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Biosorption of Ni (ll) and Pb (ll) IONS from Aqueous Solutions by Bush Mango (*Irvingia Gabonensis*) Husks

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ABSTRACT

This paper focused on the potential of an economically cheaper adsorbent of natural origin, *Irvingia gabonensis* for adsorbing Ni^{2+} and Pb^{2+} ions from aqueous solutions. The adsorption data was correlated with Langmuir, Freundlich and Dubinin-Radush kevich adsorption isotherm models. It was found that Dubinin-Radush kevich model gave the best description for the adsorption data. The maximum adsorption capacity corresponding to monolayer coverage as evaluated from the Langmuir plots were 15.11mg/g for Pb (II) and 11.95 mg/g for Ni (II), thus indicating a better removal of lead by the adsorbent. Maximum removal of these metal ions from their aqueous solutions occured at a pH 6.0. The apparent energy values, E_D, obtained from Dubinin-Radushkevich plots were less than 8kJ mol^{-1}, indicating that the adsorption of the metal ions by the adsorbent followed a physical mode. Reusability of the biosorbent was assessed by desorption in which HCl acid solution eluted 88.56 % of Pb (II) and 92.68 % of Ni (II) from the spent adsorbent. The results from this study indicated that the *Irvingia gabonensi* husk could be a useful biosorbent for the removal of these heavy metal ions from aqueous solutions and may possibly be useful for treating industrial wastewaters.

Key words: Adsorption isotherm, adsorbates, heavy metals, biosorbent, biosorption.

INTRODUCTION

The preponderance of dissolved heavy metals lost through various human technological activities into our aquatic environments has been identified as a major threat to aquatic organisms. These toxic metals pose a significant threat to organisms, which live in the sites where these wastewaters deposit and cause serious health hazards to human beings, which consume them. There is therefore a need for controlling these heavy metals emissions into our environment.

Several techniques such as ion-exchange, reverse osmosis, evaporation, precipitation, adsorption using activated carbon, solvent extraction e.t.c, which have been employed are all found to be either relatively expensive or sometimes ineffective in the removal of these metal ions. Many manufacturing industries are presently not able to meet new national and local Industrial waste water standards and are therefore in danger of being compelled to stop operations. Biosorption in recent times has offered a great alternative attraction. Biosorption is based on the ability of biological materials to accumulate heavy metals from wastewater by either metabolically mediated, or physico-chemical pathways of uptake (Antunes et al. 2003; Fourest and Roux, 1992).

The main attraction of biosorption is its cost effectiveness. While some of these technique enumerated above may be viewed as mature technologies, biosorption is in its early developmental stages and further improvements in both performance and cost can be expected. It is now obvious that biosorption could become a potent weapon in the fight against toxic metals endangering our environment. Some of the biomaterials that have been previously investigated in the removal of metal ions from aqueous solutions include: moringa stenopetala and moringa oleifera (Sajidu et al., 2006), African white Star apple shell (Anusiem, et al., 2010), Palm kernel fibre (Ofomaja et al., 2005), *Cystoseira indica* (Khani et al., 2006) and *Cicer arientinum* (Ahalya et al., 2005).

In this present work, the use of *Irvingia gabonensis* husk as a biosorbent for lead (ll) and Nickel (ll) ions from aqueous solutions was studied. The influence of initial metal ion concentrations, pH and desorption/regeneration were evaluated and maximum biosorption capacity of the biosorbent was determined from the equilibrium Langmuir adsorption isotherm model.

Materials And Methods

Adsorbent preparation

The Bush mango (*Irvingia gabonensis)* fruits were basically sourced from local market, Abakaliki in Ebonyi state of Nigeria. The edible flesh were removed while the husks were extensively washed with distilled water to remove particulate material from their surface. They were cut into small pieces and air-dried and finally oven-dried at $80^{o}C$ for 12 h in preparation for the adsorption analysis. The oven-dried husks were crushed with manual blender to smaller particles and sieve analysis was performed using the sieve screen to obtain final sample size of $180\mu m$. Thereafter, 200g of the screened ($180\mu m$ size) adsorbent was further soaked in 500mL of 0.3M HNO_3 solution, stirred for 30 min. and allowed to stay for 24 h. It was then filtered through a filter paper and washed with excess deionised water until a pH of 7.1 was obtained. The rinsed biosorbent was later air-dried for 6 h. The treatment of the adsorbent with 0.3M HNO_3 solution was to remove any soluble biomolecules that might interact with the metal ions during the adsorption process and to open-up the micropores of the adsorbent in preparation for the adsorption process.

Biosorption studies

All reagents used were of analytical reagent grades and double distilled deionized water was used in the sample preparation. 100ppm stock solutions of Ni II from NiSO4. $6H_2O$ and lead (ll) from $Pb(NO_3)_2$ were prepared at a pH 6.0 and temperature of 30°C. From the stock solutions, working solutions of 5-50 mg L^{-1} of each of the adsorbates was obtained by serial dilution. The effect of concentration on the adsorption of the metal ions was studied by transferring 50 mL of the different concentrations of the adsorbates (metal ions) into different 250 mL Erlenmeyer flasks while maintaining the pH of the solutions at 6.0. Thereafter, 0.2 g of each of the adsorbents was weighed into the flasks, corked and labelled. The effect of pH on the adsorption of the metal ions was carried out by transferring 50mL of standard solution (50mg L^{-1}) of each of the metal ions into different 250mL conical flasks and then adjusting the pH of the solutions to different pH ranging from 1.0 to 6.0 using 0.1M HCl or 0.1M NaOH. Thereafter, 0.2g of each of the adsorbents was weighed into the flasks, corked and labelled. The flasks were placed in a rotary shaker and agitated for 2 h to attain equilibrium. At the end of the adsorption process, the content of each flask was filtered, centrifuged and the residual metal ion concentrations (Ce) analyzed using buck scientific Atomic Absorption Spectrophotometer (AAS) model 210 VGP. The concentrations of the metal ions adsorbed at the different concentrations were calculated by difference. The analysis was carried out in triplicates and mean residual concentration analyzed.

Desorption studies

The regeneration of the spent adsorbent was carried out by weighing 0.2g of the spent (metal-loaded) adsorbents into different 250mL conical flasks. 50mL of the different desorbing solutions (0.2 M each) was added to the spent adsorbents, and shaking for a period of 2 h. After the agitation, the solutions were filtered, centrifuged and the concentrations of desorbed metal ions in the flasks were analyzed using Atomic Absorption Spectrophotometer (AAS).

Evaluation of metal ions adsorption capacities

The uptake capacity of the metal ions was calculated using the following mass balance equation for the biosorbent (Demirbas et al., 2004).

$$q_e = (C_o - C_e)\frac{V}{m} \qquad (1)$$

where, q_e is the adsorption capacity at equilibrium, ie. amount of metal ion adsorbed per unit mass of adsorbent (mg g^{-1}) at equilibrium, C_o is the initial concentrations of the metal ions in mg L^{-1}, C_e is residual concentrations of the metal ions after adsorption equilibrium, V is volume of the metal-bearing solution in (dm^3 or Litre) and m is the dry mass of the adsorbent (g).

RESULTS AND DISCUSSION

Effect of Adsorbent Dose

Effect of biosorbent dosage on the removal of the metal ions was investigated by varying adsorbent dosage in the range of 0.1 g L^{-1} to 0.5 g L^{-1}. It was observed that the sorption capacity increased with the increase in the adsorbent dosage (Fig. 1). The maximum adsorption capacity for Pb (II) was 8.98 mg g^{-1} while Ni (II) was 7.96 mg g^{-1} at the 0.5 g L^{-1} of biosorbent dose and at initial concentration of 50 mg L^{-1}. The observed increase in sorption capacity with increase in adsorbent dose can be explained by the fact that more and more sites become exposed or available for the metal ions to adsorb and this increases both the amount adsorbed as well as the rate of adsorption.

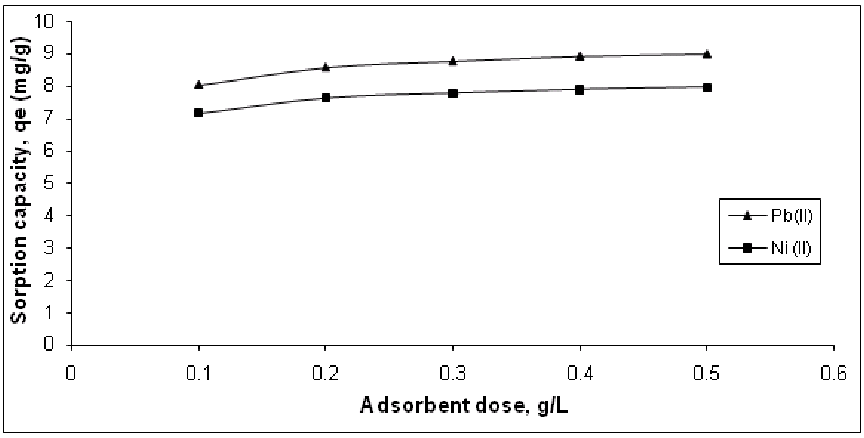

Fig. 1 Effect of adsorbent dose on the sorption capacity of the metal ions. Conc.=50mg L^{-1}; pH=6.0; Temp.=30^0C; contact time = 120 min.

Effect of pH

Metal ions biosorption is closely related to pH. This is because the pH of a solution affects both the adsorbent surface metal binding sites as well as metal chemistry in solution. Also, the level of dissociation of functional groups on the adsorbent surface and concentration of the counter ions in solution are all affected by pH (Nomanbhay and Palanisamy, 2005; Antunes et al., 2003; Terker et al., 1999). The effect of pH on the adsorption of the metal ions onto the adsorbents was evaluated by performing the batch adsorption studies at different pH values in the range of 1.0-6.0. Consideration was made as to not allowing the pH exceeding 6.0 so as to avoid metal precipitation in form of hydroxide at higher pHs. From the results, it is observed that maximum sorption capacities of 7.61mg/g for Ni (ll) and 8.58mg/g for Pb (ll) were obtained at a pH of 6.0 at the initial concentration of 50mg L^{-1} used (Fig. 2). This showed that the optimum pH for the adsorption of these metal ions using the *Irvingia gabonensis* husk as an adsorbent is 6.0. At low pH values, the hydroxonium ions (protons) and the metal cations compete for binding sites on the adsorbent surface leading to decreased metal uptake. Sekher et al. (1998) had suggested that at low pH values, the ligands at the surface of the adsorbent would be closely associated with hydoxonium (H_3O^+)

ions, which may restrict access of the metal ions (adsorbates) onto the ligands due to repulsive forces. The low adsorption capacity obtained in our results at the low pH values suggested that the ligands on the surface of the adsorbent became protonated at the low pH thereby leading to a decreased sorption capacity.

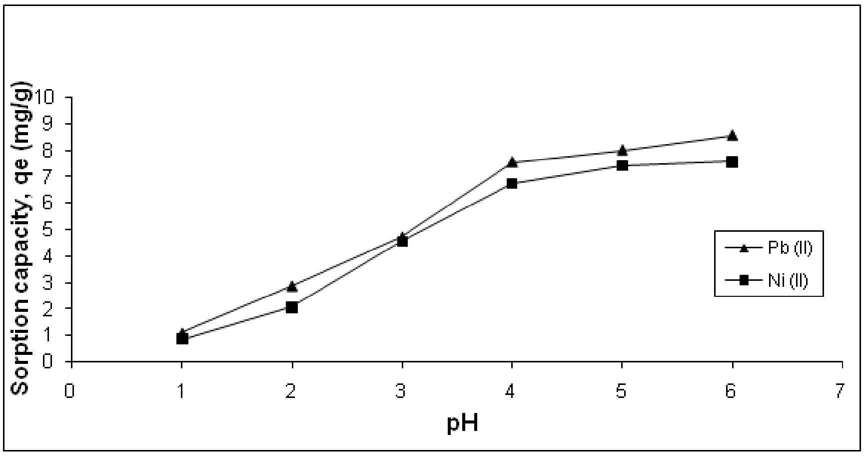

Fig. 2 Effect of pH on the adsorption of Pb (II) and Ni (II) from aqueous solutions by Husk of Irvingia gabonensis, (Conc. 50mg/L, Temp. 30°C)

Effect of Initial metal ion Concentration

The effect of concentration on the adsorption of these metal ions by the adsorbent is in Fig. 3. The plots showed that for all the initial concentrations used, there was an initial increase in the adsorption of the metal ions from solution followed by a slower adsorption towards the end of the sorption process. The curved portions of the plots at higher concentrations indicate the attainment of equilibrium for the adsorption process. The results showed that there was a greater removal of Pb (ll) than Ni (II) by the adsorbent. The relative uptake levels of the two metal ions may be attributed to their variations in their hydration energies. Nickel (II) with ionic radius of 0.072nm ($0.72\overset{0}{A}$) has hydration energy of –1592kJmol^{-1} while lead (II) with ionic radius of 0.12nm ($1.2\overset{0}{A}$) has hydration energy of –1480kJmol$^{-1.}$ It is observed here that the ionic radii of the metal ions are nearly inversely proportional to their hydration energies. The variation in the sorption capacities of the two metal ions may be explained by the fact that metal ions form aquo complexes when dissolved in water (Abia et al., 2002). The aquo complex is of the form $\left[M\left(H_2O\right)_x\right]^{n+}$, where M denotes the metal ion, n$^+$ the net charge on the complex and x the co-ordination number. Since the displacement of water molecules from the aquo ion is the basis of adsorption dynamics, the stability of the aquo ion may therefore play a role in the adsorption process The aquo ion stability, as can be assessed from the hydration energy of the metal ion was found to be dependent on the size of the ion. It can be observed that an ion with a relatively large size (and low hydration energy) will attach less strongly to the water molecules than an ion with a smaller size (and of course relatively higher hydration energy). This suggests that an ion with a large size will interact with the adsorbent sites more strongly than those with smaller sizes. This may have contributed to the greater sorption of Pb (ll) ion than Ni (ll) ion from the solutions onto the adsorbent surface.

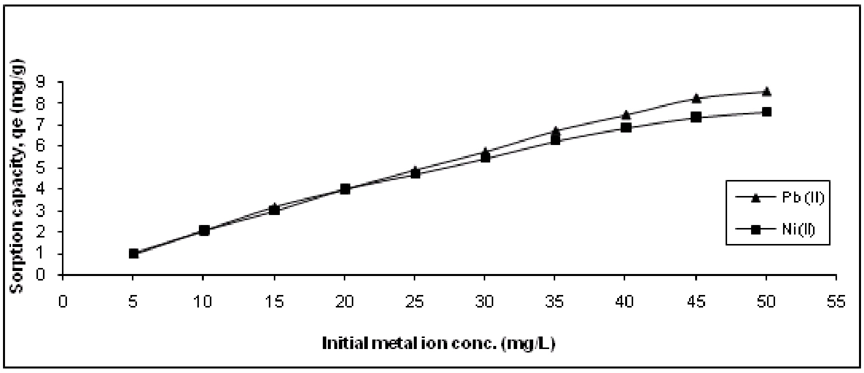

Fig. 3 Plot of sorption capacity vs initial concentration for the adsorption of Pb (II) and Ni (II) ions from aqueous solutions by Husk of Irvingia gabonensis, (pH 6.0, Temp. 30°C)

Adsorption Isotherms

Adsorption Isotherms relate the amount of solutes (metal ions in this case) adsorbed at equilibrium per unit mass of the adsorbent, (mg g^{-1}), to the concentration of the adsorbates, Ce (mg L^{-1}). The Langmuir, Freundlich and Dubinin – Radushkevich isotherm models have been employed in this study to interpret the adsorption data.

The Langmuir isotherm presents one of the first theoretical treatments of non linear adsorption and assumes that adsorption takes place on a homogeneous surface by a monolayer mechanism. The linear form of the Langmuir isotherm equation allows the evaluation of the maximum adsorption capacity (q_{max}) corresponding to monolayer coverage (or site saturation) and the Langmuir constant, K_L of the adsorption process and is represented as:

$$\frac{C_e}{q_e} = \frac{1}{q_{\max} K_L} + \frac{C_e}{q_{\max}} \qquad (2)$$

where: C_e is residual (equilibrium) concentration after adsorption, q_e is the amount of metal ions (Pb^{2+} and Ni^{2+} ions) per gram of the adsorbent (mg g^{-1} adsorbent) at equilibrium, q_{max} and K_L are the Langmuir isotherm constants relating to adsorption capacity and intensity of adsorption, respectively. K_L also expresses the affinity of the adsorbent binding sites for the metal ions. The values of q_{max} and K_L were calculated from the slope and intercept of the linear plots of C_e/q_e vs C_e) (Fig. 4)..

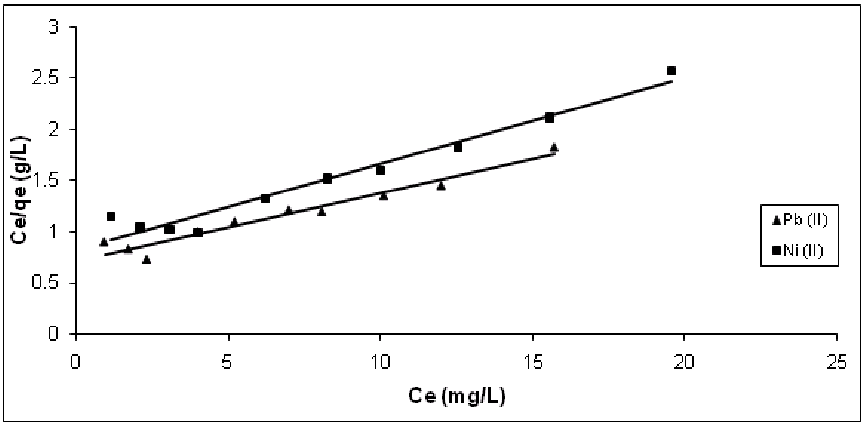

Fig. 4. Langmuir isotherm plot for the adsorption of Pb (II) and Ni (II) ions from aqueous solutions by Husk of Irvingia gabonensis, (pH 6.0, Temp. 30^{o}C)

The Langmuir constants K_L and q_{max} and R^2 values are displayed in Table 1. The high correlation coefficients (R^2 values) obtained showed that the Langmuir isotherm gives a good interpretation for the adsorption data. The q_{max} values (maximum adsorption capacity corresponding to sites saturation) obtained for the metal ions on the adsorbent showed a greater removal for Pb(ll) than Ni (ll) (Table 1).

The essential characteristics of the Langmuir isotherms can be expressed in terms of a dimensionless quantity called separation factor (or equilibrium parameter) R_L, defined as: (Ahalya et al., 2005).

$$R_L = \frac{1}{(1+K_L C_o)}. \tag{3}$$

where K_L is the Langmuir constant and C_0 is the initial concentration of the metal ions (mg L^{-1}). The R_L values indicate the shape of the isotherm. According to Kadirvelu et al. (2001) and McKay et al. (1982), R_L values between 0 and 1 indicate favourable adsorption. Also, R_L value equal to 0 indicate irreversible adsorption, $R_L = 1$ is linear and $R_L > 1$ indicates unfavourable adsorption.

The values of R_L obtained for the adsorption of the two metal ions; Ni (II) and Pb (II) by the adsorbent were all less than unity (Table 1) indicating that the isotherms were favourable under the conditions of this study and also indicated that the adsorbent could be a good material for the removal of the metal ions from aqueous solutions.

Table 1. Langmuir Isotherm Constants for adsorption of Pb (II) and Ni (II) ions by the adsorbent.

Metal ions	q_{max} (mg/ g)	K_L (L/mg)	R_L	R^2
Pb (II)	15.11	9.20×10^{-2}	0.1786	0.9477
Ni (II)	11.95	1.01×10^{-1}	0.1653	0.9576

The Freundlich model is usually applied for non-ideal adsorption onto heterogeneous surfaces involving multilayer adsorption. The Freundlich model was introduced to assess the adsorption intensity of the adsorbate towards the adsorbent. The linearized form of the Freundlich adsorption isotherm is expressed as:

$$\ln q_e = \ln K_F + \frac{1}{n}\ln C_e \tag{4}$$

K_F and n are Freundlich constants incorporating all parameters affecting the adsorption process, such as adsorption intensity and capacity, respectively. Linear plots of ln q_e vs ln C_e confirm Freundlich isotherm (Figs. 5). Values of K_F and 1/n were calculated from the intercept and slope of the linear plots and are presented in Table 2. K_f shows the ease of removal and separation of the heavy metal ions {Pb (II) and Ni (II)} from aqueous solutions. The magnitude of the exponent, n (which is linked to the distribution of bonded ions on the adsorbent surface) provides an information on the favourability and capacity of the adsorbent/adsorbate system. According to Treybal (1980), 'n' values between 1 and 10 represent favourable adsorption conditions. In all cases reported here, the values obtained (Table 2) show beneficial adsorption for the system. According to Karthikeyan et al., 2004, the fit of experimental adsorption data to Freundlich model depicts that the forces of adsorption by the adsorbent are governed by physical adsorption (physisorption). Based on the linear correlation (R^2) values, (Table 2), it can be deduced that the Freundlich model also provided a good interpretation of adsorption behaviour of the metal ions by the adsorbent.

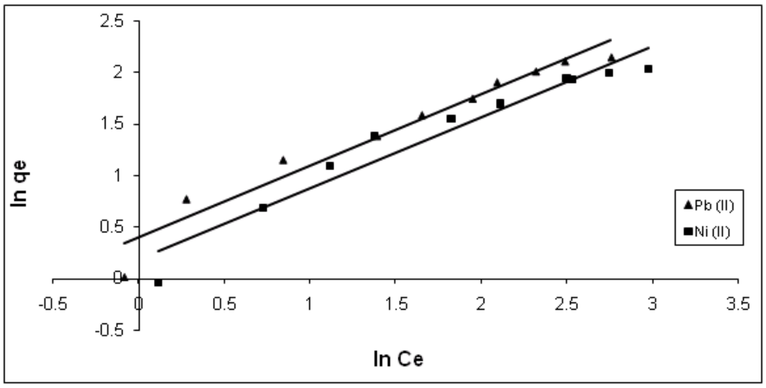

Fig. 5 Freundlich isotherm plot of ln qe vs ln Ce for the adsorption of Pb (II) and Ni (II) ions from aqueous solutions by Husk of Irvingia gabonensis, (pH 6.0, Temp. 30^{o}C)

Table 2. Freundlich Isotherm Constants for adsorption of Pb (II) and Ni (II) ions by the adsorbent.

Metal ions	K_F (mgL^{-1})	1/n	N	R^2
Pb (II)	**1.4891**	**0.6937**	**1.4415**	**0.9519**
Ni (II)	**1.2032**	**0.6906**	**1.4480**	**0.9420**

Dubinin-Radushkevich Isotherm.

The Dubinin-Radushkevich (D-R) model was applied to the equilibrium data to determine if adsorption had occurred by physical or chemical process. The linearized D-R adsorption isotherm is represented as (Bansal et al., 2009):

$$\ln qe = \ln q_D - 2B_D R^2 T^2 \ln\left(1+\frac{1}{Ce}\right) \qquad (5)$$

where q_D is the theoretical saturation capacity (mg g^{-1}) and B_D is a constant related to the apparent adsorption energy, R is the gas constant (kJ mol^{-1} K^{-1}) and T is the temperature (K). The linear plots obtained (Fig. 6) show the fit of the isotherm to the experimental adsorption data. The B_D and q_D values (Table 3) were obtained from the slopes and intercepts of the linear plots. Examination of the data from their linear regression (R^2) values shows that the Dubinin-Radushkevish isotherm provides the best description of the data for the metal ions in our study. The coefficients of correlation (R^2 values) were all high (Table 3) for the two metal ions, indicating that the model adequately described the experimental data.

The constant B_D gives an idea of the mean energy, E_D (kJ mol^{-1}) of adsorption per mole of the adsorbate as it is transferred to the surface of the adsorbent from infinite distance in the solution and this energy is usually evaluated from the energy relation

$$E_D = \frac{1}{\sqrt{2B_D}} \qquad (6)$$

values of E_D provide an information on the nature of the adsorption process, i.e. whether it is chemical or physical, with values in the range E_D = 1 – 8 kJ mol^{-1} showing physical adsorption and in the range 9 – 16 kJ mol^{-1} to chemisorption. The calculated E_D values were found to be less than 8 kJ mol^{-1} (Table 3), showing that the adsorption process of the metal ions by the adsorbent followed physical adsorption.

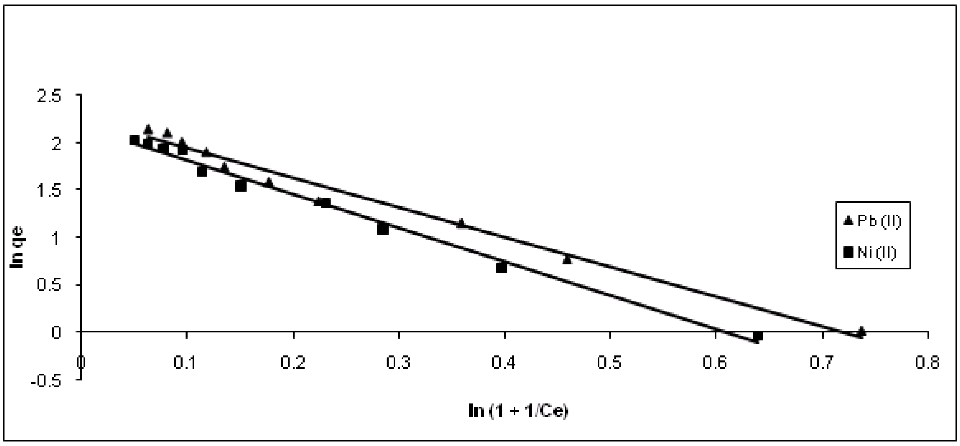

Fig. 6 Dubinin-Radushkevich isotherm plot of ln qe vs ln (1+1/Ce) for the adsorption of Pb (II) and Ni (II) ions from aqueous solutions by Husk of Irvingia gabonensis, (pH 6.0, Temp. 30°C)

Table 3: Dubinin-Radushkevich Isotherm Constants.

Metal ions	q_D (mg g^{-1})	B_D (mol^2 kJ^{-2})	E (kJ mol^{-1})	R^2
Pb (II)	9.4489	2.45×10^{-7}	1.429	0.9811
Ni (II)	8.8057	2.81×10^{-7}	1.334	0.9897

Desorption

Desorption studies assist in exploring the possibility of recycling the adsorbent. For this study, different desorbing agents were examined for their ability to elute the metal ions from the spent adsorbent. Fig. 7 showed the percentage desorption of the metal ions by the various extractants. Results showed that HCl displayed the greatest efficiency in eluting the adsorbed metal ions from the spent adsorbent. This is in contrast to the other reagents, which were not as efficient as HCl. An efficiency of 88.56% was obtained for the case of Pb (II) while Ni (II) was 92.68% when 0.2M of the HCl acid solution was used. This shows that hydrochloric acid is very suitable in the extraction of the metals from the spent adsorbent. When the acids and the sodium salts were used as desorption agents, the biosorbent's surface was covered with K^+ and H^+ ions. This atmosphere of ions would have disrupted the coordination spheres of the metal ions and as a result, these metal ions could not compete with the K^+ and H^+ ions for active sites and subsequently, the heavy metal ions were forced or released from the surface into the bulk solution.

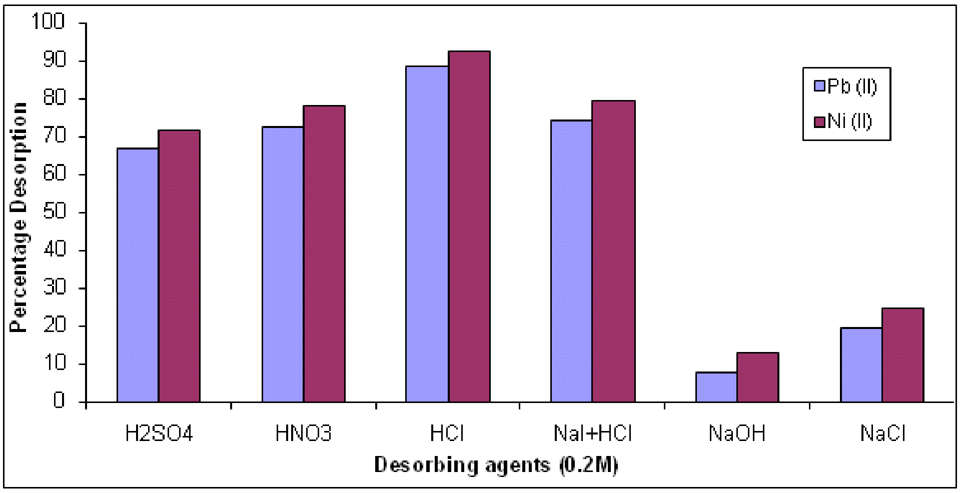

Fig. 7 Effect of various desorbing agents on the desorption of Pb (II) and Ni (II) from the metal-loaded (spent) adsorbent.

Conclusion

In this present investigation, the effects of pH, initial metal ion concentrations and desorption/regeneration were studied. The results generally showed that there was an increase in adsorption capacity with increase in adsorbent dose. Maximum metal ions removal by the adsorbent occurred at a pH 6.0. The Langmuir, Freundlich and Dubinin-Radushkevich isotherm models were applied to the experimental data. Dubinin-Radushkevich model gave the best description for the adsorption data, followed by Langmuir and finally Freundlich. According to Dubinin-Radushkevich model, the adsorption of the metal ions followed a physical mode/nature. Desorption studies revealed an excellent promise of reusing the adsorbent. The adsorbent can be regenerated by using 0.2M HCl acid, and therefore can be reused. The study clearly showed that *Irvingia gabonensis* husk is a better adsorbent for lead (II) than nickel (II) from aqueous solutions, having a greater sorption capacity. From this study, it has been shown that the use of *Irvingia gabonensis* for adsorption of Pb (II) and Ni (II) ions is technically feasible and eco-friendly. Besides that, being composed entirely of agricultural waste, it helps in reduction of waste generation.

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