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RESEARCH ARTICLE

STUDIES ON BIO-CHEMICAL THERMODYNAMICS OF LEAD BIOSORPTION FROM AQUEOUS SYSTEM USING CORN HUSK BIOMASS AS BIOSORBANT AGENT

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ABSTRACT

In our study, biosorption characteristics of Pb (II) ions from aqueous solution using corn husks biomass were investigated using batch techniques process. The effects of pH, contact time, initial metal ion concentration, and temperature were also studied. The FTIR study showed that the several functional groups such as O-H, C=O, C-O, C-H and N-H were more involved in binding Pb (II) ions to the biomass. The equilibrium datawere analyzed by the Langmuir, the Freundlich and the Temkinisotherms equations model. It was found that adsorption of Pb (II) ions onto corn husks, was best described by Freundlich adsorption model. Our biosorption kinetics data were fitted using pseudo-first-order and pseudo-second-order models, and it was found that the kinetics data fitted into the pseudo-second order model more perfectly. Thermodynamic parameters such as standard Gibbs free energy (ΔG^0), standard enthalpy (ΔH^0) and standard entropy (ΔS^0) were also evaluated simultaneously. The obtained result showed that biosorption of lead (II) ions bycorn husk was spontaneous, endothermic in nature and it can be used as a potent bioremediation agent.

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INTRODUCTION

Environmental pollution by metal ions in industrial waste waters is one of the most important causes of water contamination which can accumulate throughout the food chain, and in environment, due to their great persistence and high toxicity (Ahluwalia *et al.,* 2005). For the removal of heavy metal from contaminated waste water, there are having several conventional methods such as chemical precipitation, membrane separations, evaporation, resin ion-exchange, electro-winning and reverse osmosisetc (Kilic *et al.,* 2008). However, among these conventional methods of removing heavy metals from effluents require high energy and these processes are incapable to remove complete metal in large quantity from toxic waste sludge. Moreover, these technologies are well accepted in situations where the concentrations of heavy metal ions are relatively high in contaminated sites. To compare with conventional processes, new methods must be accepted as these are economically viable process and which can also be successful in the removal of environmental contaminantin form of toxic heavy metals (Ho *et al.,* 1998). Biosorbents, using biological agents, are alternatives to conventional methods. The term 'biosorbent' includes the usage of dead biomass such as agricultural by-products (Ahluwalia *et al.,* 2005). Agricultural by-products usually are composed of lignin and cellulose and these are as major constituents and may also include other polar functional groups of lignin, which includes alcohols, aldehydes, ketones, carboxylic, phenolic and ether groups, which gives them more advantages in biosorption process. These functional groups have the tendency to bind heavy metals by the replacement of hydrogen ions for metal ions in aqueous solution (Ofomaja *et al.,* 2007). Corn husk is the waste material after the corn is removed. In several areas where corn is used as one of the major food grain, the husk often creates environmental nuisance. The aims of this study therefore was to investigate the possible use of a corn husk as an alternative biosorbent material for removal of Pb (II) ions from aqueous system. The dynamic behavior of the adsorption was also investigated on the effect of the pH of the solution, initial metal ion concentration, contact time, and temperature.

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MATERIALS AND METHODS

Preparation of biomass: Corn husks were collected from a local market (Bengaluru) in large quantity which to be used as biosorbent for the research studies. The corn husks were then oven-dry, at 60^0C for 24 h. The dried corn husks were ground and then sieved using a 150 size mesh screen. The sieved corn husk was then packed in polythene bags (2g each) until when required for the experiments (Ofomaja *et al.,* 2007).

Fourier-transform infrared spectroscopy of corn husk: The IR spectra of the free and metal-adsorbedbiosorbents were run as KBr pellets on impact 410 Nicolet FTIR spectrometer in the frequency range 4000 - 500 cm^{-1}. Finelycorn huskbiomass (10mg) was mixed withKBr(500mg) and the two were ground together to prepare a translucent sample disk (Ofomaja *et al.,* 2007).

Preparation of Pb (II) solutions: $Pb(NO_3)_2$, NaOH and HNO_3 used are analytical grades purchased from Merck Schuchardt, Germany. Stock Pb (II) solution (1000 mg/L) was prepared by dissolving $Pb(NO_3)_2$ (1.6 g) in deionized water (100 mL) and diluting quantitatively to 1000 mL using deionized water. Pb(II) solutions of different concentrations were prepared by adequate dilution of the stock solution with deionized water. The pH of the solution was adjusted with HNO_3 and NaOH solutions for pH studies. All the glassware used were overnight immersed in 10% (v/v) nitric acid and rinsed several times with deionized water before drying (Ho *et al.,* 1998).

Effect of pH on adsorption of Pb (II) ions: The influence of pH on the biosorption process was investigated by adjusting the Pb (II) solution to a desired value within the range 1.0 to 8.0 with HNO_3 or NaOH. The metal ion solution (25mL; 100 mg/L) was then contacted with a fixed quantity of corn husk (0.5g, dry weight) in a boiling tube. The mixture was left in a water bath to maintain the constant temperature of 28 °C for 24h with constant shaking at 75 rpm. The biosorbent was then removed from the mixture by filtration through a Whitman 8 filter paper. The concentration of the metal ions remaining in solution, were then determined using Atomic Absorption Spectrophotometer (Buck 200A Analytical Model) at 283.2nm. The experiments were performed in triplicates. The percentage removal of Pb (II) ions by the corn husk was calculated from the following equation:

$$Biosorption(\%) = \frac{C_o - C_e}{C_o} X100 \ldots\ldots\ldots\ldots\ldots\ldots 1$$

where C_o and C_e are initial and equilibrium metal ion concentrations (mg/L) in aqueous solution (Ho *et al.,* 1998).

Effect of initial metal ion concentration on adsorption of Pb (II) ions: Batch adsorption studies were carried out using initial metal ion concentrations of 25, 50, 100, 150, 200 and 400 mg/L in order to test for the effect of initial metal ion concentration on adsorption of Pb (II) ions. A fixed quantity of corn husk (0.5g) was packed into boiling tubes, and the desired metal ion solution (25 mL) was added. The mixture was left in a water bath while maintaining constant temperature of 28 °C for 1h with constant shaking at 75 rpm. The biosorbent was then removed from the mixture by filtering through a Whatmann 8 filter paper. The concentration of the metal ion remaining in solution was then determined using Atomic Absorption Spectrophotometer (Buck 200A Analytical Model) at 283.2nm.The amount of metal ions adsorbed by the biosorbent was then calculated as follows:

$$q_e\left(\frac{mg}{g}\right) = \frac{(C_o - C_e)V}{1000W} \ldots\ldots\ldots\ldots\ldots\ldots 2$$

Where q_e is the amount of metal ion adsorbed on the biosorbent in (mg/g), V is the volume of metal ion solution used (mL) and W is the weight of the biosorbent used (g).The data obtained were then used to test the fitness of the sorption process to Langmuir, Freundlich and Temkin isotherms.

Effect of contact time on adsorption of Pb (II) ions: The adsorption of Pb (II) ions onto corn husk powder was studied at various time intervals. A constant concentration of 100mg/L was used. A fixed quantity of the biosorbent (0.5g) was weighed into various boiling tubes, and the metal ion solution (25mL) was added into each boiling tube and its content. The biosorbent was then filtered at different time intervals of 10, 20, 30, 45, 60, 74, 90, 120, 150 and 180 min. The filtrates were then analyzed with AAS, to determine the amount of metal ion remaining in solution. All experiments were performed in triplicates. The data obtained were then used to determine the values of k_1 and k_2 in the Langergren and Ho's pseudo first order and pseudo second order equations respectively.

Thermodynamic studies: For thermodynamics study, experiments were carried out at 35, 40, 50, 60 and 70^0Cusing initial metal ion concentration of 100mg/L. Each experiment was carried out for 1 h with triplicate determinations. The filtrates were decanted and analyzed with AAS to obtain the concentration of the metal ions remaining in solution.

RESULTS AND DISCUSSION

The FTIR study: The details of the assignment of bands due to functional groups present on the surface of the biomass before and after biosorptionare mentioned in Table 1.

Table 1. The FTIR spectra characteristics of corn husk before biosorption and corresponding possible groups

Only Corn husk (cm^{-1})	Corn husk with Pb(II)(cm^{-1})	Vibration type
3388	3394	Carboxylic/OH
3123	3123	C-H stretch
3070	3069	Aromatics/C-H stretch
2929	2929	C-H saturated
1639	1645	Carboxylic/C=O
1509	1523	N-H stretch
1450	1450	C-H stretch
1262	1382	-C-O stretch
1038	1056	-C-O stretch
873	873	Phosphoryl groups
785	785	C-H out of plane

Different adsorption mechanisms including complexation, ion exchange, and electrostatic attraction may be involved in the biosorption process and it is suggested that these processes depend on the functional groups on the surface of the biomass. In our experiment, the broad peak around 3388 cm^{-1} was assigned to the presence of –OH or -NH group on corn husk powder surface, 1262 cm^{-1} shows C-O groups, 1639 cm^{-1}

shows C=O, the peak at 2929 cm^{-1} is due to C-H vibration of CH_3 groups, 3388 cm^{-1} and 3070 cm^{-1} show the vibration of the aromatics. O-H,C=O, C-O, C-H and N-H were identified to be involved in Pb (II) ions binding to the corn husk powder surface due to the shift in the spectra band after adsorption (Chauhan *et al.,* 2004).

The effect of pH on the adsorption of Pb (II) ions: Acidity of the solution one of the most important factors which may affect the biosorption of metal ions .The acidity of the medium might show the competition between hydrogen ions and metal ions for the active sites on the biosorbent surface (Chauhan *et al.,* 2004). The solution of pH affects the metal binding sites of cell wall and the metal ion chemistry too. The effect of initial pH on biosorption is clearly mentioned in Fig. 1.

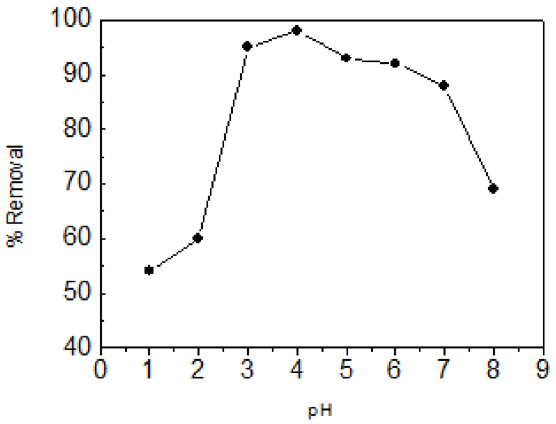

Figure 1. Graph showing the effect of pH on the sorption of Pb(II) ions aqueous solution using Corn husk. Initial lead concentration (100 mg/L).0.5g dry weight biomass with25 mL of Pb (II) Solution

In the present study, pH used from 1-8. The minimal adsorption capacity (54%) was at pH 1 and the trend increased progressively until it reached 98 % at pH 4.It was observed here that the adsorption capacity decreased progressively above pH 4. The FTIR study showed that corn husk powder surface contains acidic functional groups. It is understood that at lower pH (pH $\leq$ 2.0), the dissociation of the acidic functional groups like carboxylic acids on the surface of the adsorbent did not take place. However, due to the increased pH, the acidic functional groups might bedeprotonated. The deprotonation of the acidic functional groupshelp to increase the negative charges on the adsorbent materials (Lawal *et al.,* 2010).This clearly indicate that below pH 3, the overall surface charge on corn husk was less negative when compared with surface charge at higher pH. Higher number of negative charges on the corn husk surface help in the adsorption process of the metals ions, but when the negative charges reduced due to further increase of pH above 4, it denotes hydrolysis of lead ions (Conrad *et al.,* 2007 and Khalid *et al.,* 1998). Our observations are strongly supported by several previous studies on the biosorption of Pb (II) ions from aqueous system (Kilic *et al.,* 2008, Lawal *et al.,* 2010).

Effect of initial metal ion concentration on the adsorption of Pb (II) ions: The initial metal ion concentration plays a vital role in the biosorption process. The biosorption characteristics of corn husk, in the removal of Pb (II) ions increased, with increasing metal ion concentration (Fig. 2). This indicates that the removal of Pb (II) ions from its solution by corn husk at higher concentration will most likely be suitable choice. Similar observations were explained by Khalid *et al.,* 1998 and Kilic *et al.,* 2008. It is very obvious that, the increase in initial metal ion concentrations affect the mass transfer of Pb(II) ions between the aqueous and solid phase and this implies a higher probability of collision between metal ions and sorbents agents (Kilic *et al.,* 2008). When lead concentration increases, showed an improved probability of collision between metal ions and sorbents agents.

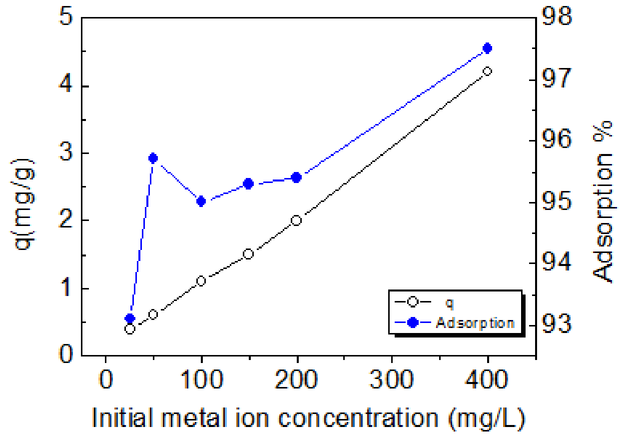

Figure 2. Effect of initial metal ion concentration on biosorption of Pb (II) ions aqueous solution using Cornhusk. 0.5g dry weight biomass with25 mL of Pb (II) Solution. pH4

Effect of contact timeon the adsorption of Pb (II) ions: The time variation for the adsorption Pb (II) ionsinto corn husk showed that more than 80% of Pb (II) ions were adsorbed within the first 10 min, whereas theprocess of sorption continued up to 3h (Fig. 3). Initial lead concentration used 100 mg/L and biomass used in form of dry mass 0.5g, with25 mL of Pb (II) Solution having pH4.It was found that the sorption process reached equilibrium after 60min, and that time almost 96% of Pb (II) ionshave been adsorbed by the corn husk biomass. The observed initialbiosorption kinetics is consistent with the biosorption of metal involving non-energy mediated reactions, where metal removal from solutions is because of purelyphysico-chemical interactions between the biomass and the metal solution. Similar results have been reported for the biosorption of lead from solution using rice husks and *Syzygiumcumini* respectively (Lawal *et al.,* 2010 and Chauhan *et al.,* 2004).

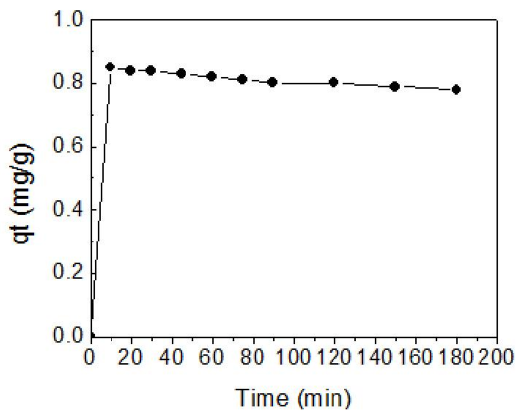

Figure 3. Time dependent study of the sorption of Pb (II) ions on Corn husk

Effect of biosorbentconcentrationon the adsorption of Pb (II) ions: The effect of biosorbentconcentrationon the adsorption Pb (II) ions is indicated in Fig.4. The nature of graph stated a progressive increase in the amount of lead adsorbed as adsorbent dosage increased from 0.1 to 0.5g. The percentage of lead removed increased from 81 to 96. It is very obvious that increasing the biosorbent dose increased the surface area which helps to providing increase available active sites for the corn husk. Similarobservation was also found for lead removal using *Moringaoleiferal* bark (Khalid *et al.,* 1998).

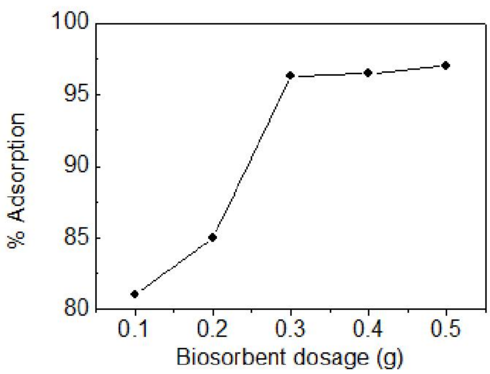

Figure 4.Effect of adsorbent dose for the adsorption of Pb (II) ions onto Corn husk. 100 mg/L lead concentration

Adsorption isotherms: To understand the relationship between the adsorbed amount of lead and its equilibrium concentration in solution, Langmuir, Freundlich and Temkin adsorption isotherms were applied.

Langmuir Model: Langmuir model suggests monolayer sorption on a homogeneous surface without interaction between the sorbed molecules. It correlates the coverage of molecules on a solid surface to concentration of a medium, when the solid surface at fixed temperature. It is represented in the linear zed form of graph as shown below (Langmuir, 1918);

$$\frac{1}{q_e} = \frac{1}{q_m K_L}\left[\frac{1}{C_e}\right] + \frac{1}{q_m} \ldots\ldots\ldots\ldots\ldots\ldots\ldots 3$$

where, q_e is the heavy metal adsorbed on the biosorbent (mg/g dry weight), C_e is the final concentration of metal (mg/L) in the solution, q_m is the maximum possible amount of metallic ion adsorbed per unit weight of adsorbent; K_L is the equilibrium constant related to the affinity of the binding sites for the metals. The adsorption constants (q_m and K_L) were obtained by plotting $1/q_e$ against $1/C_e$giving a straight line with a slope of ($1/K_L q_m$) and an intercept of ($1/q_m$) (Fig. 5).

Freundlich Models: The Freundlich isotherm model indicates that the uptake of metal ions occurs on a heterogeneous surface by multilayer adsorption and that the amount of adsorbed increases infinitely, with an increase in concentration (Freundlich, 1907). This model further assumes that in absorbantagetns, the stronger binding sites are occupied first and that the binding strength decreases slowly, with increasing degree of site occupation .It is represented in the liberalized form as shown below;

$$\log q_e = \log K_F + \frac{1}{n}\log C_e \ldots\ldots\ldots\ldots\ldots\ldots 4$$

The adsorption constants (1/n and k_f) were obtained from the graph, by plotting log q_e against log C_e from the slope and the intercept respectively(Fig. 6).

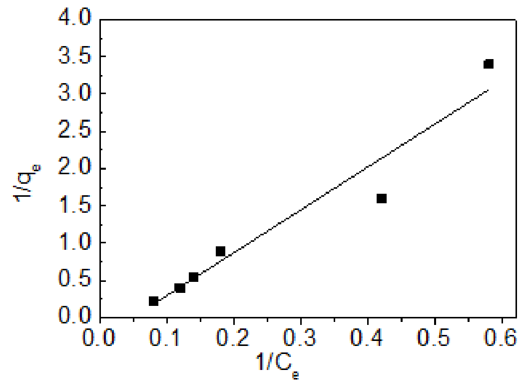

Figure 5. Langmuir isotherm for sorption of Pb (II) ion by Corn husk.0.5g dry weight biomass with25 mL of Pb (II) Solution. pH 4

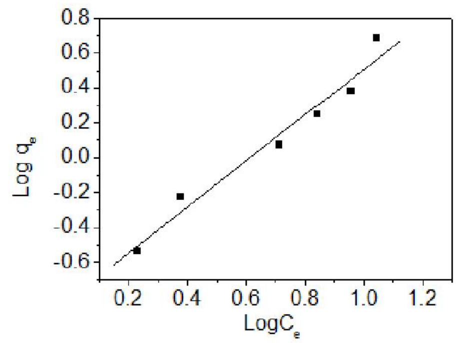

Figure 6: **Freundlich isotherm for sorption of Pb (II) ion by Corn husk. 0.5g dry weightbiomass with25 mL of Pb (II) Solution.pH4**

Temkin Model: Temkin isotherm can be expressed in the linearized form as:

$q_e = B \ln K_T + B \ln C_e$………………5

A plot of q_e versus $\ln C_e$ enables the determination of the isotherm constants B and K_T from the slope and the intercept, respectively (Fig. 7).

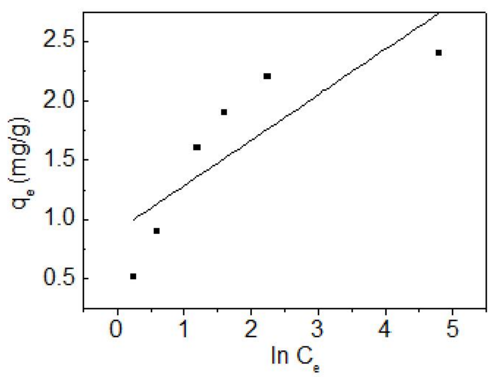

Figure 7. Temkin isotherm for sorption of Pb (II) ion by Corn husk. 0.5g dry weight biomass with25 mL of Pb (II) Solution.pH4

Temkin constants are mentionedin Table 2. K_T is the equilibrium binding constant corresponding to the maximum binding energy and constant B is related to the heat of adsorption. The equilibrium data, in Table 2, indicates the values of regression coefficients R^2 between the sorbate and sorbent systems for Langmuir, Freund lich and Temkin models. The best way of description of the adsorption process of Pb (II) ions was observed in Freund lich model based on the regression coefficientsR^2=0.964 compared with 0.957 for Langmuir and 0.719 for Temkin isotherms. Possibly the adsorption process started with the initial coverage as monolayer on the outer surface of the adsorbent agents, after which the sorption continued to the multilayer dimensions.

The pseudo-second-order kinetic model (McKay and Ho, 1999) in its integrated and linearized form has been used and is given as follows:

$$\frac{t}{q_t} = \frac{1}{kq_e{}^2} + \frac{t}{q_e} \text{..................7}$$

Wherek_2 is the rate constant of second-order adsorption. The plot t/q_t versus t gave q_e and K_2 (Fig. 9) which were found out from slope and intercept of the plot, respectively. The pseudo-first-order kinetic model give a poor description of the data for the biosorption of Pb(II) ions, this is indicating their correlation coefficient (R^2) values in Table 3.

Table 2. Model parameters for the biosorption of Pb(II) ions on Corn husk

Freundlich Model				Langmuir model				Temkin Model			
K_f	R^2	n	S.D	K_L	R^2	q_m	S.D	T	R^2	B	S.D
0.158	0.967	0.768	0.094	0.057	0.956	3.034	0.291	10.362	0.793	0.384	0.446

Table 3. Comparison of Pseudo-first and second-order biosorption of Pb(II) ions on Cornhusk

Pseudo-First- Order				Pseudo-Second- Order			
K_1	R^2	q_e	S.D	K_2	R^2	q_e	S.D
0.013	0.784	232	0.1136	-0.682	0.0997	0.974	0.4434

Kinetics of adsorption: The efficiency of sorption can define with its kinetics. Different biosorbents fit to different models. In this case, the Lagergren first-order and pseudo-second-order models were used to check the adsorption kinetics data in terms to investigating the mechanism of biosorption. The Lagergren rate equation is the most widely used model for the sorption of a solute from a liquid solution and the first-order, rate expression of Lagergren is as follows (Lodeiro *et al.,* 2006 and Low *et al.,* 2000).

$$\ln(q_e - q_t) = \ln q_e - k_1 t \text{..................6}$$

Here, q_e and q_t (mg/g) are the amounts of lead adsorbed on the corn husk at equilibrium and time t, and K_1 is the rate constant of first-order kinetic. The slope and intercept of the plot of log (q_e - q_t) versus t for corn husk was used to determine the values of q_e and K_1 (Fig. 8).

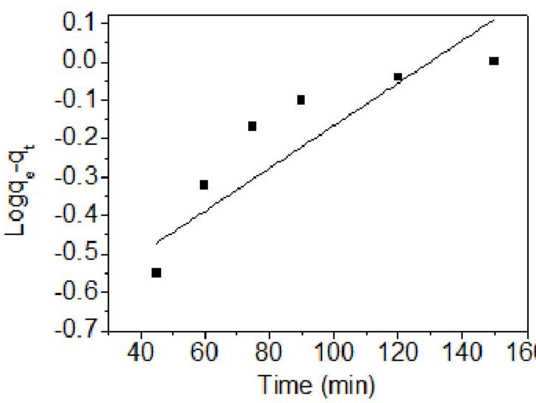

Figure 8. Pseudo-first order graph for sorption of Pb (II) ions on Cornhusk

In previous studies, where the pseudo-first-order kinetic model not able to correlate with experimental values, this was predicted to a time lag which probably caused the presence of the boundary layer or external resistance which was controlling at the beginning of the sorption process.

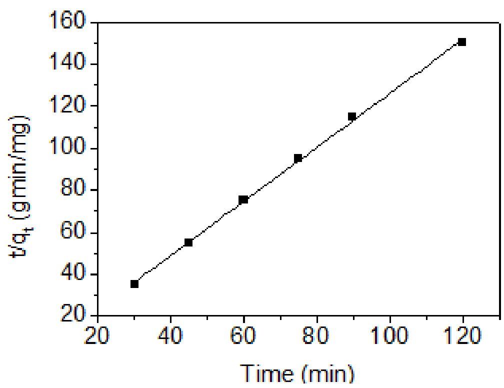

Figure 9: Pseudo-Second order graph for sorption of Pb (II) ions on Cornhusk

To understand the kinetic behavior of the biosorption with chemical sorption being the rate-controlling step, the pseudo-second-order kinetic modelis best one. The linear plot of t/q_t versus t for the pseudo-second-order model for the biosorption of Pb (II) ions onto corn husk is indicated in Fig. 9. It is clear from our data in Table 3 that the R^2 value for the pseudo-second-order model is high, which suggests that the biosorption of Pb (II) ions onto corn husk definitely follows the pseudo-second-order kinetic model. Several researcher those have studied that sorption of divalent metals on various heterogeneous sorbents had mentioned that most metal sorption kinetic follow pseudo-second-order mechanism, which is supporting our result (Ofomaja *et al.,* 2007 and Reddad *et al.,* 2002).

Thermodynamics study: The result obtained(Fig 10)from varying temperature within the range of 308 - 343 K, showed slowly increase in metal uptake as temperature increased Similar increase in metal uptake with increasing temperature had been reported earlier by (Ofomaja *et al.,* 2007 and Reddad *et al.,* 2002). The increase in adsorption with increasing temperature, indicated adsorption process has endothermic nature. It may be concluded to either the increase in the number of active surface sites available for sorption on the adsorbent agents or it may due to the decrease in the boundary layer thickness surrounding the sorbent, which leads to the mass transfer resistance of adsorbate due to decreased in boundary layer. In order to describe the thermodynamic nature of the biosorption of Pb(II) ions onto corn husk, several basic thermodynamics parameters including the change in free energy (ΔG^0), enthalpy (ΔH^0) and entropy (ΔS^0) were calculated from the following equations

ΔG^0 = -RT ln K……………..8

Here, T is temperature in Kelvin (K), R is ideal gas constant having value as 8.314 J $mol^{-1}K^{-1}$ and Kis thermodynamic equilibrium constant. The relationship between Gibbs free energy change, entropy change (ΔS^0) and enthalpy change (ΔH^0) can be expressed as:

$\Delta G^o = -RT \ln K$……………9

From the two equations above, we can write:

$\ln K = \frac{\Delta S^o}{R} - \frac{\Delta H^o}{RT}$……………………10

According to this equation, the ΔH^0 and ΔS^0 can be calculated from the slope and intercept of plot of ln K versus I/T (Fig. 10) respectively.

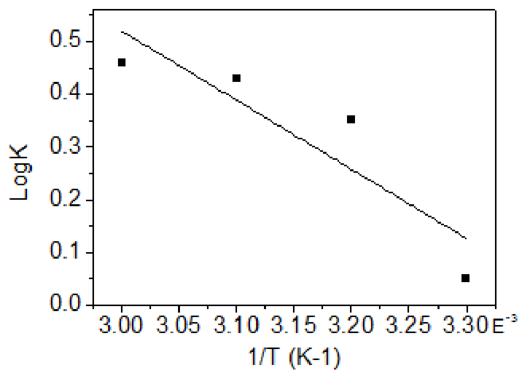

Figure 10. Thermodynamic profile of sorption of Pb(II) ion by Corn husk

The thermodynamic equilibrium constant was determined as follows:

K = C_a/ C_e……………11

Where, C_a is amount (mg) of the adsorbate adsorbed per litre, and C_e is the equilibrium concentration of solution in mg/L. The negative ΔG^0 values indicate thermodynamic feasibility and spontaneity of the biosorption process. The increase in the magnitude of ΔG^0 values for Pb (II) biosorption with increase in temperature shows aproper increase in its feasibility. The slight decrease in the value of ΔG^0 in the biosorption of Pb (II) ions by corn husk at 343 K may be due toactive binding sites in the biomass gradually got damage of (Ofomaja *et al.,* 2007 and Reddad *et al.,* 2002) or the tendency was to oppose metal ions absorption from the interface to the solution. The ΔH^0 value for Pb(II) ions was calculated and found to be 25.35 $kJmol^{-1}$, which reveals that the binding of Pb(II) ions onto corn husk was endothermic in nature. This enthalpy change supported by previous researchers in their literature. The magnitude of ΔH^0 value gives an indication of the type of adsorption, which can either be physical or chemical. The physical adsorption can define the heat of adsorption, ranging from 2.1 – 20.9 $kJmol^{-1}$, and the activation energy for chemical adsorption is of the same magnitude as the heat of chemical reactions, 20.9 – 418.4 kJ mol^{-1}. The calculated ΔH^0 value in the biosorption experiment indicates that the binding of Pb(II) ions to the corn husk is by chemical adsorption mainly. Also, the calculated ΔS^0 was observed to be positive (86.1$kJmol^{-1}K^{-1}$) which indicates the increasing randomness at the solid and liquid interface during biosorption and supported by (Ho *et al.,*1999).

Conclusion

The present study uses the corn husk for the removal of heavy metal Pb (II) from the aqueous environment. Our results indicate that the pH, contact time, temperature and initial concentrations, affect the uptake of the metal ions by the corn husk. It was very clear that the adsorption equilibrium data fitted best into the Freundlich model, when compared with the Langmuir and Temkin isotherms model. The pseudo-second-order kinetic model supported the biosorption of Pb (II) with the help of corn husk. Therefore, the major strength of our study is that among waste materials, corn husk can be used to solve an important environmental pollution problem.

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