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PRODUCTION AND EXPERIMENTAL EFFICIENCY OF ACTIVATED CARBON FROM LOCAL WASTE BAMBOO FOR WASTE WATER TREATMENT

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ABSTRACT

The production and experimental efficiency of activated carbon from local waste bamboo gotten from construction sites of the rain forest belt of the Niger Delta region of Nigeria for a typical adsorption and treatment of wastewater from a typical refinery was conducted and investigated upon. The bamboo was carbonized (pyrolyzed) at temperatures between 300^{o}C - 400^{o}C and activated using $ZnCl_2$ at 800^{o}C to produce granulated activated charcoal. Adsorption of heavy metals from the refinery waste stream (Warri Refining and Petrochemical Plant, Ekpan, Nigeria) on the activated carbon produced was examined at ambient temperature and, the influence of different operating conditions: contact time, carbon dosages and pH, metal ion concentration before and after adsorption on the adsorbent were used in determining the amount absorbed (q_t, mg/g) and the removal efficiency (R%) of the metal ions. The finding showed that bamboo activated carbon in terms of adsorptive capacity is comparable to the adsorption capacities of a similarly produced activated carbon from cocoa pod husk in another study conducted, which gave similar results obtained for both adsorbents which also fell within the standard limit of measurements. The experiment showed that both adsorbents can be effectively used for the treatment and removal of Pb and Cu from a typical refinery wastewater stream for re-use. The tests' utmost operating conditions for carbon dosage and pH also gave a 100% removal of both metal ions. Thus the effective observation on using activated carbon produced from local bamboo for the adsorption process was quite efficient and could be employed for other adsorption purposes.

Keywords: adsorption, bamboo, activated carbon, waste water treatment, refinery, efficiency

1.0 INTRODUCTION

Nigeria is a country with rich petroleum endowment, it's no longer news that a major percentage of this petroleum is mined in onshore fields, which makes mining activities co-exist with other ecological organisms. Apart from petroleum exploration and production, industrial activities tend to have negative effect on the ecological system. One very common by-product of many industrial operation, be it

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petroleum processing industries, steel processing or electroplating plant is the production of waste water (Igwe and Abia, 2006). Industrial waste water is often contaminated with various organic and inorganic compounds such as phenol, chromium, suspended solids, dissolve organic compound, heavy metals and so on and it is imperative that it should be treated to an environmentally acceptable limit (Okafor and Aneke, 2006). The current problem in industrial waste water treatment stem primarily from the increasing pollution of natural bodies like rivers, underground water and streams by organic and heavy metal compounds that are difficult to decompose biologically because these substances resist the self-purification capabilities of rivers and streams as well as physical decomposition in conventional waste water treatment plant (Olafadehan and Aribike, 2000).

Heavy metals as one of the environmental pollutants are gradually becoming most dangerous without immediate detection because it takes long for the metals to accumulate in the body depending on the distance away from the point of discharge (Igwe and Abia, 2006). Heavy metals pose health hazards to man and aquatic lives if their concentration exceeds allowable limits. Concentrations of heavy metals even below these limits even have potential for long-term contamination, because heavy metals are known to be accumulative within biological systems. Based on the industrial applications and potential pollution impact on the environment, the metals of environmental concern are Pb, Zn, Cu while Cd and Pb head the list of environmental threats because even at extremely low concentrations can causes brain damage in children (Badmus *et al.*, 2007). The increasing demand for alkaline Zn manganese batteries, instead of mercury based ones, brings serious problems when those batteries are not disposed of properly.

Moreover, in many countries concentrations of heavy metals in water are exceeding the permissible/accepted limits. Treatment processes for metal contaminated wastewater include chemical precipitation, membrane filtration, reverse osmosis, ion exchange, and adsorption (Shafaghat *et al.*, 2011). These methods have been found to be limited, since they often involve high capital and operational costs and may also be associated with the generation of secondary wastes which present treatment problems. Adsorption has advantages over other methods for remediation of heavy metals from wastewater because its design is simple; it is sludge-free and can be of low capital intensiveness (Viraraghvan and Dronamraju, 1993). Activated carbon has been reported to have high and fast adsorption capacities (Sirichote *et al.,* 2002) due to its well-developed porous structure and tremendous surface area. The biggest barrier in the application of this process by the industries is the high cost of adsorbents presently available for commercial use.

The cost of adsorption technology application can be reduced if the adsorbent is inexpensive (Onundi *et al*., 2010). Activated carbon adsorption appears to be a particularly competitive and effective process for the removal of toxic heavy metals. The adsorption capacity of an adsorbent is determined by its pore size, chemical structure that influences its interaction with polar and non-polar adsorbates, and active sites which determine the type of chemical reactions with other molecules (Onundi *et al*., 2010). However, commercial activated carbon (CAC) remains an expensive material for heavy metal removal. The use of local, natural, and cheap materials that are available in large quantities or certain waste from agricultural operations for treatment of water and wastewater containing heavy metals in developing countries is an area that is gaining interest (Hsieh and Teng, 2000). There are many studies in the literature relating to the preparation of activated carbon from agricultural wastes such as peanut husks , sugarcane bagasse, pith , nut shells, black gram husk , maize cob husk, walnut, hazelnut, almond, pistachio shell, and apricot stone and their application for the removal of lead, zinc, copper, and cadmium from water and wastewater (Badmus *et al*., 2007); (Igwe and Abia, 2006); (Okafor and Aneke, 2006); (Onundi *et al*., 2010); (Shafaghat *et al.*, 2011) . The removal of pollutant non-aqueous waste stream by adsorption into granular activated charcoal in fixed beds is an important waste water treatment process (Yeh and Thomas, 1995). The adsorption of a single component from an aqueous solution unto activity charcoal carbon has been performed experimentally with different system.

Activated carbon was used in the PACT system developed by US filter and Dupont for treatment and removal of pollutants from industrial or municipal waste water (Meena *et al.*, 2004). Ademiluyi *et al.* (2009) studied lead adsorption using local activated carbon produced from coconut shell and compared to commercially available samples for lead; the former exhibited a slightly lower adsorption capability than the latter. The use of locally prepared activated charcoal from coconut shell shows that it is more

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effective than commercially available alternative, production wise. Amuda and Ibrahim (2006) compared the adsorption efficiently of coconut shell based activated carbon (acid and barium chloride activation) with adsorption efficiency of commercial carbon (Calgon carbon F – 300) with respect to organic matter from beverage industrial waste water. Freunlich adsorption isotherm was used to analyze the adsorption efficiency of the two activated carbons. The studies indicated that the acid activated coconut shell carbon had higher adsorption for organic matter expressed as chemical oxygen demand (COD) than calgon F – 300 at all carbon dosage used. In spite of the efforts by several researchers on various low-cost adsorbents, there is still need to develop suitable adsorbents for the removal of metal ions from aqueous solutions.

Bamboo (*Bambusa vulgaris*, Var) is a fibre crop that grows in wild forest region in Nigeria and very abundant in the Niger Delta region where oil and gas prospecting are at peak. Bamboo is mainly used in Nigeria for support of decking during construction of building after which bulk percentage are discarded as construction wastes. Hence, in this study, an attempt was made to determine the effectiveness of the activated carbon derived from bamboo (Bambusa vulgaris, Var) which is widely available at construction sites all-round the institute into useful adsorbent for removal of heavy metals from industrial wastewater especially from the refinery nearby. This research work goes a step further to establish characterization properties of the produced activated carbon in terms of adsorptive capacity on heavy metals in wastewater and in comparison with activated charcoal produced from cocoa pod husks established in another study.

2.0 MATERIALS AND METHODS

2.1 Materials

The materials used for this study include waste bamboo stems which were obtained from construction sites at the Petroleum Training Institute (PTI), Effurun, Delta State of Nigeria; waste water samples were gotten from Warri Refining and Petrochemical Company (WRPC), Ekpan, Delta State. Others were: concentrated hydrochloric acid, distilled water, muffle furnace (electronic furnace), thermocouple with temperature sensors, sieves of 1.0mm – 1.7mm, measuring cylinders, beakers, pipette, 100ml dropping funnel, glass wool, conical flasks, moisture cans, magnetic stirrers, cutting machine, air oven, sample bottles, desiccators, crucibles, filter papers with several funnels, electronic weighing balance (± 0.001, Adams AFP 360L), spatula, density bottle, crusher, and pH meter (Elico (LI-129)), atomic absorption spectrophotometer (AAS) ((Perkin Elmer HGA900) and mechanical shaker (macro scientific works, India).

2.2 Experimental Methods

2.2.1 Production and Activation

The waste bamboo stems was cut into shorter sizes of 2cm long and further reduced to desired mesh sizes with the aid of a band cutting machine. In other words, cross cutting, splitting, knot removal, and round stick making were carefully done. The cut pieces of bamboo were washed with distilled water for the removal of adherent extraneous matter. The washed materials (bamboo pieces) were dried in an oven at 105°C for 17 hours to remove moisture. See Plate 1. 2000g of well-dried material (bamboo pieces) was weighed out and introduced into the reactor. They were pyrolyzed at 300 - 400°C for 2 hours and 30mins. The distillate formed during the pyrolysis was collected with a receiver connected to the condenser to avoid air pollution. The carbonized material (charcoal or char) was cooled at room temperature for 3-5 hours before discharging into a container. They were then carefully crushed into powder with the aid of crusher and sieved using sieves of 1.18mm mesh to get particles of uniform size. 200g of the well screened carbonized material was weighed out and divided into two (2) equal portions. 100g (one part) was transferred into a beaker mixed with 150ml of 0.1M zinc chloride until the mixture formed a paste. The paste was then transferred into a dry crucible, oven dried for 1 hour at 105°C, and finally introduced into the muffle furnace and heated at 800°C for 2 hours in the absence of air so as to increase the surface area of the sample for adsorption purposes. It was then cooled at room temperature, washed with distilled water until the filtrate obtained from it during washing reached a pH of about 7. The washed activated carbon was then dried for 3 hours in an oven at 150°C. The final product was then kept in an airtight polyethylene bag, ready for use. The same was repeated to the other 100g part.

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2.2.2 Experimental Tests and Analysis

Removal of Pb and Cu ions onto the activated carbon prepared from bamboo was carried out by batch method and the influence of various parameters such as effect of pH; contact time, activated carbon dose, and initial metal ion concentration were studied. The adsorption material was granular activated carbon produced for this study. The adsorbate investigated is waste water samples from WRPC. The initial pH and temperature were measured and recorded while the effluents from the bed were collected at different intervals for two hours.

For each experimental run, 50mL of metal solution of known concentration was taken in a 100mL stoppered reagent bottle; pH was adjusted to the desired value, and a known amount of the activated carbon was added. This mixture was agitated at room temperature (30 ± 1^{o}C) using a mechanical shaker at a constant rate of 100 rpm for a prescribed time to attain equilibrium. It was assumed that the applied shaking speed allows all the surface area to come in contact with heavy metals ions over the course of the experiments. At the end of the predetermined time intervals, the sample was taken out and the supernatant solution was separated from the activated carbon by centrifugation and, analysis on the concentration of each metal ion (Pb and Cu) remaining in solution was carried out using atomic absorption spectrophotometer (AAS). Blank solutions were treated similarly (without adsorbent) and the recorded metal ion concentration by the end of each operation was taken as initial one. Effect of pH was studied over the range of 2.0-80 and pH adjustments were made by the addition of dilute aqueous solution of 0.1M HCl and 0.1M NaOH. Effect of adsorbent dose was studied in the range of 1-4g of adsorbent at ambient temperatures. Kinetics and effect of contact time on adsorption were determined at different time intervals over a range of 30-120min.

The atomic absorption spectrophotometer described above with single element hallow cathode lamps of respective elements operating with an air-acetylene flame, was used to analyze the concentrations of Pb and Cu. An Elico (LI-129) pH meter was used for pH measurements. The mechanical shaker described above was used for agitating the samples. Muffle furnace was used for carbonization and activation of the adsorbent. An average feed flow rate of 0.0485m3/s was maintained. The flow rate of the feed through the bed was determined by collecting known volume of water for a specific time.

2.2.3 Parametric calculations

The following parameters were measured and recorded before and after the actual experiment and they are: Particle size (mm): 1.0 – 7.0; measured weight (g): 2.0; Measured volume (ml): 50. The amount of metal ion adsorbed during the series of batch investigations was determined using a mass balance equation:

$$q_t = v\,(C_o - C_f)/m \qquad (1)$$

where q_t is the metal uptake (mg/g); C_o and C_f are the initial and equilibrium metal concentrations in the wastewater sample (mg/l), respectively; v is the wastewater sample volume (L); and m is the mass of adsorbent used (g). The definition of removal efficiency is as follows:

$$\text{Removal efficiency, R (\%)} = v\,(C_o - C_f)/C_o \times 100 \qquad (2)$$

where C_o and C_f are the metal concentrations in the wastewater sample before and after treatment, respectively.

Adsorption on pH, corresponding amount of metal ion adsorbed and removal efficiency were all calculated using equations 1 and 2 respectively.

3.0 RESULTS AND DISCUSSION

3.1 Results

The activated carbon from bamboo stems were successfully produced however the results analysis and tests carried out which include the adsorption

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process and actions on waste water using the fixed batch adsorption mechanism, sample analysis before and after adsorption of heavy metals (Lead and Copper) in the waste water stream are the following:

3.2 Discussion

The effect of different operating conditions (contact time, carbon dosages and pH) on the removal by adsorption onto activated carbon produced from bamboo was investigated. Tables 1 to 4 present the result and the corresponding metals removal studies for calculated using equations 1 and 2 and Figures 1 to 4 presents the effects of these operating conditions. The metals removal studies which were shown in the Tables and the Figures indicate that the removal of the metals was strongly affected by the different operating conditions.

3.2.1 Effects of pH

pH is an important parameter for adsorption of metal ions because it affects the solubility of the metal ions, concentration of the counter ions on the functional groups of the adsorbent and the degree of ionization of the adsorbate during reaction. The removal of metal ions from wastewater sample by adsorption is related to the pH of the sample; as the latter affects the surface charge of adsorbents, the degree of ionization, and the species of adsorbate. The pH of the aqueous solution is an important controlling parameter in the adsorption process (Baes and Mesmer, 1976) and thus the role of H^+ concentration was examined from samples at different pH covering a range of 2 - 8 (Figure 1). The result shows that adsorption of Cu increased sharply while that of Pb increased at a steady rate at the utmost pH 8, attaining a maximum value of around 89.13% and 73.53% for Cu and Pb respectively.

According to Low *et al*. (1995), at low pH values, the surface of the adsorbent would be closely associated with hydroxonium ions (H_3O^-), by repulsive forces, to the surface functional groups, consequently decreasing the percentage removal of metal. As the solution pH increases, the onset of the metal hydrolysis and the precipitation began at pH > 7 and the onset of adsorption therefore occurs before the beginning of hydrolysis (Baes and Mesmer, 1976). When the pH of the adsorbing medium was increased from 2 to 8, there was a corresponding increase in de-protonation of the adsorbent surface, leading to a decrease in H^+ ion on the adsorbent surface. This creates more negative charges on the adsorbent surface, which favours adsorption of positively charge species and the positive sites on the adsorbent surface (Ghanem and Mikkelsen, 1988); (Kadirvelu and Namasivayam, 2003);(Abdus-salam and Adekola, 2005).

3.2.2 Effect of Contact and Time

Figure 2 shows removal of lead and copper ions from the wastewater sample using bamboo adsorbent as a function of time. The result shows that for Cu, the removal rate was rapid within the first 30 minutes, sharply increased for 90 minutes, slowing down between 90 and 120 minutes. The initial faster rate may be due to the availability of the uncovered surface area of the adsorbents, since the adsorption kinetics depends on the surface area of the adsorbents. On the other hand, percent removal for lead increased gradually with increasing contact time. The lead adsorption takes place at the more reactive sites. As these sites are progressively filled, the more difficult the sorption becomes; and the sorption process tends to be more unfavorable. This is the general characteristic of adsorption of this type of metal ion (Smith; 2002); (Abdus-salam and Adekola, 2005). These changes and differences in metal ion uptake rate could be attributed to two different adsorption processes, namely a fast ion exchange followed by chemisorptions (Nhapi *et al.*, 2011) . A sharp increase in removal rate was however noticed on the removal of lead and copper ions from the wastewater sample using adsorbent as a function of time.

3.2.3 Effect of Amount of Activated Carbon

Dosage study is an important parameter in adsorption studies because it determines the capacity of the adsorbent for a given concentration of metal ion solution. The batch adsorption experiments carried out to establish the nature of equilibrium that existed in the wastewater – granular activated carbon systems, indicated that 1g of activated carbon from bamboo could remove 3.93 mg/g of Cu and 4.4 mg/g of Pb content of the wastewater. On the whole, the effect of absorbent dose on the percent removal of Pb and Cu, at concentration of 50 mg L^{-1} indicates that increasing the absorbent dose for both adsorbents, percent removal increment up to 100% was observed at a dosage of 4g (See Figures 3 and 4).

4.0 CONCLUSION AND RECOMMENDATION

This study has shown that it is possible to produce efficient and effective adsorbents from bamboo. This

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granular activated carbon of less than 1.8mm particle size of waste bamboo from Nigerian construction sites can effectively remove heavy metals from industrial waste water effluents before discharge into the river or sea as is the case with Warri Refining and Petrochemical Company. The removal of heavy metal ions was pH dependent as the concentration of both metals after adsorption at the maximum pH of 8 was recorded to be <0.001 ppm implying that adsorption capacity increases with increasing the pH value of the solution, and at a particular pH. The order of increase of removal percentage was Pb > Cu for both absorbents.

Experimental results showed that the best pH for adsorption was 8 with contact time of 120 minutes. When the addition of the adsorbent dose increased, the percentage removal of metal ions also increased. A maximum removal of approximately 100% was due to the assumption of approximately 0ppm concentration of the metals at pH 8 and dosage of 44. This was observed for both metal ions for 4g dosage. This investigation also showed that absorbent produced from Bamboo is suitable for removing the Pb and Cu heavy metal ions in a typical refinery wastewater scheme. Other metal ions may be effectively removed. This is however open for further studies. For comparative study, steam activation and thermal activation should be carried out on Nigerian bamboo. Effect of various activating agents such as acetic acid, and sulphuric acid, hydrochloric acid etc and, alkaline based activating agents on the textural properties of activated carbon from Nigerian bamboo should be researched upon. The condensate obtained during the pyrolysis could be refined to yield 70% of diesel. Therefore further analysis should also be conducted.

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Table 1: Waste water Analysis before Treatment with Bamboo Activated carbon

S/N	Cu (ppm)	Pb (ppm)	pH
1	3.565	2.941	6.27

Table 2: Adsorption Effect on pH, metal ion and Removal Efficiency

S/n	pH Variation	Cu (pm)	q_t (mg/g)	R (%)	Pb (ppm)	qt (mg/g)	R (%)
1	pH 2	3.229	8.40	9.40	2.008	22.33	31.72
2	pH 4	1.424	53.53	60.10	0.886	51.38	69.87
3	pH 6	0.045	88.125	98.90	0.014	73.18	99.52
4	pH 8	<0.001	89.13	100	<0.001	73.530	100

Where: pH= degree of acidity/alkalinity; Cu= copper (metal ion); Pd= Lead (metal ion); R= Removal Efficiency; and q_t= metal uptake.

Table 3: Adsorption Effect on contact time, metal ion and Removal Efficiency

S/n	pH Variation	Cu (pm)	q_t (mg/g)	R (%)	Pb (ppm)	qt (mg/g)	R (%)
1	pH 2	3.229	8.40	9.40	2.008	22.33	31.72
2	pH 4	1.424	53.53	60.10	0.886	51.38	69.87
3	pH 6	0.045	88.125	98.90	0.014	73.18	99.52
4	pH 8	<0.001	89.13	100	<0.001	73.530	100

Table 4: Adsorption Effects on dosages, metal ions and Removal Efficiency

S/N	Weight Variation(g)	Cu (ppm)	q_t (mg/g)	R (%)	Pb (ppm)	q_t (mg/g)	R (%)
1	1	3.408	3.93	4.4	2.197	18.6	25.30
2	2	1.249	57.9	64.96	0.830	52.78	71.78

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3	3	0.027	88.45	99.24	0.005	73.40	99.83
4	4	<0.001	89.13	100	<0.001	73.53	100

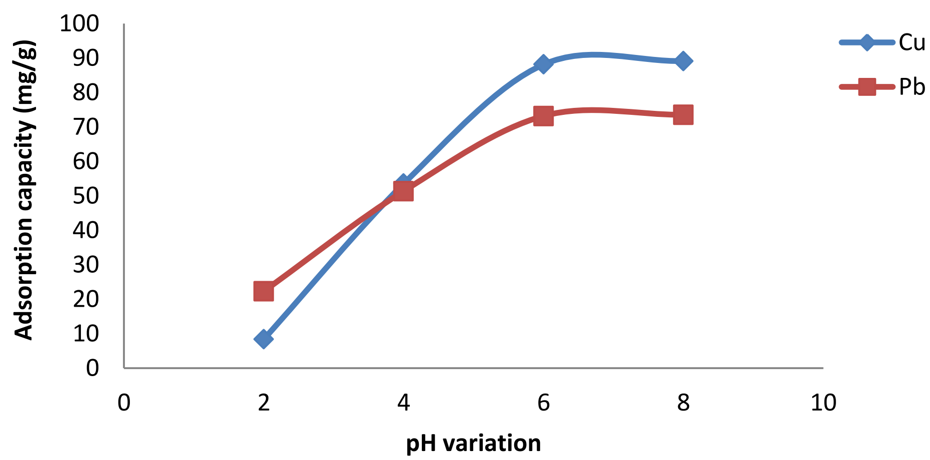

Fig. 1: A graph of Adsorption capacity against pH variation

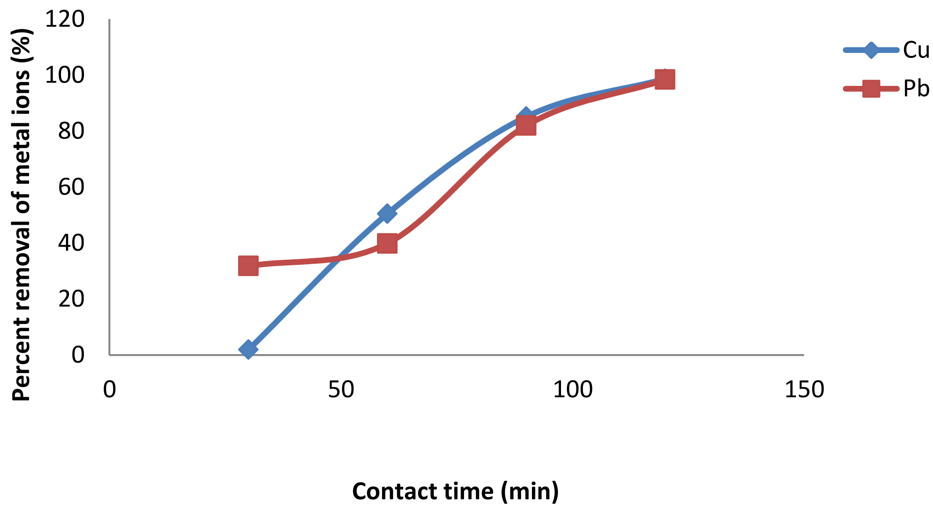

Fig. 2: Percent Removal against Contact time

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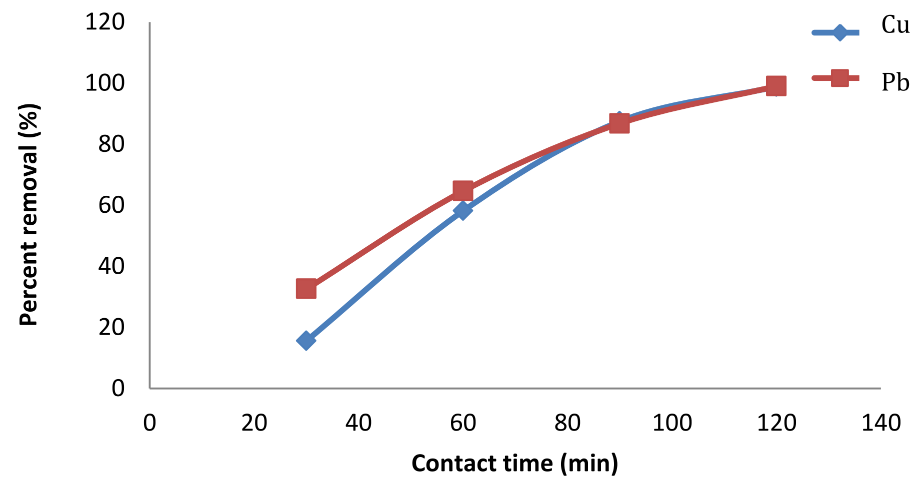

Fig . 3: Percent removal against contact time

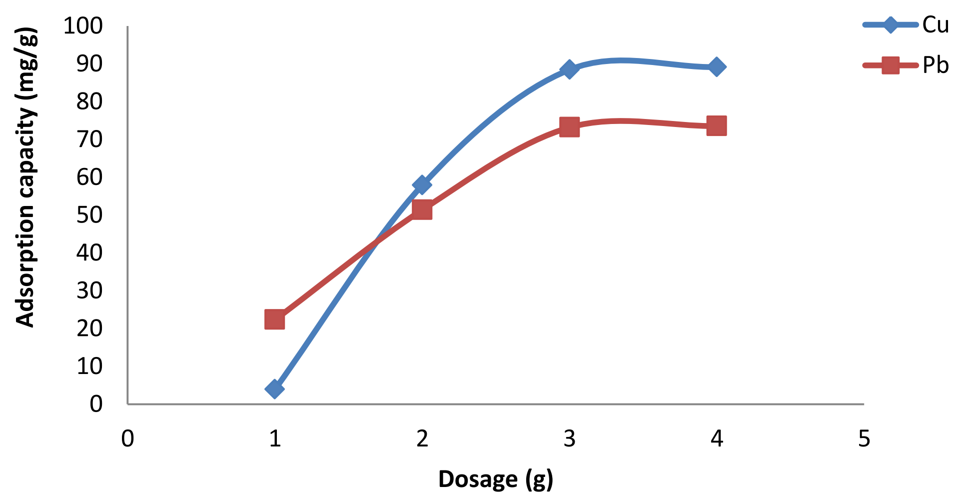

Fig . 4: Adsorption capacity against Dosage

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A: Waste local bamboo cut to size

B: Carbonized material after pyrolysis

C: Distillate obtained after pyrolysis

D: Activated carbon after chemical activation

Plate 1: Experimental procedure and products from Raw materials to finish