

# Fixed Bed Column Study for Pb (II) adsorption using Calcium-Alginate Treated Shea Butter Husk (TSBH)

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**Abstract—** The continuous adsorption of Pb (II) in a fixed column was studied in this work. Shea butter husk was treated in a polymeric mixture of calcium alginate to produce beads of approximately  $1.56 \text{ mm} \pm 0.14$  spherical shapes. Optimum parameters evaluated revealed an effective column performance at flow rate of 0.5 ml/min, bed heights of 12 cm and at initial concentrations of 50 mg/L. The percentage removal (%R) of the Pb (II) ions at 0.5 ml/min was 77.31 % with adsorption capacity of 1.888 mg/wet bead. The Thomas and Yoon Nelson model predicted the break through curve to a large extent with the correlation coefficients ranging between 0.8098-0.9676. Their rate constants ( $K_{Th}$  and  $K_{YN}$ ) were higher at lower flow rate, higher bed heights and at lower concentrations. The adsorption capacities of the column at this conditions was 6431.606 and 76, 536.111 mg/g respectively for Thomas and Yoon Nelson model. These results revealed that with the enhancement of the shea butter husks, polluted effluents concentrations can be reduced to their threshold values before discharged into the water bodies via a continuous fixed bed column.

**Keywords—**Pb (II), adsorption, column, shea butter husk.

## I. INTRODUCTION

Over the years low cost agricultural wastes such as groundnuts shell [1], cassava wastes [2], [3], corncobs [4] etc., have been used as an alternative for the removal of heavy metal ions from aqueous solutions. The conventional methods which include

chemical precipitation, ion exchange, membrane separation and adsorption by activated carbon have suffered some drawback because of the high capital cost, disposal of resulting sludge [5] and sometimes its ineffectiveness. The agricultural wastes which have received so much attention also present some limitations either as a results of their fragility, non-uniform shape or leaching tendency; thus making them un-suitable for large-scale processes. These can however be solved through entrapment in a polymeric matrices. The technique is based on the use of another substance to hold the active micro-particles together [6]. The polymers are mainly polyacrylamide, polysulfone, calcium alginate, and polyhydroxyethylmethacrylate [6]; [7].

Authors that have successfully utilized the calcium alginate for heavy metal removal under batch adsorption technique to mention a few are [8]; [9]; [10]; [11]. This study will focus on using the calcium-alginate Shea butter husk for the removal of Lead- (Pb) (II) under the fixed bed column.

The design of fixed bed column includes estimation of shape of breakthrough curves and the emergence of breakpoint [12]. The performance of the column is described by the breakthrough curve which was explained by [13] as the loading behavior of metals to be removed from solution expressed the adsorbed metal concentration as the ratio of effluent concentration to inlet concentration ( $C_t/C_o$ ) as a function of time or volume of effluent for a given bed height. The dynamics of adsorption process was studied using several simple mathematical models such as [14] and [15] models to predict the dynamic

behavior of the column and determination of the kinetic coefficient.

## 1.0 Materials and Methods

### 1.1 Preparation of calcium alginate shea butter husk

The Shea fruits gathered from Kataeregi farmland in Minna, Nigeria were subjected to the following processes: De-pulping which is the removal of the fleshy meso-carp was followed by drying which helps 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 [16]. This is further crushed with a blender and then sieved with an electric sieve shaker to obtain a smooth powder of particle size <250 µm.

The treated Shea butter husk (TSBH) 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 [7]. 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 a homogeneous mixture of alginate and the powder. Afterward, the solution was added drop wise via a syringe to a solution of 0.1 M calcium chloride dihydrate (CaCl<sub>2</sub>·2H<sub>2</sub>O) under low agitation (20 rpm). 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 treated shea butter husks (TSBH) 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 refrigerator at around 4 °C for further use.

### 1.2 Preparation of stock solution

Deionized water was used to prepare all solutions. All reagent used were of analytical grade. Stock solution (1000 mg/L) of lead was prepared by approximately dissolving 1.5980 g of lead nitrate [Pb(NO<sub>3</sub>)<sub>2</sub>] in 100 ml of deionized water and dilute to one liter in a volumetric flask with deionized water.

### 1.3 Fixed bed experiment

Fixed bed column adsorption experiments were conducted in a small 1.8 cm (I.D) diameter glass column of length 25 cm. The column was filled to a height ranging from 4 cm, 6 cm and 12 cm corresponding to 11.9, 20.8 and 25.8 g of TSBH respectively. The TSBH are of approximately 1.56 mm ± 0.14 spherical shapes. The beads were allowed to fall into the column under force of gravity. This was supported over a glass wool and glass beads to prevent falling out of the sorbent. The metal ion solution containing concentrations at 50 mg/L, 100 mg/L and 250 mg/L of Pb was fed to the column through a down flow at a flow rates of 0.5 ml/min, 2.5 ml/min and at 5.0 ml/min using flow set. The column was open to the atmosphere in order to maintain the internal pressure in the columns near atmospheric pressure [17]. The solution leaving the bottom of the column was collected at various time intervals and the samples were analysed [18] using Flame Atomic Absorption Spectrophotometer Varian SpectrAA 220FS. Flow continues until the effluent concentration almost equal influent concentration.

### 1.4 Column performance evaluation

The dynamic behavior of the column is described by the 'effluent – time concentration' profile called the breakthrough curve. The steeper the breakthrough curve the more efficient the utilization of the sorbent in the column [19]. The mass transfer zone is the surface of the bed where sorption occurs. As the fluid begins to flow through the column, the mass transfer zone varies from 0 % of the inlet concentration (corresponding to solute free sorbent) to 100 % of the inlet concentration (corresponding to the total saturation [20]). Saturation time,  $t_s$ , is established when the concentration in the effluent is higher than 90–95% of the inlet concentration and the service time is the breakthrough time,  $t_r$ , is established when the effluent concentration reaches normally the disposal allowable limits of the metal. For Pb (II) the maximum allowable limit for drinking water is 0.01 mg/L [21].

The effluent volume  $V_{eff}$  (ml) according to [7] is calculated as:

$$V_{eff} = Qt_{total} \quad (1)$$

Where  $Q$  is the volumetric flow rate (ml/min) and  $t_{total}$  is the total flow time in minutes.

The area under the breakthrough curve as given by [20] represents the total mass of metal adsorbed,  $q_{total}$ , in mg, for a given feed concentration and flow rate and it can be determined by integration

$$q_{total} = \frac{Q}{1000} \int_{t=0}^{t=t_{total}} C_R dt \quad (2)$$

Where  $C_R$  is the concentration of metal removal in mg/L.

The total metal ions sent to the column in mg can be calculated from (3)

$$m_{total} = \frac{C_i Q t_{total}}{1000} \quad (3)$$

The percentage removal of the metal ions is the ratio of mass of metal mass adsorbed ( $q_{total}$ ) to the total amount of metal ions sent to the column ( $m_{total}$ )

$$\%R = \frac{q_{total}}{m_{total}} 100 \quad (4)$$

The amount of metal adsorbed at equilibrium  $q_e$  (mg of sorbed metal/wet bead), and the equilibrium metal concentration,  $C_e$  (mg/L), can be determined using the equations below,

$$q_e = \frac{q_{total}}{m} \quad (5)$$

$$C_e = \frac{m_{total} - q_{total}}{V_{eff}} 1000 \quad (6)$$

Where  $m$  is the mass of sorbent in g.

## 1.5 Adsorption models

Developing a model that describes adsorption in a column is tasking as it is not a steady state process. Studies have shown that the developed equations are differential in nature and usually require complex numerical methods to solve [20]. However, there are other mathematical models that have been developed and tested by several authors. These can be fitted to predict the dynamic behavior of the column and allow some parameters to be estimated as well. Such models like Thomas and Yoon - Nelson [20]; [21] have been well studied. These models are described below and the experimental data obtained in the column sorption of Pb (II) shall be fitted on them.

### 1.5.1 Thomas model:

This is one of the models used traditionally for the prediction of the concentration-time profile or the breakthrough curve for column performance evaluation [22] explained that it assumes the Langmuir kinetics of adsorption-desorption and that no axial dispersion derived with the adsorption such that the rate driving force obeys second-order reversible reaction kinetics. Its main limitation is that its derivation is based on second order kinetics which considers that sorption is not limited by the chemical reaction but controlled by the mass transfer at the interface. This discrepancy can lead to errors when this method is used to model biosorption processes in specific conditions [20]. The expression using the Thomas model for adsorption column is as follow [23]:

$$\frac{C_f}{C_i} = \frac{1}{1 + \exp[K_T (q_o m - C_o V_{eff}) / \Theta]} \quad (7)$$

Where,  $K_{TH}$  is the Thomas rate constant (ml/(min mg)) and  $\theta$  is the volumetric flow rate (ml/min),  $q_o$  is the maximum solid phase concentration of the solute (mg/g),  $m$  is the amount of sorbent in the column and  $V_{eff}$  is the effluent volume,  $C_i$ ,  $C_f$  is the influent and effluent concentration in (mg/L) respectively. The linearized form of the Thomas model is as follows:

$$\ln\left(\frac{C_i}{C_f} - 1\right) = \frac{K_T q_o m}{\Theta} - \frac{K_T C_o}{\Theta} V_{eff} \quad (8)$$

The  $K_T$  and  $q_o$  can be determined from the plot of  $\ln((C_i/C_f)-1)$  against  $V_{eff}$  at a given flow rate

### 1.5.2 The Yoon and Nelson (1984) model:

This was developed based on the adsorption of vapour or gases in activated coal. It stated that decrease in the probability of sorption for each sorbate molecule is proportional to the probability of sorbate sorption and the probability of sorbate breakthrough on the sorbent [24]. Reference [25] stated that the Nelson model is less complicated than other models and also requires no detailed data concerning the characteristics of sorbate, the type of sorbent and the physical properties of sorption bed. The Yoon and Nelson equation regarding to a single component system is expressed thus [26]:

$$\frac{C_f}{C_i} = \frac{1}{1 + \exp[k(\tau - t)]} \quad (9)$$

Where  $k$  is the rate constant ( $\text{min}^{-1}$ ),  $\tau$  the time required for 50 % sorbate breakthrough (minutes) and  $t$  is the breakthrough (sampling) time (minutes). The linearized form is given as:

$$t = \tau + \frac{1}{k} \ln \frac{C_f}{C_i - C_f} \quad (10)$$

The plot of sampling time ( $t$ ) versus  $\ln[C_f/(C_i - C_f)]$  gives  $\tau$  as the slope of the straight line and  $k$  the intercept.

To establish the relationship among sorption capacity of the column ( $q_0$ ), inlet concentration ( $C_0$ ), liquid flow rate ( $\Theta$ ) and the 50% breakthrough time ( $\tau$ ), reference [27] gave the equation as:

$$q_0 = \frac{1}{2} C_0 \Theta (2\tau) = C_0 \Theta \tau \quad (11)$$

## 2 Results and Discussion of Results

### 2.1 Effect of flow rate

Effect of flow rate on Pb (II) on TSBH at constant bed height and initial metal ion concentration in a continuous flow bed column was examined by varying the flow rate from as low as 0.5 to 5 ml/min. The plots of normalised Pb (II) concentration ( $C_f/C_i$ ) versus bed time at different flow rates are shown in fig 1.

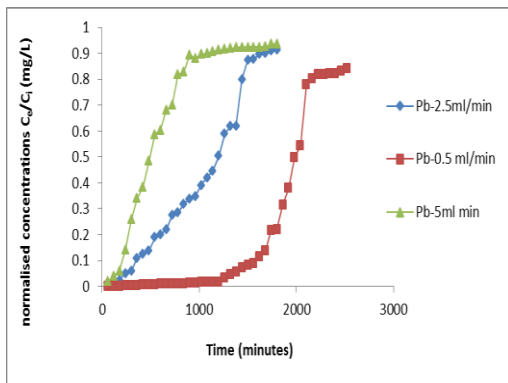


Fig 1: Effect of flow rate on the breakthrough curve for Pb (II) adsorption on MSBH at  $C_i=50$  mg/L and bed depth 12 cm

The results show that adsorption of Pb (II) on the TSBH was strongly dependent on flow rate. From the contact point where the influent first struck the sorbent, the Pb (II) rapidly get adsorbed on the bed. This is a result of availability of sorption sites [28]. As the flow continues, there is gradual saturation of

the sites and thus less effectiveness in the uptake of the metal ions until a point where the influent concentration almost equals effluent concentration (bed is fully saturated/exhausted). The column performed well at lower flow rate. Earlier break point and exhaustion time were achieved when the flow rate was increased from 0.5 ml/min to 5 ml/min. The comparative normalized concentration ( $C_f/C_i$ ) for Pb (II) ion was 0.01 mg/L [29]. This value was attained at 360 minutes for flow rate 0.5 ml/min while; at flow rate of 5 ml/min it was 240 minutes. These fast break point time is attributed to the fast discharge of the Pb (II) ions into the TSBH. This invariably means that the residence time for the solute to interact with the surface of the sorbent is too short to diffuse into the pores of the sorbent and as such the metal ions leave before equilibrium occurred. The percentage removal of the metal ions decreased with increase in flow rate [12]. This is evident in the percentage removal (% R) that was lower at higher flow rates as compared to the lower flow rates (see Table 1). The longer residence time at lower flow rate allows the positively charged solute (Pb II) to diffuse and reach the negatively charged carboxylate or hydroxyl group ( $-\text{COOH}-$ ;  $-\text{OH}-$ ) active sites of the Shea butter husks encapsulated in calcium alginate.

Amount adsorbed of metal in mg/wet bead is a function of total metal adsorbed which were higher at higher flow rate for both metals. This is so because the total metal sent to the column is a function of flow rate and time, although much metal are sent at higher flow rates, amount adsorbed of the total is smaller when compared at lower flow rate. This can be ascertained thus; total metal sent at 0.5 ml/min was 63 mg out of which 48.7 mg was adsorbed and percentage removal was 77.31 % while at 5 ml/min, total metal sent was 450 mg, out of which 141.214 mg was adsorbed and percentage removal was 32.38%.

### 2.2 Effect of bed height

The amount adsorbed of metals in the fixed bed column is dependent on the quantity of sorbent in the column [30]. The experiment was performed at heights of 4 cm, 6 cm and 12 cm. The flow rate and initial metal concentration were kept at 0.5 ml/min and 50 mg/L respectively. The amount of metal ion

adsorbed and percentage removal increased with increase in bed height. At a height of 4 cm, the amount of Pb (II) adsorbed increases from 45.19 mg/g to 61.40 mg/g at 12 cm while percentage removal increases from 45.19 % at 4 cm to 97.5 % at 12 cm. This increment is as result of increase in the amount of sorbent in the column which provided more binding sites for the metal ion [7]. It is also observed that the break through and exhaustion time increased with rise in the bed height as there exist large amount of sorbent in the bed for the solute to travel through before their exit from the column [31]. For an inlet concentration of 50 mg/l and for a break through concentration of 0.01 mg/L for Pb (II) ions, the time varies approximately from 60 minutes to 240 minutes as the bed heights increases from 4 cm to 12 cm. Reference [30] explained at lower bed depth, the axial dispersion phenomenon predominate in mass transfer and this reduces the diffusion of the metal ions into the whole sorbent mass which consequently bring about the reduction in breakthrough time. With increase in bed heights, the residence time is increased and allowing the metal ions to diffuse deeper inside the sorbent Authors with similar results are [20]; [30] and [7].

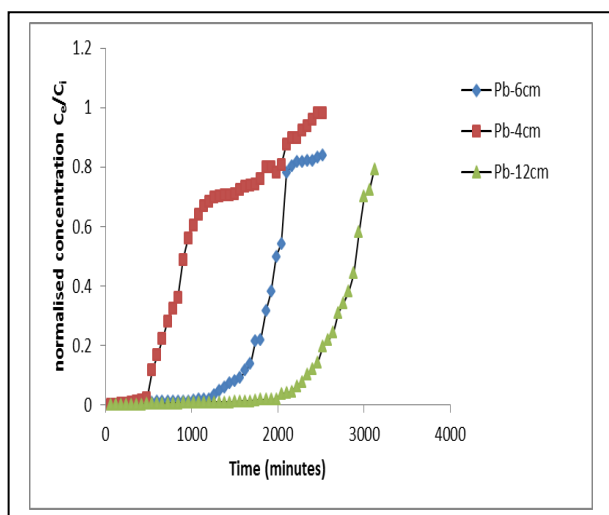


Fig. 2: Effect of bed height on the breakthrough profile for Pb (II) adsorption on MSBH at  $C_i=50$  mg/l, and flow rate 0.5 ml/min.

### 2.3 Effect of initial metal ion concentration

The effect of concentration change was studied at 50 mg/L, 100 mg/L and 250 mg/l while keeping the bed heights and flow rates at 12 cm and 0.5 ml/min respectively. A change in the inlet concentration

affects the operating characteristics of the fixed bed column. At concentration of 50 mg/L, the breakthrough occurred lately because the lower concentration gradient causes a slower mass transfer coefficient as a result of the decreased in diffusion coefficient [32]. For the sorption Pb (II) the breakthrough occurred after 360 minutes of contact at 50 mg/l while at 100 mg/L and 250 mg/L, it was much less than 60 minutes of contact. At higher concentration, the binding sites get more quickly saturated and the breakthrough curve less steeper (see fig. 4). The amount of total sorbed metal, equilibrium metal uptake and total percentage removal increased with increasing concentration of Cu (II). Authors with similar results are [21]; [31].

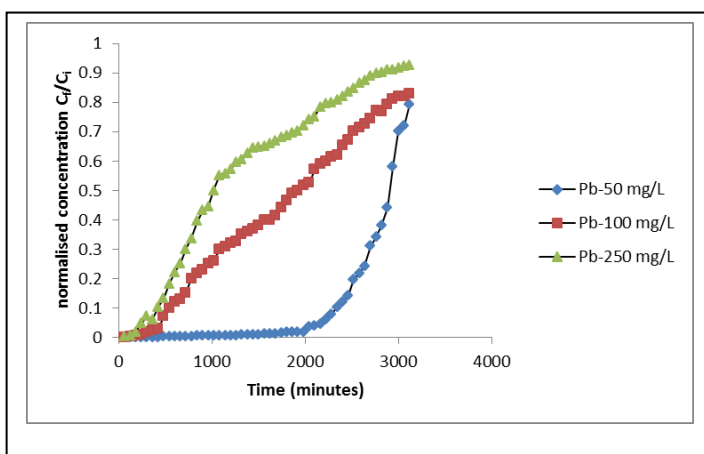


Fig. 3: Effect of initial metal concentration on the breakthrough profile for Pb (II) adsorption on TSBH at bed height 12 cm, and flow rate 0.5 ml/min.

### 2.4 Break through curve modeling with Thomas model

The Thomas model does adequately reproduce the experimental data. The correlation coefficient,  $R^2$  ranges from 0.8098- 0.9976. The adsorption capacities were quite high at higher bed heights, higher solute concentrations and at higher flow rate. These trends were similar to the experimental date although there were significant differences in their values. The kinetic ( $k_{Th}$ ) constant decreases with increase in flow rate from 0.5 ml/min to 5.0 ml/min which implies that breakthrough curve is favorable at lower flow rate [24].

Table 1: Parameters predicted from Thomas model on Pb (II) adsorption on TSBH.

Flow rate (ml/min)	$K_{Th} \times (10^{-5})$ ml/mg.min	q (mg/g)	$R^2$
0.5	7.2	1973.676	0.9565
2.5	7.2	5221.250	0.9676
5.0	6.6	66156.000	0.8098
Bed Heights (cm)	$K_{Th} \times (10^{-5})$ ml/mg.min	q (mg/g)	$R^2$
4	7.2	4049.197	0.8354
6	7.2	1973.676	0.9565
12	5.4	6431.606	0.9351
Concentrations (mg/L)	$K_{Th} \times (10^{-5})$ ml/mg.min	q (mg/g)	$R^2$
50	5.4	2966.516	0.9351
100	2.0	37775.872	0.8270
250	0.76	7197.572	0.8434

The  $k_{Th}$  values also decreases with increasing influent concentrations which also indicated sorption is more favorable at lower concentration as there is delay in the saturation of the sorbents more favorable at lower concentration as there is delay in the saturation of the sorbents which thus offers better interactions between the sorbates and sorbents [32]. The values of Thomas parameters are indicated are on Table 1.

## 2.5 Yoon and Nelson Model:

This model was used to investigate the breakthrough behavior of Pb (II) in the fixed bed column. The plot of  $\ln(C/(C_0-C))$  versus time yield the slope which is the parameter  $k_{Yn}$  (rate constant) and the intercept,  $\tau$  (the time required for 50% solute breakthrough). The obtained values were used to calculate the bed capacity at chosen flow rates and concentrations level. Values obtained were in close agreement with the experimental data to some extent and are presented in Table 2. As the flow rate increases, the rate constant also increases while the  $\tau$  decreases. This is an indication of earlier

break through at higher flow rate and shorter time for the sorbate to travel the length of the column. The correlation coefficient,  $R^2$  ranges from 0.8098 to 0.9676. The bed capacity values ' $q_{0Yn}$ ' decreases with increase in flow rate as a result of the inadequate level of interaction between the sorbate and the sorbent (intra-particle diffusion). Large differences were recorded between the experimental and predicted values of bed capacity. This may be due to assumption made on neglecting effect of axial dispersion [21]. The time necessary to reach the 50% breakthrough decreases as the inlet solute concentration increase [32].

Table 2: Parameters predicted for Yoon-Nelson model

higher concentration which is an indication of higher performance at lower concentrations.

Parameters	$\tau_{calc}$ (mins)	$\tau_{exp}$ (mins)	$q_{calc}$ (mg/g)	kyn	$R^2$
Concentrations (mg/L)					
50	2296.083	2900	57402.083	0.0036	0.9351
100	1948.350	960	97417.500	0.0020	0.827
250	1485.579	400	185697.368	0.0019	0.8434
Bed Heights (cm)					
4	1344.333	920	33608.333	0.0036	0.8354
6	2036.833	2000	50929.833	0.0036	0.9565
12	3061.444	2900	76536.111	0.0027	0.9351
Flow rate (mg/L)					
0.5	2036.833	2000	50920.85	0.0036	0.9565
2.5	1077.670	1200	134708.75	0.0036	0.9676
5.0	620.67	500	155167.500	0.0033	0.8098

## CONCLUSION

The adsorption of Pb (II) was investigated in a continuous fixed bed column using Treated shea butter husk. It was found that sorption is greatly influenced by the flow rate, bed heights and influent metal concentration. From the obtained results the following conclusions were drawn:

1. Increasing the flow rate increases the breakthrough time and amount of metal ion adsorbed and thus makes the curve become less steep.
2. The amount of total sorbed metal, equilibrium metal uptake and total percentage removal increased with increasing influent concentration
3. It is also observed that the break through and exhaustion time increased with rise in the bed height
4. The Yoon-Nelson model 50% breakthrough time corresponded to some extent with experimental data which is an important parameter for bed exhaustion The kinetic constant 'kyn' reduces at

5. This model can be combined for predicting design parameters under the same process conditions from simple experiments.

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## BIOGRAPHIES

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**Prof. Joseph O. Odigure** is a Professor of Chemical Engineering. He has served as Dean of School of Engineering and Engineering Technology and Foundation Coordinator, School of Information and Communication Technology, Chairman, Committee of Deans of Engineering and Technology of Nigerian Universities. He has authored and co-authored over 70 patents, research journal article, books and conferences proceedings and serves as referee to various international journals.

He served in more than twenty international, national and local committees, he was the principal consultant in the Design and Construction of Fertilizer Blending Plant, by Akkary Nig. Ltd. Minna and in the Dependability assessments of collapsed building on plot No SDD48 at the prototype housing scheme site, Gwarimpa, Abuja. 1998. Team member and Consultant, Design and construction of Essential Oil Plant by National Research Institute for Chemical Research Technology, Zaria in 2004. Member of Research team into Building collapse in Nigeria set up by the Nigeria Building and Road Research Institute, Abuja 2009.

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