

Full Length Research Paper

Removal of heavy metal ions in aqueous solutions using palm fruit fibre as adsorbent

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The ability of the biomass of palm fruit fiber in removing Pb, Cu, Ni and Cr from aqueous solution was investigated as a function of concentration, contact time and pH variations. Palm fruit fiber from the study locations were washed with deionized water, air-dried and ground using electric grinder. The powdered fiber was sieved and treated with 0.3 M HNO₃ solution for 24 h, washed with deionized water until pH 7.2 and oven dried at 60°C. The biomass was added to 1 M stock metal ion solutions made from Copper sulphate, Potassium dichromate, Lead nitrate and Nickel sulphate. The concentrations, contact time and pH of each stock solution were varied. The mixtures were shaken, filtered and analyzed by GBC Avanta Atomic Absorption Spectrophotometer version 2.02. The results showed mean percentage recovery of 51.08% Pb, 54.75% Cu, 46.96% Ni and 44.91% Cr for concentrations, 96.96% Pb, 9.79% Cu, 49.21% Ni, and 7.63% Cr for contact time and 87.48% Pb, 82.86% Cu, 56.71% Ni and 37.68% Cr for pH. The application of the biomass to waste water showed percentage removal of 73% Pb, 78% Cr, 82% Cu and 87% Ni. The mean percentage removal value revealed Pb as the highest and Cr as the least adsorbed. The sorption capacity of the biomass decreased with increasing concentration of metal ion but increased with decreasing pH and increasing contact time. Chemical modification of the biomass enhanced its capacity. Thus the palm fruit fiber biomass is cost effective and has great potential for use as adsorbent in removing heavy metals from aqueous solutions.

Key words: Palm fruit fiber, heavy metal, biomass, ion, adsorbent, contact time, percent removal.

INTRODUCTION

Man's activities through industrialization, urbanization, technological development and agricultural activities discharge heavy metals into the environment; water, land and air which has become a matter of concern over the past few decades, due to the characteristics of metals to cause objectionable effects by impairing welfare and reducing the quality of life in the environment. The presence of metal ions in the environment is detrimental to many living species (Benhima et al., 2008). The removal of heavy metals is very important because they

are non-biodegradable. Heavy metals affect flora, fauna and other abiotic components of the ecosystem by irreversibly binding to the active sites of enzymes, resulting to metabolic alterations and changes that pose severe health injury and hazard (Rau and Amit, 2002).

Waste water collected from industries, municipalities and communities must ultimately return to the receiving water and land (Raji and Anirudhan, 1997). For example when waste water is released to land without treatment, it undergoes various processes such as diffusion, mechanical dispersion, chemical reaction and adsorption, the end product of which is the accumulation of metals in the soil for years. Furthermore, when untreated waste water contaminated with heavy metals is released into aquatic system, some microorganisms and plants in the

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system have the ability to bioaccumulate and biomagnify these heavy metals in the environment. The ingestion of these contaminants may affect not only the productivity and reproductive capacities of these organisms, but may also ultimately affect the health of man that depends on these organisms and plants as major source of protein (Davies et al., 2006).

Virtually all metals including the essential and non-essential metals are toxic to aquatic organisms as well as humans if exposure levels are sufficiently high (Law, 1993), and the presence of heavy metals has a potentially damaging effect on human physiological and biological systems when tolerance levels are exceeded (Fatoki et al., 2002).

A wide range of physical and chemical processes and techniques are available for the removal of heavy metal ions from waste water. These include reduction – precipitation, ion exchange, reverse osmosis, carbon sorption and electrolytic methods (Osinowo and Olayinka, 1998; Blanco et al., 1999; Babel and Kurniawan, 2003; Igwe and Abia, 2003; Mohan and Pittman Jr., 2007; Chiban et al., 2011a, b, c; Soudani et al., 2011 a, b). A survey of the advantage and disadvantage of these techniques reveals that; reduction-precipitation methods offer economic advantage but the draw back is the production of sludge in huge quantity and when different heavy metal ions are contained in same effluent, simultaneous treatment is difficult because of change in pH required for their precipitation (Okoye et al., 2001). Ion exchange and reverse osmosis are found to be effective and can remove 90-95% of most metals (Osinowo and Olayinka, 1998) but cost of operation is economically not appealing (Saifuddin and Kumaran, 2005). Electrolytic method is economically expensive; energy requirement is high and release other products that require disposal (Juang and Shiau, 2000). Adsorption using commercially activated carbon (CAC) can remove heavy metals from waste water but remains expensive material for most industries.

A review of these techniques and processes requires the need to explore a low cost technique and materials that are capable of being efficient and can compete favourably with other techniques; hence the principle of adsorption using biopolymer wastes products for the removal of metal ions in aqueous solution. The focus of recent researches is to use natural adsorbents which can be economic and cost effective (Benhima et al., 2008; Chiban et al., 2011a, b).

The use of palm oil milling wastes (fiber-biopolymers) is considered from the fact that natural biopolymers are capable of lowering transition metals ions to parts per billion (Saifuddin and Kumaran, 2005). Furthermore, certain wastes from agricultural operation may have the potential to be used as low cost adsorbents as they represent unused resources and are widely available and environment friendly (Deans and Dixon, 1992).

In Nigeria, oil palm (*Elaeis Guineenses*) is an important commercial crop and the expansion of this industry has generated enormous amount of vegetable wastes such as

fiber, empty fruit bunches, palm fronds, palm kernel shells, trunks and effluents/sludge. The discharge or release of these wastes especially effluents and fibers without proper treatment may damage the environment both in the receiving water and foul smell to neighbourhood and communities. When these wastes are released into the receiving water, it increases the biochemical oxygen demand (BOD) of the water and at higher concentration, death of fishes and other living organisms occur due to proliferation of micro-organisms that consume the dissolved oxygen in water leading to depletion of oxygen in the system.

The use of processed palm oil wastes-fibers will add economic value; provide a potentially inexpensive material that will be used for the removal of metal ions from aqueous solution and help reduce the cost of waste disposal. Waste fibers are equally available at little or no cost; however there is the associated problem of disposal due to the enormous wastes generated in the industry that requires its treatment and disposal. Efforts are only made to convert empty fruit bunches to agricultural manure leaving the fibers and effluents to pollute the environment hence the need to carry out this study to explore the usefulness of this fibers. Thus this study is aimed at utilizing waste palm fruit fibers as adsorbents in the removal of copper, chromium, lead and nickel in aqueous solution by varying the experimental conditions such as contact time, metal ion concentration and pH which could be applied in reduction of heavy metal pollution in the environment.

MATERIALS AND METHODS

Study area

The study area Oshika, Elele-Alimini and Ubima are located in Ahoada West and Ikwerre Local Government Areas of River State, Nigeria. The industrial oil mill at Ubima is about 10,000 hectares of oil palm estate and extends up to Elele-Alimini. Oshika lies between longitude 6° 33' 42.54" E and latitude 5° 04' 0.24" N. Elele-Alimini lies between longitude 6° 43' 31.26" E and latitude 5° 03' .96" N. Ubima lies between longitude 6° 52' 57" E and latitude 5° 8' 14" N. Between the Risonpalm and Elele Alimini are series of local palm oil processing mills releasing huge quantity of palm fruit fibers waste on land and into aquatic environments. The major activities in the area are farming of cash crops and processing of palm fruits. Figure 1 is the map of the study areas showing the sampling locations.

Preparation of biomass (Organic wastes)

Fresh palm fruit fiber collected from the study locations were washed properly with deionized water, air-dried and ground using an electric grinder. The powdered fiber material was sieved through a mesh size of 105 µm to obtain fine biomass. The finely sieved biomass was treated with 0.3 M HNO₃ solution for 24 h, followed by washing with deionized water until pH of 7.2 was achieved and oven dried at 60°C with constant mixing. The prepared biomass was stored in desiccators.

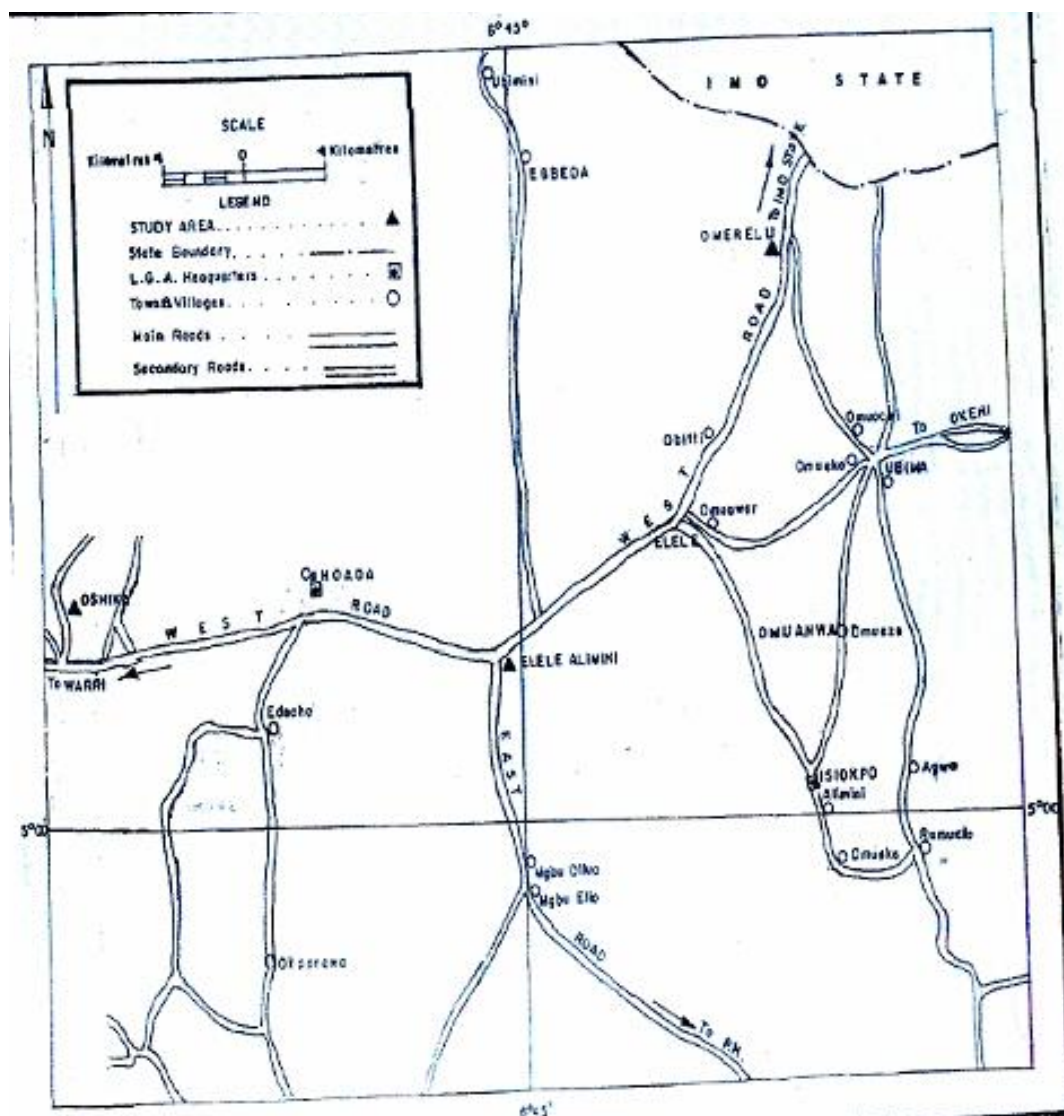


Figure 1. Map of the study areas showing the sampling locations.

Metal salts used and preparation of stock solution of metal ions

The following metal salts were used to prepare the stock solution of metal ions in aqueous form: Copper sulphate ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$), Potassium dichromate ($\text{K}_2\text{Cr}_2\text{O}_7$), Lead nitrate ($\text{Pb}(\text{NO}_3)_2$) and Nickel sulphate $\text{Ni}(\text{SO}_4) \cdot 6\text{H}_2\text{O}$.

For each of the metal ions, a stock solution of 1M was prepared separately by dissolving the appropriate weight of the metal salts in 1000 ml of distilled water. The solutions were prepared using a standard flask. The range of concentrations used was prepared by serial dilution of the stock solution with deionized water.

Batch sorption experiment

The sorption studies were carried out at room temperature using a conical flask, containing 50.0 ml of the test solution, with a known metal ion concentration.

Before mixing with adsorbent, the pH of the solution was

adjusted to 7.0 with 1M HCl and/or NaOH. A known amount of dried pretreated fiber (2 g) referred to as biomass was added into the conical flask containing metal ion solution and tightly covered with cellophane and shaken thoroughly, allowing sufficient time for adsorption equilibrium (also to be examined). The content of the flasks was filtered on Whatman filter paper of 0.42 μm porosity and centrifuged at 2800 rpm for 5 min and the supernatants were analyzed by GBC Avanta Flame Atomic Absorption Spectrophotometer version 2.02.

Determination of the effect of metal ion concentration

A 50.0 ml solution of 0.1, 0.2, 0.4, 0.6, 0.8 and 1.0 M Pb^{2+} concentrations was prepared and poured into six conical flasks labeled A, B, C, D, E and F respectively with the pH maintained at 7.0 for all. Then 2 g of the pretreated biomass was added to each of the standard metal ion solution of Pb^{2+} and the flasks tightly covered with cellophane and shaken vigorously for 2 h. The same process was repeated for various standard solutions of the other

Table 1. Concentrations and % Removal of metals in waste water during pre and post application of biomass.

Application	Pb	Cr	Ni	Cu
Pre ($\times 10^{-3}$ mg/g)	25.0	20.50	8.50	16.0
Post ($\times 10^{-3}$ mg/g)	6.75	4.51	1.11	2.88
% Removal	73	78	87	82

metal ions of Cu^{2+} , Cr^{2+} and Ni^{2+} .

Determination of the effect of pH

A 50.0 ml of the standard Pb^{2+} solution was poured into each of six conical flasks and the pH condition varied from 2 to 12 as 2, 4, 6, 8, 10, and 12 respectively by the addition of either dilute HCl or NaOH using a pH meter. Then 2 g of the pretreated biomass was added to the standard metal ion solutions of Pb^{2+} and the flasks tightly covered with cellophane and shaken vigorously for 2 h. The suspensions were filtered and analyzed. The same process was repeated for standard solutions of the other ions of Cu^{2+} , Cr^{2+} and Ni^{2+} .

Determination of the effect of contact time

A 50.0 ml solution of the standard Pb^{2+} solution was poured into each of six conical flasks and the contact time varied from 20 to 120 min as 20, 40, 60, 80, 100, and 120 min respectively and maintaining the pH condition at 7.0. The same process was repeated for standard solutions of the other ions of Cu^{2+} , Cr^{2+} and Ni^{2+} .

Analysis of metal content

The metal ions Pb^{2+} , Cu^{2+} , Cr^{2+} and Ni^{2+} content in each experiment were determined with a GBC Avanta Flame Atomic Absorption Spectrophotometer (FAAS) version 2.02. Analytical grade standards were used to calibrate the instrument, which were checked periodically throughout the analysis for instrument's response.

Application of the experiment

Waste water was allowed to flow through a 4 inch plastic pipe containing palm fruit fiber biomass wrapped with white sieve cloth. The waste water samples were collected before and after passing through the biomass and analyzed for heavy metals, Pb, Cu, Cr and Ni. The statistical tools used to analyze data from the measurements include ANOVA, correlation and regression.

Calculation of metal ions uptake by biomass

The metal uptake was calculated by simple concentration difference method, while the adsorption capacity that is; amount of metal ions (mg/g) adsorbed per g (dry mass) of biomass was calculated using the equation below:

$$Q = \frac{V(C_i - C_r)}{W \times 1000}$$

Where; Q = mass (g) of metal ion adsorbed per g of sample (Biomass); C_i = initial metal ion concentration mg/l; C_r = residual metal ion concentration mg/l at equilibrium; W = weight of biomass in reaction mixture; V = volume of metal ions solution used (ml).

Percentage removal of metal ions can also be computed using the following equation:

$$\%R = \frac{C_i - C_f}{C_i} \times 100$$

Where; % R = Percentage removal, C_i = Initial metal ion concentration; C_f = Final metal ion concentration.

Furthermore, the apparent capacity of the biomass for metal ions was determined as follows:

$$C = \frac{\%R \times C_i}{100} \times \frac{V}{W}$$

Where; C_i = Initial metal ion concentration (M) (mg/kg); V = Volume of metal ion solution used (ml); W = Weight of biomass used (g); C = Apparent capacity.

RESULTS

The results of the use of palm fruit fiber biomass as adsorbent for the removal of metal ions in solutions are presented in Table 1 and Figures 2 to 7.

Table 1 shows the concentration and percentage (%) of metal ions removed from waste water before and after application of the biomass. The concentrations of metal ions before and after treatment indicate Pb as the highest and Ni as the least. The trend of the metal ion concentrations removed is of the order $\text{Pb} > \text{Cr} > \text{Cu} > \text{Ni}$. That is the percentage removal of Ni as the highest and Pb as the least.

The percentage of metal ions removed from the different concentrations of solutions varied between 1.17% (Pb in 0.8 M) and 92.32% (Pb in 0.1 M) (Figure 2). The % removal generally decreased with increasing concentration of the metal ion in solution for all the metals except Pb which suddenly increased at 1 M. The trend of

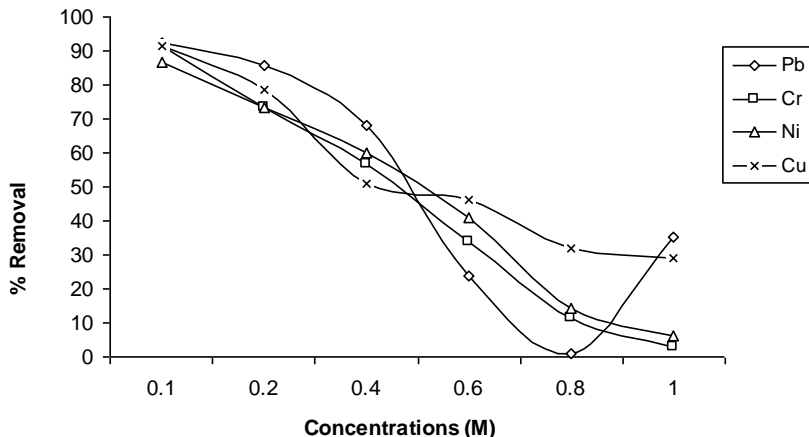


Figure 2. Variations of % removal of metal ion by palm fruit fibre biomass with concentration.

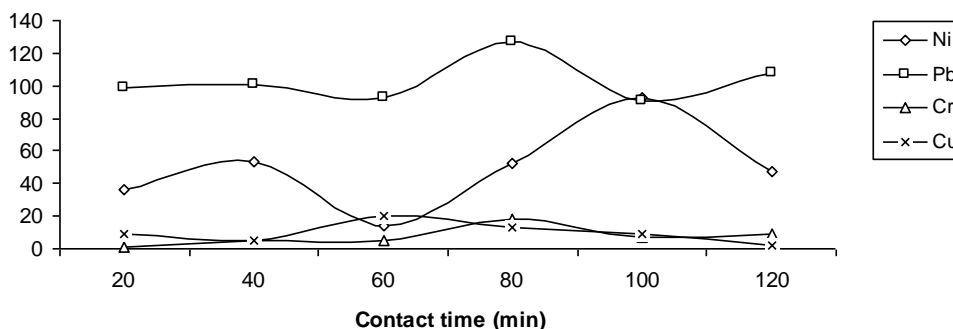


Figure 3. Variations of % removal of metal ion by palm fruit fiber biomass with contact time.

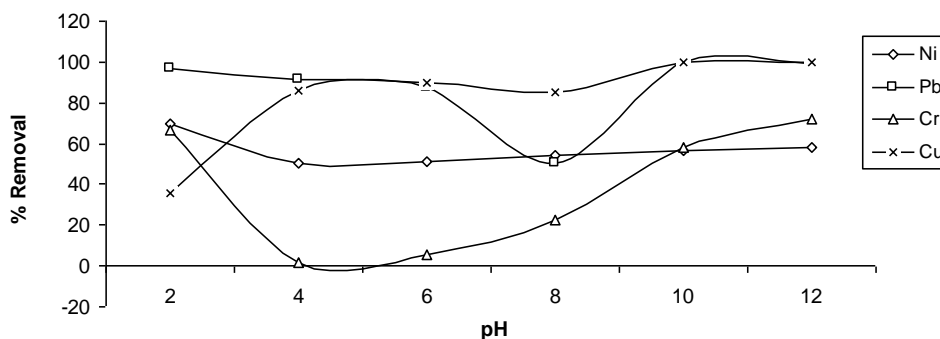


Figure 4. Variations of % removal of metal ion by palm fruit fibre biomass with pH.

percentage removal of metal ions was $Pb > Cr > Cu > Ni$ for 0.1 M, $Pb > Ni > Cr > Cu$ for 0.2 M, $Pb > Ni > Cr > Cu$ for 0.4 M, $Cu > Ni > Cr > Pb$ for 0.6 M, $Cu > Ni > Cr > Pb$ for 0.8 M and $Pb > Cu > Ni > Cr$ for 1 M (Figure 2).

Figure 3 shows the percentage (%) of metal ions removed from the solutions resulting from varying the contact time of biomass from 20 to 120 min. The trend of percentage removal of metal ions was $Pb > Ni > Cu > Cr$ for 20 min, $Pb > Ni > Cr > Cu$ for 40 min, $Pb > Cu > Ni >$

Cr for 60 min, $Pb > Ni > Cr > Cu$ for 80 min, $Ni > Pb > Cu > Cr$ for 100 and $Pb > Ni > Cr > Cu$ for 120 min. The results of percentage removal did not show any regular pattern with increasing contact time for Cr and Cu but increased after 60 min for Pb and Ni.

The percentage (%) removal of metal ions from the solution by the biomass due to varied pH is shown in Figure 4. The % removal ranged between 2.02% Cr at pH 4 and 100% Cu at pH 10 and 12 and 100% Pb at pH 10.

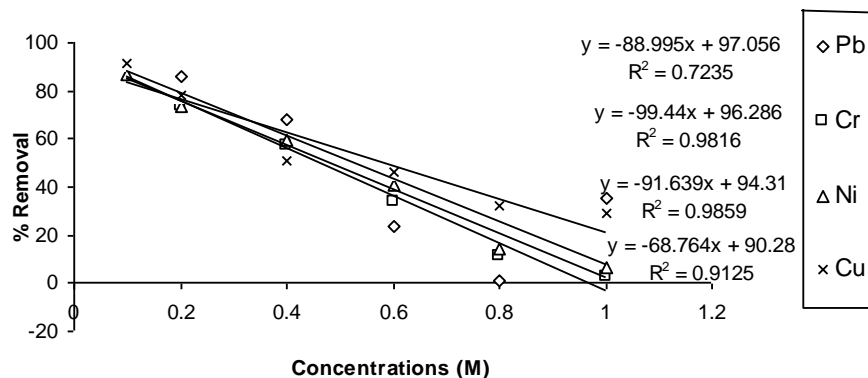


Figure 5. Variation in % removal of metal ion with concentrations of metal solutions.

The trend of percentage removal of metal ions was Pb > Ni > Cr > Cu for pH 2, Pb > Cu > Ni > Cr for pH 4, Cu > Pb > Ni > Cr for pH 6, Cu > Ni > Pb > Cr for pH 8, Pb > Cu > Cr > Ni for pH 10 and Cu > Pb > Cr > Ni for pH 12. Generally, the results showed that Cu removed increased with increasing pH up to pH 12 though with slight decrease at pH 8, Cr showed similar trend from pH 4. The percentage removal of Ni and Pb decreased with increasing pH up to pH 6 for Ni and pH 8 for Pb.

DISCUSSION

Effect of metal concentration

The effect of metal ion concentration showed that the percentage of metal ion removed by the fiber biomass decreased with increasing concentration of metal ion solution. This is because at lower concentrations, the biomass removed large amount of metal ion from the solution. However, as the concentration of metal ion increased the binding sites gradually became occupied and reduce the amount of metal ion being removed. The sorption capacity of the biomass decreased with increase in concentration. This observation agrees with the report of Ayawei et al. (2005) who studied the effect of concentration on the adsorption of metal ion by *Rhizophora mangle* waste biomass. The observations also indicate that surface saturation is dependent on the metal ion concentration and that the active sites took up the available metal more quickly at low concentrations. At higher concentrations, the surface saturation occurred on the biomass surface leading to reduction in biomass concentration as metal ion concentration increases. The differences in the metal ion removal could also be attributed to differences in the size (ionic radii) of the metals.

The relationship between sorption and concentration was fitted into linear regression equation:

$$y = mx + c$$

The slopes of the regression equations for the effect of concentrations were all negative but significant (Figure 5). The slopes of the regression equations for all the metals are negative but greater than unity. This implies higher variation of concentration than % removal of metal ions (low % removal at high concentration). The squared correlation coefficients R^2 of the linear model, (0.7235, 0.9816, 0.9859, 0.9125) for Pb, Cr, Ni, Cu respectively were significant. This implies that sorption is feasible and the metal uptake was high at low concentrations.

Analysis of variance (ANOVA) on the differences between the metal concentrations and uptake, adsorption and % removal by the biomass were not significant ($P > 0.05$). However, the values between the metal adsorption were significant ($P < 0.05$) indicating dependence of % removal and adsorption on concentration of metal ion.

The effect of concentration on uptake and adsorption showed high correlation between Pb and Cr ($r = 0.8994$), Ni ($r = 0.8838$), Cu ($r = 0.8788$); Cr and Ni ($r = 0.9961$), Cu ($r = 0.9695$); Ni and Cu ($r = 0.9547$). However high but negative correlations were observed between concentrations and Pb ($r = -0.9552$).

The application of the biomass to waste water showed high percentage removal of the metal ions viz 73% Pb, 78% Cr, 87% Ni and 82% Cu. The percentage removal indicates that Pb which was highest in concentration was the least removed and Ni which was least in concentration was the highest removed (Ayawei et al., 2005). This implies that metals with low concentration are more highly removed.

Effect of contact time

Effects of contact time (Figures 3 and 6) showed that the amount of metal ions removed by the biomass at different contact times did not show regular pattern. The amount of sorption observed increased to a certain point (Soudani et al., 2011) and decreased later with increasing contact time. This observation indicates that

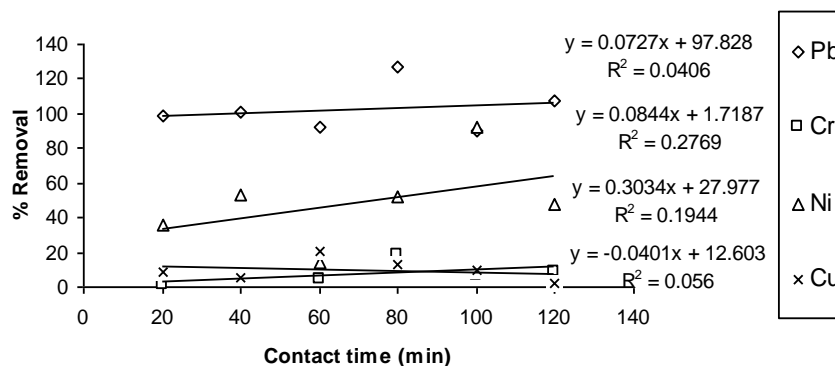


Figure 6. Variation in % removal of metal ion with biomass contact.

the removal of the metal ion by biomass does not depend on the contact time. This observation agrees with the report of Ayawei et al. (2005). The results also indicate that the metal ion removal by the biomass is relatively rapid for all metals investigated. The optimum removal of each metal occurred between 60-100 min. The non uniform pattern of sorption observed could be attributed to non uniform surface area or binding of the biomass.

The regression of metal uptake with contact time was fitted into linear regression equation:

$$y = mx + c$$

The slopes of the regression equations on variation of metal uptake with contact time of the biomass were positive and less than unity for all metals except Cu which is negative. This indicates low rate of variation of % removal as contact time increases except for Cu whose effect is different but negligible. The squared correlation coefficients, R^2 shown in variation of metal uptake with biomass contact time were not significant ($P > 0.05$) (Figure 6). This implies that sorption is not feasible and the metal uptake does not depend on the contact time rather, on ionic radii (sizes) and concentration of the metal ion in solution.

High positive correlation exists between contact time and Ni ($r = 0.6289$), Cr ($r = 0.5260$); Pb and Cr ($r = 0.7274$). The trend of the mean amount of metal removed from the solution by the biomass followed the order, Pb > Ni > Cu > Cr implying that Pb was highly adsorbed and Cr was least adsorbed. This could be attributed to differences in ionic radii (sizes) and binding of biomass. This observation agrees with the order reported by Chiban et al. (2011a, b) and Soudani et al. (2011) in industrial wastewater samples.

Analysis of variance (ANOVA) showed no significant difference ($P > 0.05$) between biomass capacity and the metal ions. However, the differences between percentage removal, amount adsorbed and uptake were significant ($P < 0.05$). This is in contrast with the outcome of the effect

of concentration indicating that contact time does not have major influence on % removal of metal ions.

Effect of pH

The effect of pH on the sorption of metal ion by palm fruit fiber biomass showed that the amount of sorption of metal ions initially decreased except for Cu but later increased with increasing pH. The highest sorption of all the metal ions except Ni was observed at pH 10-12. The highest value of 69.71% observed at pH 2 for Ni could be attributed to binding and irregular measurement of pH. However, the results showed that the amount of sorption fairly depend on the pH of the metal ion solution. This observation implies that high pH favours high sorption of metal ion. This observation agrees with the report of Hashem (2007) who found that in the adsorption of lead ions in aqueous solution by okra waste the pH increased with sorption capacity and lead removal was pH dependent.

High positive correlation was observed between pH and Cu ($r = 0.7972$) and between Ni and Cr ($r = 0.7857$). ANOVA showed significant difference ($P < 0.05$) between the values of metal ions adsorbed indicating non dependence of adsorption on pH. The trend of the mean amount of metal removed from the solution by the biomass followed the order, Pb > Cu > Ni > Cr.

The graph (Figure 7) of variation of metal uptake with pH indicates positive slopes and the regression equations were positive for all metals except Ni whose % removal was high at low pH. Furthermore the slopes of Pb and Ni were less than unity indicating low rate of variation of % removal at higher pH (high % removal at low pH). The slopes of Cr and Cu were greater than unity indicating that the % removal increased with increased pH (higher variation of pH than % removal of metal ions). The squared correlation coefficient, R^2 for Cu (0.6356) was significant while the R^2 for other metals were low and not significant. This implies that the sorption of these metals do not solely depend on pH.

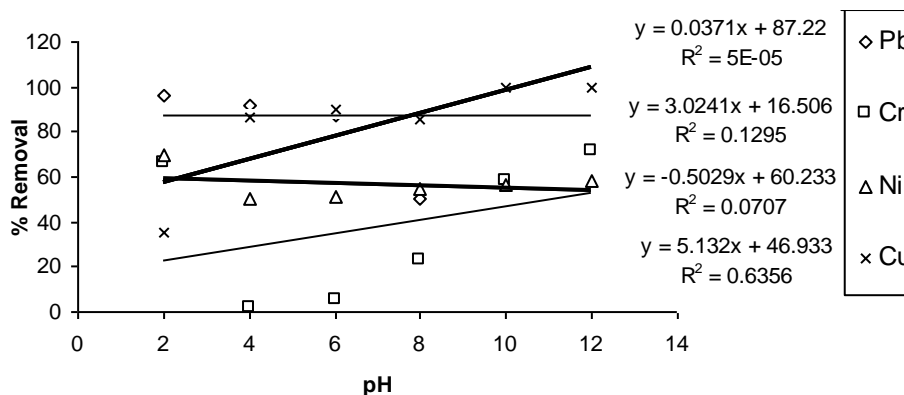


Figure 7. Variation in % removal of metal ion with pH of metal solutions.

It is observed that the % removal of the metal ions followed same order of $Pb > Cu > Ni > Cr$ at concentrations of 0.2 and 1 M contact time of 60 min and pH 4. Also the same order of $Pb > Ni > Cr > Cu$ at concentration of 0.4 M, contact times of 40, 80 and 120 min and pH 2.

Conclusion

The findings of this study have shown that in testing the sorption capacity of palm oil fruit fiber, some metals had 100% removal by this biomass. Therefore it can be concluded that this biomass is environmentally friendly, has great adsorption potential and can be used in the removal or recovery of heavy metal ions from aqueous solutions at low concentrations (treatment of wastewater). Metals with low concentration were more highly removed (high % removal) than metals of high concentration. The sorption capacity of this biomass decreased with increase in concentration. Although the sorption of metal ion did not show any particular trend with contact time and pH, higher sorption occurred at higher contact time and pH. Chemical modification of the adsorbent increased the percentage removal of the metals. The maximum sorption capacities depend on the type, nature and size of the metal ion. The use of palm fruit fiber for the removal of heavy metals has the advantages of availability, efficient and easy disposal. Based on the observed order of frequency of % removal of the metal ions a range of concentrations, 0.2 – 0.4 M, contact times, 60 – 80 min and pH 2 – 4 is recommended for use in the removal of heavy metals from solutions and polluted water.

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