



Equilibrium Sorption of Lead (II) in Aqueous Solution onto EDTA-modified Cocoa (*Theobroma cacao*) Pod Husk Residue

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ABSTRACT

The adsorption of lead (II) ions onto EDTA- modified cocoa pod husk (CPH) in an equilibrium system was investigated. Kinetics, mechanism and thermodynamic parameters were estimated. Adsorption capacity was rapid and time dependent as appreciable level of Pb (II) uptake was achieved at an optimum contact time of 50 minutes. Three kinetic models were used to fit the kinetic data; Pseudo-first order, Pseudo-second order and intraparticle diffusion. Adsorption of the metal ions follows the Pseudo-second order and intraparticle kinetics as these provide a better fit to the experimental data with high R^2 values ranging from 0.9776 – 1. Thermodynamic parameters such as change in Gibbs free energy (ΔG°) change in enthalpy (ΔH°), and entropy change (ΔS°) were calculated. The ΔG° ($-5.499 \text{ kJmol}^{-1}$), ΔH° (60.36 kJmol^{-1}) and ΔS° ($212.44 \text{ Jmol}^{-1}\text{K}^{-1}$) of the adsorption process of metal ions onto the modified adsorbent was found to be feasible, endothermic, spontaneous and physiosorption in nature. These results indicate that modified CPH is a promising sorbent for the removal of Pb (II) in industrial effluent and wastewaters containing same.

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INTRODUCTION

The increasing rate of industrialization often generate heavy metal load into the environment and this has constituted serious threat to plant and animal lives today. Among these heavy metals is lead, a toxic metal commonly found in industrial workplaces. These contaminations are derived from various activities such as mining of ores, tanneries, electroplating, vehicular movements as well as petrochemical refineries. Heavy metal contamination to the environment even at low concentration can be toxic [1]. Because of the potential health hazard associated with their discharge, serious concern has been raised [2]. The heavy metal lead for example can interfere with enzymatic activities as well as the formation of red blood cell. Lead is a heavy metal poison, which can form complexes with the oxo- groups in enzymes thus affecting all steps in hemoglobin and

porphyrin production [3]. Lead ingestion by man can result in encephalopathy, seizures, and mental retardation [4]. Since most of the heavy metal such as lead are non degradable into non toxic end products, their concentrations must be reduced to permissible limit before discharge into the environment.

The conventional technologies such as chemical precipitation, chemical coagulation, ion –exchange/chelation, solvent extraction/liquid membrane separation, electrodialysis, ultrafiltration, reverse osmosis etc are well known and available for the treatment of waste water containing heavy metal ions [5-7]. Due to high cost, sensitive operating conditions, and the production of wastes, these methods may not be well embraced in the industries [8]. It is against this back draw that alternative sorbent materials are sought. The use of agricultural residues as adsorbent have received great attention for many years, although the cost of these

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processes may sometimes be expensive [9-11]; however, their use as adsorbent for heavy metal ions cannot be overemphasized.

Essentially, agricultural residues consist of cellulose and lignin as their major constituents. They may also contain other polar functional groups of lignin such as aldehydes, ketones, alcohols, carboxylic, phenolic and ether groups. Several workers have reported the use of agricultural residues as alternative and efficient materials for heavy metal ions abatement [12-18]. The cocoa industry generates high volume of cocoa pod husks which are alternate host of pathogens in cocoa plantations. The present study was a continuous effort in exploiting opportunities for agricultural biomass management. The present study reports the adsorption of Pb (II) onto EDTA-modified cocoa pod husk.

METHODS

Preparation of Adsorbent

Cocoa pod husk used for this experiment was obtained from the fermentation unit of the Cocoa Research Institute of Nigeria, Ibadan, Nigeria. The pod husks were washed thoroughly with water to remove dirt. The pods were chopped into small pieces, washed again and air dried and crushed into powder using a manually operated grinder. This was further dried in an oven at 105°C for 6 hours and allowed to pass through a sieve. The meal retained on sieve of mesh size 250µm was retained for the study.

Activation of Adsorbent

The screened portion of the pod husk retained on the 250µm mesh was then steeped in dilute nitric acid solution (2% v/v) for 24hrs. After 24 hours duration, the meal was filtered using a Whatman No. 41 filter paper and then washed with distilled water, dried at 105°C for 12hours and then stored in a desiccators.

Modification of adsorbent

About 100g of the activated cocoa pod husk was taken out for chemical activation according to the method described in literature [19]. In a typical experiment, 50g of the husk was hydrolyzed with 7% (v/v) aqueous sulphuric acid for 90 minutes. The mixture was filtered and the residue was washed with deionized water and dried. 20g of the hydrolyzed husk was treated with 0.3 dm³ of pyridine and 5.67g EDTA under reflux for 24 hours. The mixture was filtered, washed severally with water until free of pyridine and then dried in the air. The modified husk became EDTA –modified cocoa pod husk (ECPH).

Preparation of Adsorbate

All reagents used for this study were of analytical reagent grade and were used without further purification.

Doubly-distilled and deionized water was used in the preparation of all sample solution. Stock solution of Pb²⁺ metal ion of 1000mgL⁻¹ concentration was prepared using lead nitrate (Pb(NO₃)₂). From the stock solution, aliquot of 100mgL⁻¹ (initial concentration) of metal ion was prepared by serial dilution with deionized water.

Adsorption studies

Adsorption studies for Pb²⁺ were carried out for each adsorbent (unmodified cocoa pod husk and EDTA-modified cocoa pod husk) at pH 5, temperature 29°C, and initial metal ion concentration of 100mgL⁻¹. In a typical experiment, about 25mL of standard solutions of the metal ion was transferred into various flasks. Then, 0.5g of the adsorbent was weighed into different flasks and agitation was carried out at various contact times. Following each contact time, the content of each flask was filtered rapidly using the Whatman filter paper and filtrate collected. The equilibrium concentration of the filtrate of the metal ion was analyzed using Buck scientific model 210 VGP Atomic absorption spectrophotometer (AAS). Experimental readings were carried out in triplicates and average reading was used in the calculations. The amount of Pb²⁺ adsorbed (q_e) in mg/g for the metal ion solution by the various adsorbent at varying contact time was calculated using the mass balance equation as expressed below:

$$q_e = (C_0 - C_e)v/m \quad (1)$$

Where

q_e = amount of metal ion adsorbed in mg/g by the adsorbent at equilibrium

C₀ = initial metal ion concentration in mgL⁻¹

C_e = metal ion concentration at equilibrium in mgL⁻¹

V = volume of initial metal ion concentration used

m = mass of adsorbent used.

The effect of temperature on the sorption of Pb²⁺ by the adsorbents was also carried out by treating 0.5g of the sorbent with 25mL of 100mgL⁻¹ Pb²⁺ at pH 5. The flasks containing the adsorbate and adsorbent were introduced into a thermostated water bath at varying temperatures for 50 minutes. The solutions were then filtered and equilibrium concentration analyzed spectrophotometrically.

RESULTS AND DISCUSSION

Adsorbent surface characteristics

The surface characteristic of the adsorbents is shown in Table 1. The particle size of the adsorbents which were chosen at the beginning of the study remained the same. The surface charge density, specific surface area, pore volume of the modified husk were higher than the unmodified. The observed increase is as a result of the modification of the adsorbent with EDTA enhances the

chelating ability of the adsorbent as this helped to increase the available binding sites.

TABLE 1. Surface characteristics of unmodified and EDTA-modified cocoa pod husk

Parameter	Unmodified CPH	Value EDTA -modified CPH
Particle size (μm)	250	250
Bulk density (g/cm^3)	0.072	0.098
Pore volume (cm^3)	3.9	4.6
Specific surface area (m^2/g)	2.3 E26	2.48 E26
Surface charge density (meq/m^2)	6.2 E24	7.1 E24
Porosity (%)	19.98	23.01
CEC ($\text{mg}/100\text{g}$)	56.3	61.4

Sorption characteristics

The variation in the amount of Lead (II) ion uptake by unmodified and EDTA-modified cocoa pod husk is shown in Figure 1. From the figure, it is obvious that there was sharp increase in the adsorption of the metal ion at short contact time for the adsorbents. For unmodified adsorbent, a maximum of 94.6% was achieved at 50 minutes while the modified had 98.2% uptake also at the same time period. The implication of this is that modification of adsorbent significantly impacted on the sorptive characteristics of cocoa pod husk. The observed trend may be explained by the fact that a large number of vacant surface sites are available for the adsorption during the initial stage and with the passage of time, the remaining vacant surface sites are difficult to be occupied due to repulsive forces between the solute molecules on the solid phase and in the bulk liquid phase.

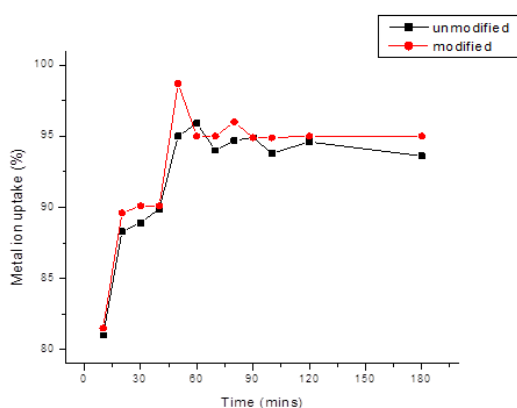


Figure 1. Changes in Pb (II) ion adsorbed against contact time on unmodified and modified CPH.

Adsorption kinetics was investigated for better insight into the dynamics of adsorption of Pb^{2+} onto modified CPH and to predict models that will allow the estimation of the amount of metal ion adsorbed with respect to time. The kinetic of Pb^{2+} adsorption on the

adsorbents (modified and unmodified CPH) was analyzed using Pseudo-first order, Pseudo-second order and intraparticle diffusion models. Conformity between experimental data and the model predicted values was expressed by the correlation coefficients, (R^2) which may be close or equal to 1.

Pseudo first order kinetic model: Pseudo first order kinetic equation is generally expressed:

$$\text{Log}(q_e - q_t) = \text{log}(q_e) - (k_1/2.303)t \quad (2)$$

Where: q_e and q_t are adsorption capacity at equilibrium and time t , respectively ($\text{mg}\cdot\text{g}^{-1}$).

k_1 = the rate constant of Pseudo first-order (min^{-1}).

The values of $\text{log}(q_e - q_t)$ were linearly correlated with t . A plot of $\text{log}(q_e - q_t)$ versus t should give a linear relationship from which k_1 and q_e can be determined from the slope and intercept of the plot [20]. The constants for Pseudo first order and correlation coefficient are shown in Table 2. From the table, it is obvious that the value of q_e of modified CPH is higher than the unmodified, however it does not successfully describes the kinetic data in the whole data range of the adsorption process.

Pseudo-second order equation: The sorption data was also analyzed in terms of a pseudo second order mechanism using the equation below:

$$(t/q_t) = 1/k_2q_e^2 + (1/q_e)t \quad (3)$$

Where:

k_2 = rate constant of the Pseudo second-order ($\text{g}\cdot\text{mg}^{-1}\cdot\text{min}^{-1}$).

The product of the pseudo second order rate constant, k_2 and the square of the equilibrium quantity q_e is defined as h_0 , the initial adsorption rate ($\text{mg}\cdot\text{g}^{-1}\cdot\text{min}^{-1}$). Equation becomes:

$$t/q_t = 1/h_0 + t/q_e \quad (4)$$

The plot of (t/q_t) and t should give a linear relationship from which q_e and k_2 can be determined

from the slope and intercept of the plot, respectively [21]. From the result (Table 2), the value of K_2 , q_e and R^2 of modified adsorbent are higher than for the unmodified. The initial rate of adsorption (h_0) for the modified husk is higher than for the unmodified. This implied that the obtained R^2 value range of 0.9776 to 1 provides a better fit for the sorption process than the Pseudo first order model.

Intraparticle diffusion model: The mechanism of sorption is either film diffusion controlled or particle diffusion controlled. Prior to adsorption, several diffusion process known to affect adsorption takes place. The sorbate will have to diffuse through the bulk of the solution to the film surrounding the sorbent and then into the micropores as

well as macropores. Models developed for the study of mechanism of sorption through intraparticle diffusivity include:

McKay and Poots model: This model, which was developed by [22], observed that the fraction of solute adsorbed can be expressed in terms of square root of time as given in the equation below:

$$q_t = X_i + K t^{0.5} \quad (5)$$

Where

X_i = boundary layer diffusion effects (mgg^{-1}),

K = rate constant for intraparticle diffusion ($\text{mgg}^{-1}\text{min}^{-0.5}$)

A plot of q_t versus $t^{0.5}$ should give a straight line with slope, K and the extrapolation of the linear plot to the time gives the intercept of the plot, X_i (mgg^{-1}) which is proportional to the boundary layer thickness. The kinetic parameters as well as R^2 values for McKay and Poots for this study is shown in Table 2. The boundary layer provides insight into the ability of metal ion uptake to the adsorbent phase or remain in solution. The result showed that Pb^{2+} ions had highest value for X_i for modified adsorbent than for unmodified. Since diffusion takes place, the boundary layer is looked upon as a viscous drag that exists between the adsorbent surface and the metal ion solution across its surface. It is however reported [12],[23] that at elevated temperature, the thickness of the boundary layer decreases due to increased tendency of the metal ion to escape from the adsorbent phase to the solution phase, and this results in a decrease in adsorption as temperature increases. As shown in the table, the boundary layer thickness varies from 94.51 to 98.99 mg/g thus indicating high tendency for adsorption capacity. The R^2 value for the modified adsorbent is close to 1, however the unmodified sorbent showed low R^2 value. Generally, it can be concluded that the model fits the experimental data, thus confirming that the process of adsorption is intraparticle diffusion controlled.

Thermodynamics of adsorption

To investigate the thermodynamic behavior of the adsorption of Pb (II) ions onto modified CPH, thermodynamic parameters such as change in free energy (ΔG°), change in enthalpy (ΔH°), and change in entropy (ΔS°) of the adsorption process were estimated. The adsorption of Pb^{2+} onto the adsorbents can be regarded as a heterogeneous and reversible process when equilibrium is reached. The apparent equilibrium constant for the process can be shown [24], [25] to be:

$$K_c = C_{ad}/C_e \quad (6)$$

Change in Gibbs free energy of adsorption process is as follow:

$$\Delta G^\circ = -RT \ln K_c \quad (7)$$

TABLE 2. Kinetic models constants for the adsorption of Pb^{2+} by virgin and EDTA –modified Cocoa pod husk

Model	Unmodified CPH	EDTA- modified CPH
Pseudo- first order		
K_i (min^{-1})	0.0295	0.0311
q_e (mg/g)	18.4	23.9
Correlation coefficient R^2	0.544	0.6513
Pseudo-second order		
K_2 (g/mg min)	0.146	0.119
q_e (mg/g)	76.01	92.09
h_o (mg/g min)	122	173
Correlation coefficient R^2	0.9776	1.000
Intraparticle diffusion		
X_i (mg/g)	94.51	98.99
K_p ($\text{mg/g min}^{0.5}$)	0.0014	0.0028
Correlation coefficient R^2	0.0174	0.9668

Where ΔG° = standard Gibbs free energy change of adsorption

R = universal gas constant ($8.314 \text{ Jmol}^{-1}\text{k}^{-1}$)

T = temperature (K)

From thermodynamics,

$$\Delta G^\circ = \Delta H - T\Delta S \text{ or } \Delta G^\circ = - \Delta(T) + \Delta H \quad (8)$$

A plot of T against ΔG° gives a straight line with slope $-\Delta S$ and intercept of ΔH .

For this study, a plot of T against ΔG° is shown in Figure 2. From the plot, a slope of $-212.44 \text{ Jmol}^{-1}\text{K}^{-1}$ was obtained and an intercept value of 60.36 kJmol^{-1} . This therefore implied that the entropy is $212.44 \text{ Jmol}^{-1}\text{K}^{-1}$ while the enthalpy is 60.36 kJmol^{-1} . It could be seen that the value of the free energy decrease with increase in temperature for the removal of Pb ions. The implication of this is that the adsorption of Pb ions onto modified CPH is endothermic in nature. The negative value of ΔG° is an indication of the spontaneity of the adsorption process. The positive value of standard entropy change, ΔS° , shows increased randomness at the solid/solution interface occurring in the adsorption process reflecting the affinity of modified CPH toward the metal ions. It has been reported that the change of standard free energy for physisorption process is in the range of -20 to 0 kJmol^{-1} while chemisorptions process varies from -80 to -400 kJmol^{-1} [26], [27]. For the present study, the overall free energy change obtained for the adsorption of Pb ions onto modified CPH is $-5.499 \text{ kJmol}^{-1}$ at 310K . This large negative value further confirmed the fact that the adsorption of Pb ions onto CPH is physisorption in nature.

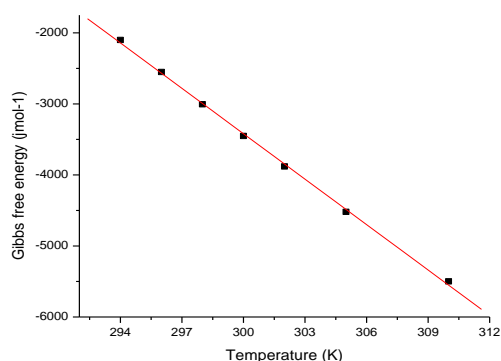


Figure 2. Change in Gibbs free energy with temperature of Pb^{2+} adsorption by modified CPH.

CONCLUSION

The present investigation showed that EDTA –modified CPH is an effective adsorbent for the removal of $Pb(II)$ in aqueous solution. Increase in surface charge density, specific surface area, pore volume of the modified husk over the unmodified was observed. This is as a result of the modification of the adsorbent with EDTA which enhances the chelating ability of the adsorbent as this helped to increase the available binding sites. Kinetic and thermodynamic parameters indicated that modified CPH is a promising adsorbent for the removal of $Pb(II)$ containing effluents.

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Persian Abstract

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چکیده

جذب سطحی یونهای سرب روی پوسته کاکائو EDTA اصلاح شده در یک سیستم تعادلی بررسی شد. سنتتیک، مکانیسم و پارامترهای ترمودینامیکی تخمین زده شد. توانایی جذب سطحی سریع بود و وابستگی آن به زمان همانند جذب Pb (II) قابل تحسین بود و زمان بهینه تماس در ۵۰ دقیقه بدست آمد. ۳ مدل سنتتیک برای تطبیق دادن داده های سنتتیک مورد استفاده قرار گرفت؛ شبه نوع اول، شبه نوع دوم و نفوذ درون ذره ای. جذب سطحی یون های فلزی از سنتتیک شبه درجه دو و درون ذره ای پیروی می کنند چون تطابق بهتری را با داده های تجربی با مقدار R^2 بالا بین ۰٫۹۷۷۶ تا ۱ را می دهند. پارامترهای ترمودینامیکی مثل تغییر در انرژی آزاد گیبس، تغییر در آنتالپی و تغییر در آنتروپی محاسبه شدند. $[\Delta G]^{(1)} = -5.499 \text{ kJ}[\text{mol}]$ و $[\Delta H]^{(1)} = 60.36 \text{ kJ}[\text{mol}]$ و $[\Delta S]^{(1)} = 212.44 \text{ J}[\text{mol}]^{-1} \text{ K}$ فرآیند جذب سطحی یون های فلزی روی جاذب اصلاح شده، امکان پذیر، گرماگیر، خودبه خودی و جذب فیزیکی در طبیعت بدست آمد. این نتایج بیان می کنند که CPH اصلاح شده یک جاذب کاربردی برای حذف Pb(II) از فاضلاب های صنعتی و پساب ها می باشد.