Competitive Adsorption and Displacement Behaviour of Some Heavy Metals on Groundnut (*Arachis Hypogaea)* Shell

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Abstract: Adsorption is recognized as an effective process of metal ion removal from contaminated water. Groundnut shell has been investigated as a metal adsorbent because of its advantages in terms of both adsorption efficiency and economic viability. In this study, the competitive adsorption of Cd^{2+}, Cu^{2+}, Zn^{2+}, and Pb^{2+} from groundnut shell and their displacement behaviors were investigated under harmonized optimum adsorption conditions for the metal ions. It was observed from the results obtained that groundnut shell favored adsorption of the ions in the order $Pb^{2+} > Zn^{2+} > Cd^{2+} > Cu^{2+}$. The extent to which the ions displaced each other showed that Cd^{2+} had the highest displacement characteristics followed by Zn^{2+} with Cu^{2+} being the last. The occurrence and extent of displacement were shown to be closely related to pH, as this influences the metal species present in solution as well as the corresponding ionic charges. The preference of groundnut shell adsorption for one metal over another was also dependent on the solution pH and concentration. Properties such as electron configuration and ionic size also contributed to the effectiveness of the metals in competitive adsorption. Cd^{2+} and Zn^{2+} were shown to be more easily displaced than Pb^{2+} and Cu^{2+}. These findings are important in choosing species for adsorption and displacement in complex solution or mixture with respect to its relevance.

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1. Introduction:

Widespread industrialization has damaged the environment alarmingly. This is mainly due to the generation of a large amount of hazardous waste and the pollution of both usable surface water and soil(Abudaia et al., 2013).Water pollution caused by heavy metals has posed a significant threat to the environment and public health because of their toxicity, accumulation in the food chain and persistence in nature (Song et al., 2013).Therefore, the removal of heavy metals from industrial wastewater is important in order to safeguard public health, as well as the environment especially aquatic environs.

Adsorption has been universally accepted as one of the most effective industrial wastewater treatment processes with low cost, ease of handling, low consumption of reagents, scope for recovery of value-added components through desorption and regeneration of adsorbent and complete removal of minute concentration of some of these heavy metals in wastewater (Igwe and Abia, 2006; Chigondo et al., 2013).

A number of materials have been studied in the past in terms of efficiency for metallic adsorption as well as economic viability, one of which is groundnut shell; a carbonaceous fibrous solid agricultural waste found in abundance which encounters disposal problem and is generally used for its fuel value. The heterogeneity of its surface and pore structure provides numerous adsorption sites which may also act selectively in terms of affinity for certain metals and organic molecules (Chauvet, 2003).

Based on these, it becomes necessary to study the physical and chemical processes involved in the adsorption of heavy metals on groundnut shell using certain heavy metals with emphasis on their adsorptive competitiveness and displacement behaviors.

2. Research Methodology:

2.1. Reagents

All the chemicals used were of analytical grade and these include; cadmium (II) nitrate (($Cd(NO_3)_2 \cdot 4H_2O$, purity: 98%), copper (II) nitrate (($Cu(NO_3)_2 \cdot 3H_2O$, purity: 98%), lead (II) nitrate (($Pb(NO_3)_2$, purity: 98.5%), zinc (II) nitrate(($Zn(NO_3)_2 \cdot 6H_2O$, purity: 98%), sodium hydroxide (2.5 M NaOH, purity: 98%), nitric acid (HNO_3, purity: 71%) BDH Chemicals Limited England and deionized water.

2.2. Preparation of Metal Stock solutions

Exactly 2.80 g of $Cd(NO_3)_2$, 3.40 g of $Cu(NO_3)_2$, 1.62 g of $Pb(NO_3)_2$ and 4.55 g of $Zn(NO_3)_2$ were respectively dissolved in 100 mL of deionized water in 250 mL beaker which was later transferred to 1 Liter volumetric flask and made up to the mark with deionized water to make 1000 mg/L of each metal stock solution. Samples of lower concentrations (5.0 mg/L) were prepared from these stock solutions by appropriate dilutions.

2.3. Adsorbent sampling and preparation

Groundnut shells were collected from Jato, Kwande Local Government Area of Benue state, Nigeria. The shells were washed severally with clean water and boiled for 3 hours in order to remove dirt and other colored compounds. The shells were rinsed with deionized water on cooling. The washing continued until the filtrate became colorless. The shells were then dried in an oven (Gufex/DGH 9101) at 70 ºC for 24 hours and thereafter ground using mortar/pestle; the powder was sieved with a plastic mesh to obtain particle sizes ranging from 0.25 - 0.5mm and stored in sterile, closed glass bottles as an adsorbent for the research work.


(a) **(b)**

Plate 1: Dried Groundnut shell (a) and Prepared Groundnut shell adsorbent (b)

2.4. Characterization of the Adsorbent

2.4.1. Determination of pH

A 1.0 g portion of the adsorbent was added into 50 mL of distilled water in a 200 mL beaker and the mixture was allowed to stand for about 6 hours after stirring. The mixture was filtered and the pH of the filtrate measured with a pre-calibrated pH meter (Labtech-14). This procedure was repeated twice and the average value recorded as described by Wuana *et al.*,(2009).

2.4.2. Determination of moisture content

This was done by weighing 5.0 g of the adsorbent into a crucible. This was placed in the oven and heated for 5hours at a constant temperature of 105°C. The sample was then removed and put rapidly into a desiccator and re-weighed. This procedure was repeated several times until a constant weight was obtained. The ratio of the change in weight to the original weight expressed in percentage gave the moisture content of the adsorbent. This procedure was repeated twice and the average value recorded as described by Dada *et al.*, (2012).

2.4.3. Ash content determination

Exactly 2.0 g of the adsorbent was placed in a porcelain crucible (W_i) and transferred into a preheated furnace set at a temperature of 900 °C. The furnace was left on for an hour after which the crucible and its content were transferred into a desiccator and allowed to cool. The crucible and content were reweighed (W_f) and the weight noted. This procedure was repeated twice and the average was taken. The percentage ash content (on dry basis) was given by (Dara, 1991):

$$\frac{(W_i - W_f)}{W_i}\ x\ 100\% \qquad (1)$$

where W_i is initial weight of crucible with the sample, W_f is final weight of crucible with the sample.

2.4.4. Bulk density

A 5.0 g portion of the adsorbent was put into a 100 mL measuring cylinder of a narrow bore and tapped gently until it occupied a minimum volume. This procedure was repeated twice and the average result recorded. The bulk density (in kg^{-3})was calculated by the formula as described by Ahmedna *et al*. (2000).

$$\frac{Mass\ of\ the\ sample}{Volume\ of\ the\ sample} \qquad (2)$$

2.4.5. Attrition test

Exactly 1.0g of adsorbent was dispersed in 100 mL of water and was allowed to stand for 2 hours with constant stirring. After filtration, the residue was dried in the oven and weighed. The mass of the residue was then correlated to the original mass. Loss on attrition was calculated as follows (Toles *et al*., 2000);

$$Loss\ of\ attrition\ (\%) = \left[\frac{M_{i-M_f}}{M_i}\right] x\ 100 \qquad (3)$$

Where m_i is the initial mass, m_f is the final mass.

2.4.5. Determination of Heavy metal in the untreated adsorbent

1.0 g of the adsorbent was weighed accurately using analytical weighing balance and digested with 10 mL concentrated HNO_3 acid in a Pyrex beaker, in a fume cupboard. The resulting solution was then filtered and the filtrate analyzed using AAS (ICE 3000) in order to determine the amount of Cd^{2+}, Cu^{2+}, $Pb^{2+}$and Zn^{2+} in the adsorbent.

2.4.6. Metal adsorption and displacement experiment

5.0 mg/L of a target metal solution (Cu^{2+}) was prepared and 250 mL of the metal solution mixed with 1.0 g of the adsorbent in three separate columns. The mixtures were allowed to stand for 24 hours, after which each mixture was filtered and the filtrate analyzed for residual metal ions after rinsing with buffer solution.

The adsorbents were transferred into the three separate columns. Exactly 5.0 mg/L of the competing metals (Pb^{2+}, $Cd^{2+,}$ and Zn^{2+}) were prepared and 250 mL of each metal solution was introduced into the columns and allowed to stand for 24hours.The mixture was filtered and residue rinsed with buffer solution, the concentration of the competing metals infiltrate and residue were analyzed with AAS. This experiment was repeated using Pb^{2+}, $Cd^{2+,}$ and Zn^{2+} as target metals. Simple calculations were done to ascertain the amount of metal ion displaced by the competing metal ion by comparing the concentration of metal ion in the filtrate and residue.

3. Results and Discussion:

3.1. Adsorbent characteristics

The results obtained are shown in Tables 1-7. The adsorbent characteristic is shown in Table 1. These properties describe the suitability of the adsorbent for adsorption process. The adsorbent had a mean pH of 5.60±0.03 with 5.34±0.40% moisture content. Other properties included; Ash content (28.20±0.5%), Bulk density (266.0±1.0 kg/m^3) and Attrition value of 11.40±0.0%. The pH of an adsorbent is close to the acceptable range of 6 – 8 for water and wastewater treatment (Okieimen *et al*., 2004). The value obtained may be dependent on factors like preparatory method, inorganic matter content and chemically active oxygen groups on its surface as well as the kind of treatment to which the adsorbent was subjected.

Moisture content suggests extensive porosity in the structure of adsorbents. The moisture content of the Groundnut shell adsorbent was 5.34±0.4%. It has been observed that if the moisture content of an adsorbent is high, its adsorptive capacity will be reduced (Sugunadevi *et al*., 2002). Okafor *et al*. (2015) reported moisture content of 6.0% for adsorbent prepared from maize cob, this is similar to the result obtained in this study.

The adsorbent’s low ash content of 28.20±0.5% indicated high carbon content (Yang and Lua, 2003). A similar result was reported by Martinez *et al*. (2006) for adsorbents prepared from olive stones and walnut shells. The adsorbent showed attrition of 11.40% which is high compared to10.38% reported for *Delonix regia* fruit pod (Sugumaran *et al*. 2012).Attrition is an important parameter in understanding loss of adsorbent during handling and regeneration. The result showed that the adsorbent has acceptable attrition loss.

Bulk density gives an estimate of the packing volume of an adsorbent and is important during adsorbate uptake. A value of 266±1.0 kg/m^3, higher than the minimum requirement of 250 kg/m^3 for application in removal of pollutants from wastewater (AWWA, 1991) was obtained. An adsorbent with high bulk density usually possesses lower burn - off time.

The concentrations of the metal ions in the adsorbent are shown in Table 2. Only Cu^{2+} was not detected in the sample. Other metal ions had the following concentrations: Zn^{2+} (0.005mg/L), Cd^{2+} (0.019mg/L) and Pb^{2+} (0.176mg/L).

3.2. Metal Adsorption and Displacement

The adsorption of the metal ions onto groundnut shell adsorbent as seen in Table 3 showed a preference towards Pb^{2+} ions compared to the other metals ions in solution. Pb^{2+} showed an adsorption value of 2.4450 ppm which was the highest. This could possibly be because of its smaller hydration radius and other factors that favor adsorption of Pb^{2+} in solution. Zn^{2+}, on the other hand, showed a good adsorption value of 1.3232 ppm which could not be compared to Pb^{2+} although it was quite better than that of Cd^{2+} with 1.0694 ppm adsorption value.Cu^{2+} with 0.8711 ppm does not show a very good adsorption character, this could possibly be because of its electronegativity, standard reduction potential, and metal ion binding potential. The trend for this adsorption is $Pb^{2+} > Zn^{2+} > Cd^{2+} > Cu^{2+}$.

Four main sets of displacement experiments were conducted using single phase metal systems. The first set considered Cu^{2+} as the target metal for displacement, with Zn^{2+}, $Cd^{2+}$and Pb^{2+}as the competing metals. The second set considered Cd^{2+}as the target metal for displacement, with Cu^{2+}, Zn^{2+}, and Pb^{2+}as the competing metals. The third set considered Pb^{2+} as the target metal for displacement, with Cu^{2+}, $Zn^{2+}$and Cd^{2+}as the competing metals. The fourth set of experiments studied the possibility of Zn^{2+}displacement by Cu^{2+}, Cd^{2+}, and Pb^{2+}.The more suitable metal ions were preferentially adsorbed while the less preferred ions stayed in or were released to aqueous solution. In the analysis and comparison of displacing ability among the metals, competitiveness was inferred based on the statistical coefficients, as well as the affected release of Pb^{2+}, Cu^{2+}, Cd^{2+} or Zn^{2+} into solution. The results are as shown in Tables 4– 8.

3.3. Cu –Displacement Behaviour

Cu^{2+} showed relatively high efficiency for adsorption due to its position in the activity series, electro-negativity, standard reduction potential, metal ion radius and intrinsic binding constants. According to Table 3, Cu^{2+} showed a very instinctive adsorption and hence less effective displacement by competing for metal ions.

3.4. Cd –Displacement Behaviour

The competitive displacement of Cd^{2+} from the groundnut shell is a function of the pH, the concentration of Cd^{2+} and the competing metals, the type and strength of complexation of Cd^{2+} compared to that of the competing metal. The results obtained from the experiment were consistent with the arrangement in the activity series and adsorption efficiency. Adsorption trends predicted by ionic properties indicated that Cd^{2+} is generally a less-preferred adsorptive compared to the other metals, thus a viable candidate for displacement.

The addition of competing for metal and the release of Cd^{2+} ions into solution indicated the relative ease of Cd^{2+} desorption at the pH 5.6. According to previously determined binding data, Cd^{2+}binding is strongly pH dependent and increases with increasing pH (Benedetti *et al*., 1995). It is thus expected for Cd^{2+} to be more easily displaced at pH 5.6. Among the competitive metals, Cu^{2+} showed to be the most effective followed by Zn^{2+}.

3.5. Pb-Displacement Behaviour

Previous studies have shown that Pb^{2+} has relatively high efficiency for adsorption and metal cation exchange. Its smaller hydration radius and higher intrinsic binding constant among others, makes it more favorable for adsorption compared to other metals in the experiments, except for Cu^{2+}. It is thus expected that there was a significantly lower concentration of aqueous Pb-displacement experiments, compared to the release of aqueous Cd^{2+} and Zn^{2+} displacement experiments.

Taking the overall release of aqueous Pb^{2+} into consideration, Cd^{2+} was shown to be more competitive than Zn^{2+} and Cu^{2+} at pH 5.6. Even if Pb^{2+} is in itself a strongly adsorbing metal ion, its electron configuration makes it less favorable for chelation compared to the other metals. Bonding and electron pairing in the chelation of transition metals mainly involves electrons in the *d*-orbital, and the electron configuration of Cu^{2+}, $Cd^{2+}$and Zn^{2+}facilitates this type of covalent bonding. Lead, on the other hand, differs in electron configuration because as its *d*-orbital electrons are surrounded by the 6s-orbitals and 6p-orbitals, making the chelate-forming electrons less accessible compared to Cu^{2+}, Cd^{2+}, and Zn^{2+} (Datukan, 2006). Thus, even if Pb^{2+} is overall a strongly adsorbing metal, there is still a possibility of displacement by these competing metals.

3.6. Zn-Displacement Behaviour

The displacement experiments of Zn^{2+} by competing metals did not correspond with any trend. This is due to pH and the presence of other ions or substances competing for space on the adsorbent and the position of Zn^{2+} on the reactivity series which makes it difficult to be displaced in the solution. Aside from the explanations that have already been mentioned in this section, there are other contributing factors that have to be noted, for example, the kind of buffer solution used in the experiments. The experiments at pH 5.6 made use of an acetate buffer (NaOAc solution). Despite the relatively low strength of the buffer used, the differences in anion components, as well as the differences in the amount of Na^+ in solution may influence metal-ligand interactions, as well as other factors related to adsorption and displacement. Previous studies have found that the presence of Na^+ reduced the sorption of some metals (Brown *et al*., 2000). The reduction of adsorption when monovalent ions are present indicates a buffering action of sorption sites. To some extent, metal adsorption by ion-exchange may involve the exchange of the heavy metals with Na^+ as well as H^+ ions (Devi *et al*., 2002).

Table 1: *Characteristics of the adsorbent*

Parameters	Values
PH	5.60±0.03
Moisture content (%)	5.34±0.40
Ash content (%)	28.20±0.5
Bulk density (kg/m^3)	266.0±1.0
Attrition (%)	11.40±0.0

Table 2: *Concentration of metal ions in the untreated adsorbent*

Metal Ions	Concentrations (ppm)
Zn^{2+}	0.005
Cd^{2+}	0.019
Pb^{2+}	0.176
Cu^{2+}	ND

Table 3: *Adsorption of Cd^{2+}, Zn^{2+}, Cu^{2+} and Pb^{2+} onto adsorbent*

Metal Ions	Initial concentration (ppm)	Filtrate (mg/L)	Residue (ppm)
Cd^{2-}	5.0	3.9256	1.0694
Zn^{2+}	5.0	3.6711	1.3232
Cu^{2-}	5.0	4.1234	0.8711
Pb^{2+}	5.0	2.5397	2.4550

Table 4: *Displacement behavior of Copper*

Metal Ions	Filtrate (mg/L)	Residue (ppm)	Amount Displaced (mg/L)
*Cu^{2+}			
Zn^{2+}	2.1920	2.0021	0.1899
Cd^{2+}	2.6154	2.3211	0.2943
Pb^{2+}	0.6721	0.4001	0.2720

*= competing for metal ion

Table 5: *Displacement behavior of Cadmium*

Metal Ions	Filtrate (mg/L)	Residue (ppm)	Amount Displaced (mg/L)
$^{*}Cd^{2+}$			
Cu^{2+}	2.8031	2.1920	0.6110
Zn^{2+}	2.7707	2.2102	0.5545
Pb^{2+}	1.9202	1.4932	0.4270

*= competing for metal ion

Table 6: *Displacement behavior of Lead*

Metal Ions	Filtrate (mg/L)	Residue (ppm)	Amount Displaced (mg/L)
$^{*}Pb^{2+}$			
Cu^{2+}	2.1900	2.0001	0.1899
Zn^{2+}	2.2033	2.0110	0.1920
Cd^{2+}	1.2902	0.9625	0.3277

*= competing for metal ion

Table 7: *Displacement behavior of Zinc*

Metal Ions	Filtrate (mg/L)	Residue (ppm)	Amount Displaced (mg/L)
$^{*}Zn^{2+}$			
Cu^{2+}	2.7202	2.2900	0.4302
Cd^{+}	2.7140	2.1241	0.5899
Pb^{2+}	1.3420	0.9021	0.4399

*= competing for metal ion

4. Conclusion:

From the experiments performed, the ease of $Cd^{2+}$and Zn^{2+} displacement was shown as a product of the inherent characteristics of both Cd^{2+} and Zn^{2+} and the competing metals. Apart from initial concentration, electron configuration, pH and ion size are thought to influence metal binding to organic matter. Periodic properties explain the preference of groundnut shell towards Cd^{2+} and Zn^{2+} over Cu^{2+} or Pb^{2+}as they influence the distribution of charge and the type and strength of complexes formed. The electron configuration of Cd^{2+} and Zn^{2+}enables these metals to form strong chelates via electron pairing in the d-orbital. The following is the trend for adsorption and displacement behaviors of the metal ions as results indicate

For metal adsorption: Pb^{2+} (2.4550)> Zn^{2+}(1.3232) > Cd^{2+}(1.0694) >Cu^{2+}(0.8711).

For displacement behaviour: Cd^{2+}(60%) > Zn^{2+}(50%) > Pb^{2+}(25%) > Cu^{2+} (23%).

Groundnut shell adsorbent is a viable material for the treatment of metal-contaminated water, as it is able to bind metals through several mechanisms. However, the long-term efficiency of groundnut shell as an adsorbent in terms of metal selectivity, as well as the time and of its metal binding capacity needs to be considered in planning wastewater treatment alternatives. The possible displacement of adsorbed ions upon additional metal loadings is a material limitation that has to be addressed and remedied in order for groundnut shell to be developed as part of any wide-scale treatment of toxic heavy metals. These findings are important in choosing species for adsorption and displacement in complex solution or mixture with respect to its relevance.

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