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Nickel (II) And Lead (II) Removal from aqueous solution using Immobilized *Lonchocarpus laxiflorus*, Barkstem as an Adsorbent

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Abstract

Batch adsorption techniques were used to study the biosorption of Lead and Nickel from aqueous solutions by immobilized *Lonchocarpus laxiflorus* stembark. The effects of optimum pH, contact time, metal ion concentration, biosorbent dose, solution temperature were investigated. Experimental results showed that *Lonchocarpus laxiflorus* stembark was effective in removing these metals from aqueous solutions as the equilibrium biosorption of both metals was attained within 20 minutes of interaction with 99% of the metals removed within this period. Sorption of these metals was dependent on pH as maximum removal was attained at pH 4 and pH 6 for Lead and Nickel ions respectively. Both the Langmuir and Freundlich isotherm models were suitable for describing the adsorption of Ni (II) and Pb (II) on *Lonchocarpus laxiflorus* stembark. The capacity of *Lonchocarpus laxiflorus* stembark for the adsorption of Ni (II) and Pb (II) was 19 and 19.54 mg/g respectively.The FTIR spectrum exhibited that *Lonchocarpus laxiflorus* stembark biomass structure have hydroxyl and carbonyl groups on their surfaces and the results obtained from SEM micrograph shows its surface exhibited a rough structure with irregular surface Fig 2.

Key words: Biosorption, Equilibrium isotherm, Immobilized *Lonchocarpus laxiflorus*, Pb(II) and Ni (II) removal.

Introduction

Water pollution is a major environmental problem faced by modern society that leads to ecological disequilibrium and health hazards. Heavy metal ions such as copper, cadmium, lead, nickel, and chromium, often found in industrial wastewater, present acute toxicity to aquatic and terrestrial life, including humans. Thus, the discharge of effluents into the environment is a chief concern. The rate at which effluents are discharged into the environment especially water bodies, has been on the increase as a result of urbanization, industrialization of many sectors such as food, pharmaceutical, leather, textile, cosmetics, paper, printing etc. and waste generated from these industries contain heavy metals which is the cause of the environmental contamination (Vijayaraghava *et al.*, 2004; Hasfalina *et al.*, 2012). Lead can cause several unwanted effects, including disruption of the biosynthesis of hemoglobin, anemia, rise in blood pressure, kidney damage, subtle abortions, birth defects, disruption of nervous systems, brain damage, decline fertility of men through sperm damage, diminished learning abilities of children, abdominal pains, adrenal insufficiency, blindness, cardiovascular disease, Liver dysfunction, and a host of others diseases (Volesky, B. 1993). The permissible limits for lead in drinking water being 0.01 mg/ (WHO, 2000).

Nickel also pose health hazard to certain individuals causing serious health problems including: anorexia, kidney dysfunction, apathy, disruption of hormone and lipid metabolism, muscle tremors, fever, intestinal cancer, heart attack, headache, nausea, oral cancer, skin problems and vomiting (Ajay *et al.*, 2005). World Health Organisation (WHO) and United states Environmental Protection Agency (USEPA) have established the maximum contamination limits (MCL) for Ni(II) in drinking water is 0.5mg/L.(WHO, 2006).

Biosorption of heavy metals is one of the most promising technologies involved in the removal of toxic metal ions from wastewater. It is a potential alternative to conventional processes for the removal of metals due to the low cost, easily obtained, minimization of the volume of chemical and/or biological sludge to be disposed of, high efficiency in detoxifying very dilute effluents and no nutrient requirements (Gadea-Torresdy *et al*., 2005).

A great interest has recently been directed to the biosorption of heavy metals from solutions using different bio- materials as adsorbents. Among the various resources in biological wastes, both dead and live biomass, exhibit particularly interesting metal-binding capacities. Biosorption, using waste biomass from other industries as sorbent materials, can be a cost effective metal removal technique. So far, no research has been performed on Farin Sansamii; inspite of their great potential as biosorbents. As a low cost, Farin Sansamii is an attractive and inexpensive option for the biosorption removal of dissolved metals. The use of conventional methods for the removal of heavy metal can be expensive, prohibitive for developing economics and most times do not work well for low concentration of the polluting metals (Gadea-Torresdy *et al*., 2005) thus it becomes imperative to search for alternatives.

Therefore, the use of plants which posses the natural ability to uptake heavy metals for the remediation of the environment is currently used. Besides plant biomass can effectively bind toxic metals and as such can be used to remove metals from solution (Babel *et al.*, 2003). The ability of plant materials to bind metals has been attributed to the presence of various functional groups such as carbonyl amino, sulphate and hydroxyl groups which can attract and sequester metal ions (Volesky, 2007). These functional groups pick up metal ions from solution through physio-chemical mechanism namely ion-exchange, complexation, coordination and physical adsorption (Gardea-Torresdey *et al.*, 1998a).

Various low cost adsorbents such as onion skin (Kumar *et al.*, 1981) tea leaves (Abdel-Ghani, *et al*., 2007) Black Oak bark, Red- wood bark, Chistosan, Rice hull, Zeolite, Bentonite, Xanthate, Sawdust, (Baily, *et al*., 1999) are known to adsorb heavy metal ions from solutions in their native state and with suitable chemical treatment, the adsorption capacity can be significantly enhanced .The aim of this work is to investigate the adsoption capacity of Lead and Nickel from aqueous solutions by immobilized lonchocarpus laxiflorus stembark biomass.

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Materials and Methods

Analytical reagents of grade chemicals obtained from the British Drug House (BDH,) and distilled water was used. The reagents used are Nickel sulphate $(NiSO_4).6H_2O$, Sodium alginate, calcium chloride, Metal ion solutions of different concentration were obtained by diluting the respective stock solutions ($1000mg/cm^3$). Standard acids and base solutions (0.1M HCl , 0.1M NaOH and 1M HNO_3) were used for pH adjustments. Morphology and characterization of powdered biomass were determined by using Sanning Electron Microscopy (SEM) and Fourier Transform Infrared Spectroscopy (FTIR)

Plant collection and treatment

The Stembark of *Lanchocarpus laxiflorus* plant were collected from a tree behind Modibbo Adama Federal University of Technology Yola, Nigeria. The plant bark was washed thoroughly under running water to remove dust and any adhering particle and then rise with distilled water. The sample was air dry for 2 weeks and the dry stembark was grinded in analytical mill and sieve to obtain adsorbent of known particle size range. The biomass powder was kept in an air tight bottle for further study (Igwe and Abia, 2006).

Preparation of sodium alginate and calcium chloride stock solution

Sodium alginate was prepared by weighing 4.00 g and making it up to 100 cm^3 mark with distilled water in a volumetric flask and left overnight for complete dissolution. 0.12 M of calcium chloride was prepared by weighing 26.28g in to 1000cm^3 volumetric flask and making up to mark with distilled water according to a standard procedure described by (Osemeahon *et al.*, 2012).

Immobilization of the Stem bark of *Lonchocarpus laxiflorus* Plant

Sodium alginate was used for immobilization of the Stem bark of *Lonchocarpus Laxiflorus* plant. 50 cm^3 of sample solution prepared by dissolving 4g of each test sample in 100 cm^3 of distilled water and mix with 50 cm^3 of 4% stock solution of sodium alginate and stir vigorously in 250 cm^3 beaker, to obtain a homogenous mixture. After mixing, the solution the solution was drawn through hyperdemic needles and was added drop wise to a stirred solution of 1M $CaCl_2$. A retention time of 1 h was allowed for the reaction to obtain complete precipitation of the immobilized leave powder of *lonchocarpus laxiflorus* plant. The beads thus formed i.e sodium alginates were kept in fresh $CaCl_2$ solution. Before sorption studies, the beads were removed and allowed to dry at room temperature. The dried solid mass was stored in a polythene bag for further use. (Mishra S. P. 2013).

Preparation of synthetic waste water

Nickel sulphate $Ni(SO_4).6H_2O$ and Lead(II) nitrate $Pb(NO_3)_2$ solution of 1000mg/cm^3 was prepared by dissolving 0.4478 and 1.6 gs of their respective salts in 250 cm^3 of distilled water and make up to 1000 cm^3 in a volumetric flask.The above stock solution was used to prepare working solution of various concentrations ranging from 10ppm to 100ppm with distilled water, using the formula

$$C_1V_1=C_2V_2$$

Where C_1 is the concentration of the stock solution, V_1 is the volume of the stock solution, C_2 is the concentration of the dilute solution and V_2 is the volume of the dilute solution.

Biosorption Experiments

0.5g of the sample was added to 100ml conical flask containing nickel (II) and lead (II) solution. Thus the mixture was agitated at 180 rmp at room temperature for 2 h to attain equilibrium. The desire pH was adjusted with 1M NaOH and 1M HCl using a pH meter. The synthetic waste water sample was filtered and analyzed for residual metal ion concentration using AAS. The method by Madhavi *et al.*, 2011 ; Jimoh *et al.*, 2012 was used for the determination of Nickel and lead. The amount of nickel and lead sorbed per unit mass of the biosorbent (mg/g) was evaluated by using the following equation

$$\mathbf{q_e=V(C_o-C_e)}/M \qquad (1)$$

Where, q is the amount of metal ion adsorbed in mg/g; C is the initial metal ion concentration in mg/cm^3; Ce is the final concentration in mg/cm^3 V is the volume of metal ion solution in liters; M is the mass of the leaves, sten and root of *Lanchocarpus laxiflorus* powder used in gram. The percentage Removal of the metal ion was also determined using;

$$\textbf{Removal } \% = [(C_o-C_e)/C_o] \times 100 \qquad (2)$$

The effect of pH solution on the sorption capacity of immobilized LLSB was investigated in this experiment at room temperature (30^0C) and equilibrium time of 2 h. 30 cm^3 of the prepared aqueous solution was measured in a conical flask, followed by the addition of 0.5 g of the sample in the flask. The pH was varied from 1 to 7

The effect of contact time was studied using an initial concentration of 20 mg L^{-1}. The time intervals chosen for this experiment were 30, 60, 90, 120, and 150 minutes. 0.5g of the immobilized Stembark samples was separately mixed with $50cm^3$ solution of initial concentration 100 mg L^{-1}.

The effect of initial metal ion concentration on adsorption was studied as well at equilibrium time of 120 minutes and varying the concentration from 10 - 80 mg cm^{-3}. 0.5g of the immobilized stembark samples were each separately mixed with $50cm^3$ solution of aqueous solution from the prepared $100mg/cm^3$. The solution was then filtered and the concentration of Nickel in the filtrate was ascertained by spectrophotometric methods.

Results and Discussion

Characterization of biosorbents

The FTIR spectroscopic analysis of the plant biomass indicated the presence of hydroxyl, Amide I and amide II (protein) and carboxylate groups as the main functional groups involved in the complexation of metal ions for biosorption processes as shown in figure 1. Strong vibration peaks in the region of 3604.40 – 3400.62 cm^{-1} were due to vibration of OH or –NH functional group. The 2930 cm^{-1} were likely due to the presence of C-H asymmetric stretching vibration in –COOH group (Kumar *et al*., 2012) of methylene groups on the surface. The absorption bands at 1630 cm^{-1} may be assigned to Amide I and amide II (protein) respectively (Pradhan *et al.*, 2007). While the peaks in the range of $1437cm^{-1}$- 1328 could be attributed to carboxylate group (Pradhan *et al.*, 2007). The broad peaks at 1245 and $1240m^{-1}$ could be due to (C- C) or (C- H) or (C-O) stretching of the carboxyl groups (Singh *et al*., 2010). The peaks in the region (1051- 1061)) cm^{-1} were due to the presence of C- C Stretching of the polysaccharides (Singh *et al*., 2010). And 642 cm^{-1} could be associated with different stretching vibrations of C – O stretching on the ring structures (Suantek *et al*., 2011). The bands at 464 and 453 are metal –halogen bands (Struart, 1986).

The morphology structure and elemental analysis of the LL stembark were observed through SEM analysis at 200x magnification. SEM analysis reveals a highly porous structure (Fig 2). The external surface of the stembark is full of cavities with well developed porous structure. The external surface show a rough area having different pore diameters distributed over the surface of stembark which may responsible for metal removal

Table 1: Elemental composition of LLSB by SEM

Element Number	Element Symbol	Element Name	Confidence	Concentration	Error
7	N	Nitrogen	100.0	43.8	1.3
38	Sr	Strontium	100.0	24.2	1.3
9	F	Fluorine	100.0	16.0	3.4
52	Te	Tellurium	100.0	11.6	2.5
33	As	Arsenic	100.0	4.5	4.8

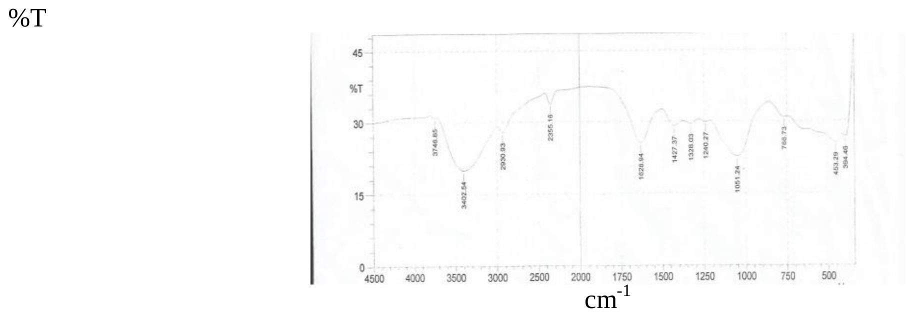

Figure1
FTIR analysis of LLSB

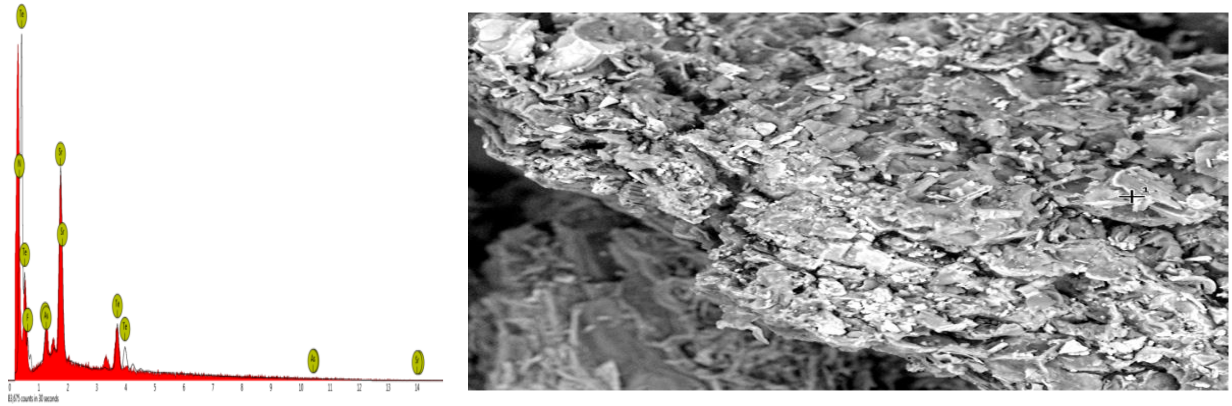

Figure 2
SEM photograph of LLSB (200x magnification)

Effect of pH on the metal ion uptake

For biosorption of heavy metal ions, pH is the most important environmental factor. The pH value of a solution strongly influences not only the site dissociation of the biomass surface, but also the solution chemistry of the heavy metals: hydrolysis, complexation by organic and/or inorganic ligands, redox reactions, precipitation, the speciation, and the biosorption availability of the heavy metals. Fig. 3 shows the effect of pH on nickel and lead ions removal by LLSB biomass. The LLSB has high selectivity for Ni with percent adsorption of 99.9%. The adsorption of metal ion was increased at specific time and becomes constant when it reaches equilibrium. The fast initial uptake was due to the accumulation of metal ions on surface of adsorbent which is a rapid step. This may attributed to the highly porous structure of adsorbents and the particle size, which provide large surface area for the sorption of metals on the binding sites (Nordiana *et al.*, 2013).

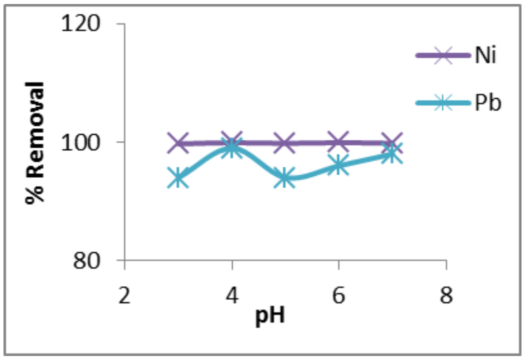

Figure 3: Effect of pH on sorption capacity of LLSB for removal of Nickel (II) and Lead (II) ion

Effect of Contact Time

Figure 4 shows the effect of contact time on adsorption. This was achieved by varying the contact time from 20-120 minutes. the adsorption of lead and Nickel ions was increased at specific time and becomes constant when it reaches equilibrium .The initial rapid uptake was due to diffusion of metal ions onto the surface of adsorbent have reach equilibrium. For nickel, is from 93.46-93.71% and lead is 99.54-99.94% bystembark.

General it shows an interaction between the metal ions and the site of biosorbents responsible for ions uptake. The fast initial uptake was due to the accumulation of metal ions on surface of adsorbent which is a rapid step. This may attributed to the highly porous structure of adsorbents and the particle size, which provide large surface area for the sorption of metals on the binding sites (Nordiana *et al.*, 2013). The adsorption process was faster, it could be because of the largest amount of metal ions attached to the adsorbent within 30minutes. After the adsorption have increases up to 120minutes and almost constant that means it have reached equilibrium and the adsorption did not change with further increase in contact time. The constant stage probably due to the less abundant availability of active sites thus, the sorption becomes less efficient in constant stage (Singh *et al.*, 2006) .

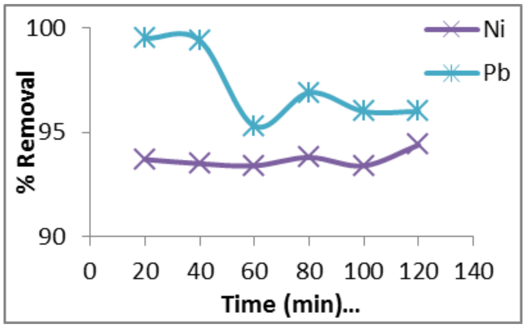

Figure 4: Effect of contact time on sorption capacity of LLSB for removal of Nickel (II) and Lead (II) ion

Effect of Dose

The biomass dose is an important parameter which determines the capacity of a biosorbent for a given initial concentration. The biosorption of Cr, Fe, Cd, Ni and Pb ions as a function of the biomass dosage of .2g to 2g at initial metal ions concentration of 100 mg/L has been investigated and results are shown in Fig. 5

Generally it was observed that as the adsorbent dose was increased from 0.2g to 2g there was increase in the adsorption of metal ions on to the surfaces of LLSB as shown in Fig. 5. The increase in adsorption with increase in adsorbents dose may be attributed to the increase in adsorbent surface area and availability of more adsorption sites (Mahajan *et a*l., 2013). At a given metal ion concentration, the lower the biomass in suspension, the higher will be metal/biomass ratio and the metal retained by sorbent unit unless the biomass reaches saturation. It was found that the stembark was effective at sequestering the metal ions. This may be due to the presence of more available binding sites and least overlapping of sites. Similar trend have been found by Mahajan and Sud (2011, 2012).

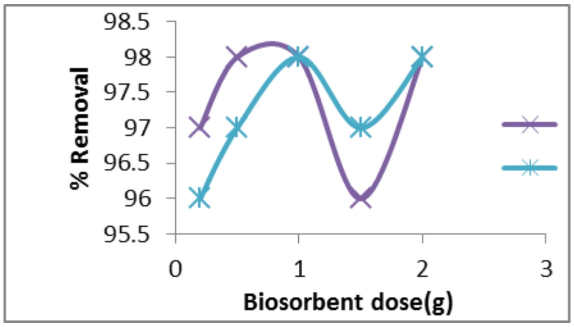

Figure 5: Effect of dosage on sorption capacity of LLSB for removal of Nickel (II) and Lead (II) ion

Effect of temperature

The effect of temperature on the biosorption capacity of Nickel and Lead ions on stembark was studied at different temperature in the range of 25^0C - 60^0C when initial concentration were 100 mg/L at maximum pH values as shown in Figure 2. Generally, the sorption of the metal ions increases slightly with increase in temperature up to 40^0C and then started decreasing. Temperature higher than 40^0C caused a change in the texture of the biomass and reduced its sorption capacity. This is because with increasing temperature, the attractive forces between biomass surface and metal ions are weakened and the sorption decreases (Horsfall *et al.*, 2005) Careful examination of the figures revealed that maximum removed was observed between the temperatures of 25 to 40ºC. The decrease in adsorption with increasing temperature, suggest weak adsorption interaction between biomass surface and the metal ion, which supports physisorption. The effect of temperature on biosorption also depends on the heat of sorption. Usually for physical sorption, heat of sorption is negative and sorption reaction is exothermic and preferred at lower temperature.

Effect of Initial Metal Concentration

The adsorption of metal ions concentration by *L. laxiflorus* plant biomass was studied at different metal concentration ranging from 10-80mg/cm^3 at maximum pH values. The result shows that almost 99% of the metal ions with uptake capacity of about 15mg/g are removed from 10-80mg/L as shown in Fig. 7 Generally, the rapid increase in the uptake of Nickel and Lead metal ions can be attributed to the interaction between the metal ions and the active sites of the adsorbent. This is because the higher the concentration of metal ions, the higher the amount of, Ni and Pb present in the solution, thus the more adsorption of the metal ions occur on the adsorption site of the absorbent. Pb and Ni recorded a decrease in the metal uptake at higher concentration after attaining the maximum valves. This appears to be due to the increase in the number of ions competing for the available binding sites in the biomass. This reduction may be due to in unit biosorption of metal ion with the enhanced metal concentration. These findings indicate that the biomass is effective in dilute metal solutions and also the ratio of sorptive surface of the biomass to metal availability is high. The same results were reported on immobilized biomass of Rhizopus arrhizus for chromium (vi) and Nickel biosorption (Prakasham *et al*.,1999 ; Kumar *et al*., 2012)

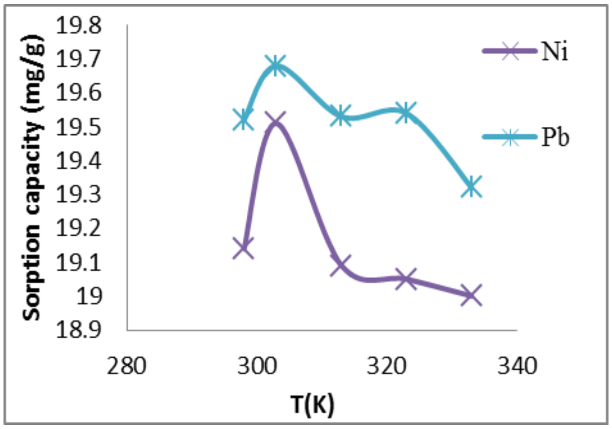

Figure 6: Effect of Temperature on sorption capacity of LLSB for removal of Nickel (II) and Lead (II) ion

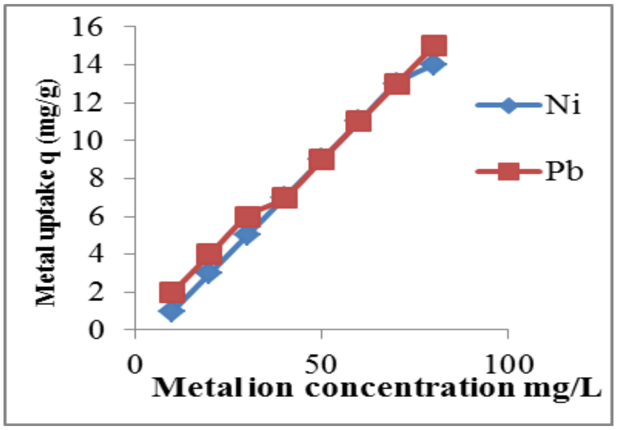

Figure 7: Metal uptake of Nickel and Lead by LLSB biomass at different concentrations in mg/ L

Adsorption isotherms

Adsorption isotherms of Ni and Pb on LLSB was shown in Fig 8 and 9. The adsorption parameters for LLSB are in Table2.The metals uptake capacity of the biomasses was evaluated using Langmur and Freundlich isotherms. The Langmur isotherm (1916-1917) represents the equilibrium distribution of metal ions between the solid and liquid phases and is valid for monolayer adsorption onto a surface containing a finite number of identical sites. The model assumes uniform energies of adsorption on the surface and no transmigration of adsorbate in the plane of the surface. The Lanagmur isotherm is represented by the following equation

$$Ce/qe = 1/Q_o b + Ce/Q_o$$

Where C_e is the equilibrium concentration (mg/L), qe is the amount adsorbed at equilibrium time (mg/g,) Qo is the maximum quantity of metal ions per unit weight of biomass to form a complete monolayer on the surface(mg/g), and b is a constant related to the affinity of binding sites with the metal ions (Lmg^{-1}) (Bansal *et al*., 2009). The plots of Ce/qe versus Ce are linear, which shows that the adsorption of heavy metals studied followed the langmur isotherm model. Qo and b were determined from the slope and intercept of the plot. The correlation coefficient for the different metals studied range from 0.57 – 0.991. The R^2 values were very high for all the adsorbents, which indicated that the data fit reasonably well to the Langmur isotherm in the present adsorption studies. High degree of correlation for linearised langmur suggests monolayer sorption on specific sites or single surface reaction for metal ions (Owunka *et a*l., 2011). The values for the slope were found to be less than unity, implying that significant adsorption took place at a low metal ion concentration (Fig: 8 and Table 2).

The linearised form of Freundlich adsorption isotherm was used to evaluate the sorption data Fig 10 and 11.. The isotherm represents the relationship between the amount of metal adsorbed per unit mass of the adsorben (qe) and the concentration of the metal ion in solution at equilibrium (Ce) (Mahajan & Sud 2011).

$$Ln\ x/m = Ln\ K_f + 1/nLnCe$$

Where K_f(Lg^{-1}) is an indicator of adsorption capacity and n indicates the effect of the concentration on the adsorption capacity and represents the adsorption intensity. The plot of Lnqe versus LnCe for various initial concentrations was linear indicating the applicability of the classical adsorption isotherm to the LLSB.The values of n range between -0.45 to 2.27 which indicates good adsorption (Table2). According to Nirmal Kumar & Cini Oommen (2012), n values between 1 and 10 represent beneficial adsorption. The value of n, which is related to the distribution of bonded ions on the sorbent surface, was found to be greater than unity, indicating that adsorption is favourable.

Table 2: Langmuir and Freundlich Isotherm constants and correlation coefficient for sorption of metal ions from aqueous solution using LLSB

Model	**parameter**	**Heavy metals**				
		Cr	**Fe**	**Cd**	**Ni**	**Pb**
Langmur	qmax (mg/g)	32	-37	81	124.38	172.93
	bL/mg	0.06	-0.42	34	0.41	0.61
	R2	95	66	96	76	87
Freundlich	n	1.18	0.53	0.64	0.77	0.79
	R2	0.973	0.9304	0.6185	0.9091	0.792

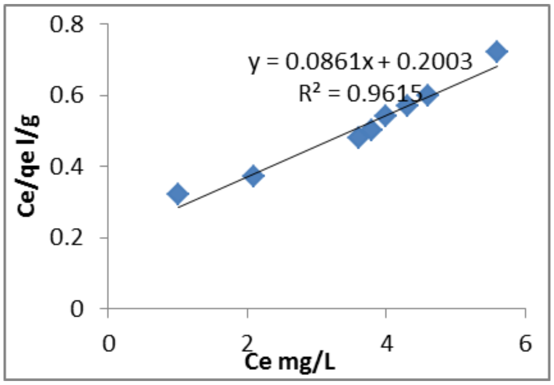

Figure 8. Langmuir Isotherm for the sorption of Ni (II) LLSB at room temperature

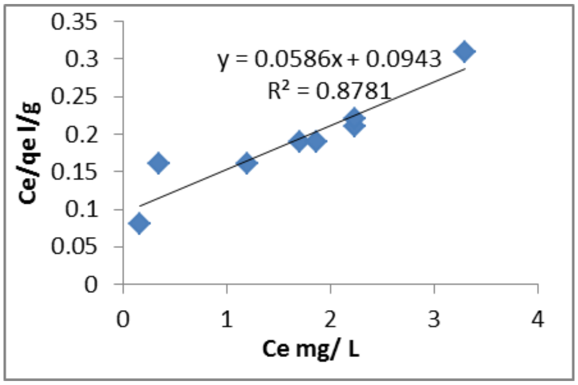

Figure 9. Langmuir isotherm for the sorption using of Pb (II) using LLSB at room temperature

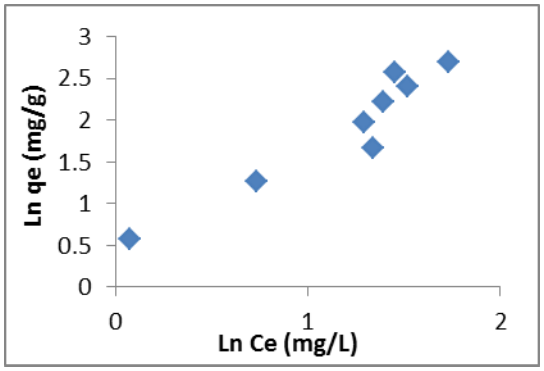

Figure 10. Freundlich adsorption Isotherm for the sorption of Ni (II) using LLSB at room temperature

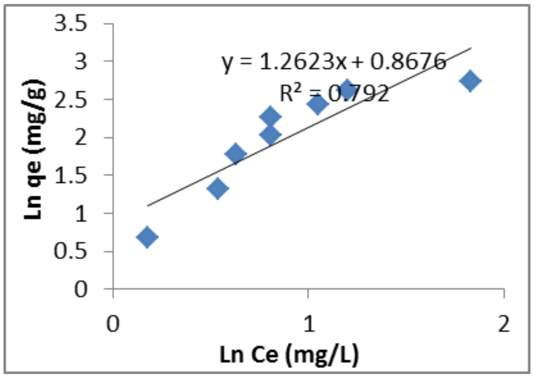

Figure 11. Freundlich adsorption isotherm for the adsorption of Pb (II) using LLSB at room temp.

Conclusions

The use of immobilized *Lonchocarpus laxiflorus* stembark is found to be suitable adsorbent for the removal of Nickel and Lead ions from aqueous solution due to its high adsorption capacity, availability, and low cost.

The biosorption of both metals on the adsorbent was rapid as over 99% of the metals were removed within the first 25 minutes of interaction. It was also observed that the sorption fitted well for Langmur isotherm model based on the correlation coefficient (r^2) data. The nature of the material allows capturing of Nickel and Lead ions effectively due to the active sites of the groups and the rough structure with an irregular surface. On the basis of these facts, it can be concluded that the lonchocarpus Laxiflorus stembark has an adequate morphology for metal ion adsorption. Also it is new, effective, natural, low-cost and alternative adsorbent.

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