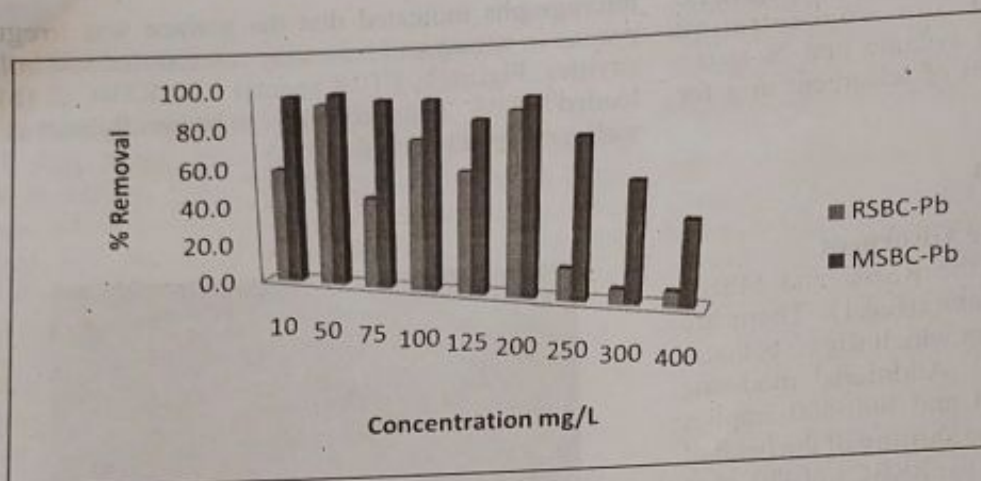


Figure 3: Percentage removal of Pb(II) on RSBC and MSBC at various concentrations

The percentage removal of Pb (II) on to the RSBC and MSBC was high (Figure 3). There was a sharp increase in percentage removal of the metal ions from the starting initial concentration for MSBC compared to the RSBC. Approximately 96% of Pb(II) was removed at initial metal concentration of 10mg/l while 58.7% was removed for RSBC at the same concentration. This high rate of adsorption may be due to the enhanced adsorptive sites the MSBC had for the removal of the Pb(II). Nevertheless, percentage removal for RSBC increased as a result of increase in concentration which provided the driving force to overcome mass transfer resistance between the metal ion and adsorbent¹⁵.

At lower initial concentration almost all the sites were filled during adsorption as there was greater interaction between the metal ion and adsorptive sites, but as the concentration increased more Pb(II) were left unadsorbed due to saturation of the sites. The higher percentage removal observed for MSBC despite at higher (250mg/L) concentration was due to its stability, which may be attributed to the calcium alginate. This played an important role in the mechanical strength, rigidity, enhanced porosity of the adsorptive sites, resistance to abrasion and leaching of the organic component to the solution. The instability indicated for the RSBC was because of interference from its organic constituents that may have leached into the aqueous

media. This thus necessitated the need for modification as seen using MSBC. Several authors have successfully used calcium alginate^{16,17,18}.

3.3 Effect of Time

The amount adsorbed in mg/g versus time gave a high increase in adsorption at lower contact time (<30mins of agitation time) before reaching equilibrium (Figure 4). The equilibrium concentration¹⁹, at 4hr contact time was found not to be greater than approximately 5% after 2hr; based on this observation, a 2hr contact time was assumed for the sorbent to reach saturation. This was in accordance with previous studies²⁰. The amount adsorbed is higher and occurred faster (< 5 mins) at the start of agitation for MSBC than RSBC at the beginning, because of the stability and enhanced porosity the adsorbent has after modification. RSBC on the other hand recorded similar amount adsorbed after about 30 minutes of agitation. On attachment of the metal ion onto the adsorptive sites there was a gradual exhaustion of the capacity of the adsorbent. This resulted in the slowing down of the adsorption at higher time range²¹. The rapid metal ion removal at smaller time range has significant economic importance in the scale-up process as this will facilitate the use of smaller quantity of sorbent that will give a column dimension of maximum efficiency²².

3.4 Effect of pH

The pH of metal solutions has been identified as the most important variable governing metal sorption²³. Percentage removal of Pb (II) was found to be very low at lower initial pH as a result of the dissociation of the water molecules to form hydrogen ions which are themselves strong competing ions with the sorbates. As the pH values increases, sorption increases as a result of decrease in the protons which consequently lowers the electrostatic repulsion between the Pb (II) and surface sites²⁴. In Figure 5, the MSBC showed better stability in the removal efficiency as compared to RSBC. Percentage removal increased until it reached a pH greater than 6 where possibility of precipitation cannot be overruled²⁵.

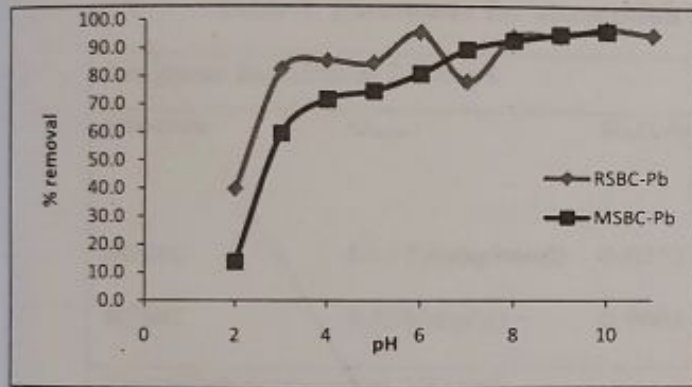


Figure 5: Percentage removal of Pb (II) on RSBC and MSBC at various initial pH

3.5 Adsorption Equilibrium

In-order to check for the adequacy and the fits of the experimental data with theoretical models, the Langmuir, Freundlich, Temkin and Dubunin-Raduskevich isotherms were used. Experimental runs were carried out at high concentration of Pb at 250mg/L, 200rpm, agitation time of 120mins, and 0.5g or 40 beads of RSBC and MSBC were used respectively.

The Langmuir equation is given by:

$$\frac{C_e}{q_e} = \frac{C_e}{q_m} + \frac{1}{K_L q_m} \quad (3)$$

where q_m is the mono-layer adsorption capacity (mg/g) and K_L is the Langmuir isotherm constant that is related to free energy of adsorption (L/mg). When C_e/q_e is plotted against C_e , a straight line with slope $1/q_m$ and intercept $1/q_m K_L$ is obtained (Figure 6). The values are enlisted in Table 1. A dimensionless constant, called separation factor R_L describes the type of Langmuir isotherm as: $R_L=0$ (sorption is irreversible), $0 < R_L < 1$ (sorption is favourable) and $R_L > 1$ (sorption is

unfavourable)¹³. The correlation coefficient R^2 was greater than 0.99 and approaches unity which showed the adequacy and fitness to the isotherm. The near overlap of the graphs of the two sorbents showed the closeness of the correlation coefficient (R^2 for MSBC=0.994; R^2 for RSBC=0.998). The free energies of sorption Langmuir constant (K_L) as calculated from equation (3) were 0.02 and 0.06 L/mg for RSBC and MSBC respectively. This was higher for MSBC, an indication of adsorption energy between the adsorbent and metal ions. The separation factor showed a favourably high adsorption even at high concentration²⁶

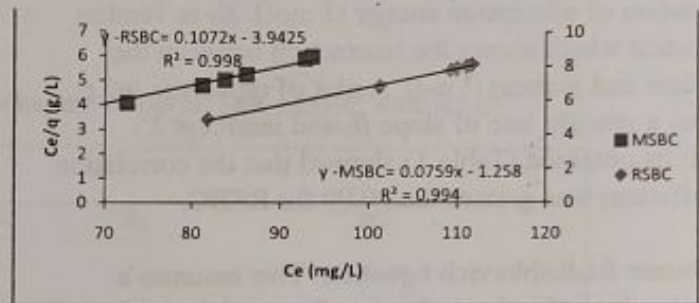


Figure 6: Langmuir Isotherm on the Adsorption of Pb(II) on RSBC and MSBC

The Freundlich equation is given by:

$$\ln q_e = \ln K_f + \left(\frac{1}{n}\right) \ln C_e \quad (4)$$

where, K_f is a constant which represents the sorption capacity (mg/g) and $1/n$ is a constant which gives the intensity of adsorption. A plot of $\log q_e$ against $\log C_e$ gives a straight line of slope $1/n$ and intercept $\ln K_f$. $1/n$ in the range of 0 to 1 represents a favourable adsorption.

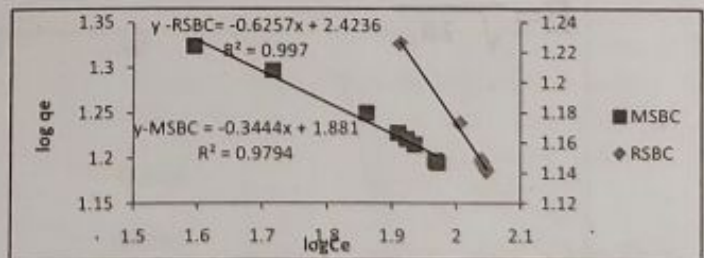


Figure 7: Freundlich Isotherm for Pb(II) adsorption RSBC and MSBC

The values for Freundlich constants and correlation coefficients (R^2) were presented in Table 1. Values of n computed from the slope were 1.598 for RSBC and 2.903 for MSBC which showed a favourable adsorption. The Temkin equation is given as:

$$q_e = B_1 \ln K_T + B_1 \ln C_e \quad (5)$$

$$B_1 = \frac{R_T}{b}$$

where the heat of adsorption, b , represents the variation of adsorption energy (J/mol). K_T is Temkin constant which shows the interaction between the sorbate and sorbent (l/mg). A plot of q_e versus $\ln C_e$ gives a straight line of slope B_1 and intercept K_T . Results obtained (Table 1) showed that the correlation coefficient was greater than 0.99 for RSBC.

Dubunin Radushkevich equation: This assumes a fixed volume for the sorbent surface and determines the heterogeneity of the energy of sorption within that space¹³

$$q_e = q_D \exp[-B_D [RT \ln (1 + \frac{1}{C_e})]^2] \quad (6)$$

The constant, B_D , is related to the mean free energy of sorption per mole of the sorbate as it is transferred to the surface of the solid from infinite distance in the solution and this energy can be computed using the following relationship as given by¹⁴

$$E = \frac{1}{\sqrt{2B_D}}$$

Where 'R' is a gas constant in $\text{kJmol}^{-1}\text{K}^{-1}$, T is the temperature in Kelvin, C_e (mg/L) and q_D is the sorption capacity in mg/g. The plots of $\ln q_e$ versus $\left(\ln\left(1 + \frac{1}{C_e}\right)\right)^2$ gives a slope: $-B_D R^2 T^2$ and the intercept $\ln(q_D)$.

The linear correlation coefficient was low for the MSBC but greater than 0.9 for RSBC (Table 1). The maximum sorption energy required was 0.0469 KJ/mol for RSBC and 0.3164 KJ/mol for MSBC which is in the range of $0 < E < 8$ kJ/mol and this suggested that adsorption followed a physical adsorption process²⁹.

Table 1. Parameter for absorption isotherms of pb (II) on MSBC and RSBC

Langmuir Isotherm at 250mg/l				
Samples	Q_{\max}	K_L (L/mg)	R_L	R^2
MSBC	13.175(mg/bead)	0.0272	0.128	0.994
RSBC	9.328(mg/g)	0.0603	0.062	0.998
Freundlich Isotherm at 250mg/l				
Samples	K_F	N	R^2	
MSBC	76.032(mg/bead)	2.903	0.979	
RSBC	265.216(mg/g)	1.598	0.997	
Temkin Isotherm at 250mg/l				
Samples	B_1	K_T (L/mg)	R^2	
MSBC	396.143	44.446	0.988	
RSBC	280.472	59.018	0.998	
Dubunin-Radushkevich Isotherm at 250mg/l				
Samples	q_D (mg/g)	B_D	E_D (KJ/mol)	R^2 (non-linear)
MSBC	534.69	-44.944	0.1055	0.8866
RSBC	256.684	-227.26	0.0469	0.9864

4.0 Conclusion

The effects of raw shea butter cake and entrapping shea butter cake on Pb (II) biosorption was evaluated. The presences of functional groups especially the -OH, -COOH- and -NH₂ in both cakes were found to be responsible for the binding of the Pb (II). Optimum pH of 6 was obtained and percentage removal of Pb (II) was greater than 90% at initial metal ion concentration of 10 mg/L to 200mg/l and dropped to less than 50% as concentration increased to 400 mg/L for MSBC. The stability and high sorption removal was as results of the enhanced porosity and absence of leaching of the organic constituents, which was well observed in the RSBC performance. Sorption rate was very fast for MSBC (<5 mins) than RSBC (30 mins) and maximum equilibrium time was 120mins. The results of the isotherm studies showed that biosorption experimental values adequately fitted all the isotherms with the highest correlation coefficient of values greater than 0.99 obtained for Langmuir isotherm. The Langmuir maximum adsorption capacity was 13.175 mg/bead and 9.328mg/g for MSBC and RSBC, respectively. Sorption energy was low which indicated that it was physical adsorption and metals adsorbed can be recovered. This exceptional behaviour of the MSBC thriving well in polluted effluents can serve as an alternative agro-waste material for the treatment of effluents generated during mining operations at homes rather than carelessly discarding them in the environment.

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