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Original Research Article

Comparison of Heavy Metal Adsorption by Activated Carbons Prepared from Cassava and Bamboo Biomass

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ABSTRACT

The utilization of cassava peels and waste bamboo as precursors for producing activated carbon for the adsorption of zinc (Zn^{2+}), nickel (Ni^{2+}) and cadmium (Cd^{2+}) from agricultural wastewater was investigated in this study. The biomass materials (cassava peels and waste bamboo) were carbonized at temperature range of 200 °C- 420 °C for a period of 90 min and 120 min for cassava peel and waste bamboo respectively. Activation with $ZnCl_2$ was done at impregnation percentage of 0, 33.3 and 66.6% for cassava peel carbon while 0, 10 and 20% was used for waste bamboo. The scanning electronic micrograph (SEM.EVO/MA15) was used to view the morphology of each adsorbent tested. The pH of adsorbents used was also studied based on impregnation ratio. pH of each adsorbent used increased with increase in impregnation percentage with bamboo activated carbon having a high pH value range of 6.8-7.2 as compare to cassava peel with 5.3-5.4 pH value. The pore space and surface area for impregnation percent of 66.6 and 20% for cassava adsorbent and bamboo adsorbent respectively had a combination of mesopores and macropores with prevalence of macropores indicating that higher percentage of $ZnCl_2$ increased the pore space. The cassava peel impregnated at 66.6% showed a greater removal efficiency of 66.67% for Ni^{2+} when compared to waste bamboo impregnated at 20% which has 100% adsorption efficiency for Ni^{2+}. Adsorption efficiency of other metals studied for both adsorbents fell in the range of 50-99%.

1. INTRODUCTION

Heavy metals in water, soil and plant are pollutants known to cause severe environmental problems like eutrophication and algae boom (FAO and IWMI, 2017). In the quest to combat these problems different mechanisms have been developed with adsorption techniques recently occupying a prominent position in operative heavy metal separation (Ijaola et al., 2013). Adsorption makes use of many porous media and activated carbon is seen as the most effective adsorbent in pollutant removal when it concerns water and wastewater (Ansari and Omidvari, 2005).

The adoption of agricultural by-products which are renewable and readily available source of raw material for activated carbon production is a common phenomenon. In recent times, agricultural by-products such as almond shell (Bansode et al., 2003), maize cob (Igwe and Abia, 2007), longan seed (Mopoung and Nogklai, 2008), rambutan seed (Norlia et al., 2011), flamboyant pond back, milk bush kernel shell and rice husk (Alade et al., 2012) bamboo, coconut shell and palm kernel shell (Ademiluyi and David-West, 2012; Olafadehan et al., 2012; Ijaola et al., 2013), have been investigated by various authors in the production of activated carbon.

Activated carbons are carbonaceous adsorbent materials that have been processed to have an extremely porous media with a very large surface area available for adsorption or chemical reaction (Williams and Reed, 2004). Activation of carbon can be achieved through physical or chemical activation methods. Physical activation involves carbonization of materials at high temperatures under an inert condition using gases like argon, nitrogen, followed by activation using mild oxidizing agent such as carbon dioxide (CO_2), oxygen (O_2) or steam at high pressure (Williams and Reed, 2004). Chemical activation is achieved by chemical impregnation of the carbon material under heat. During chemical activation process, the biomass is impregnated with specific ratio of acid, strong base or salt (phosphoric acid, potassium hydroxide, sodium hydroxide, zinc chloride etc.) or before carbonization (Adinata et al., 2007). Phosphoric acid and zinc chloride are the most commonly used for activating lignocellulosic materials (Ahmadpour et al., 1998; Puziy et al., 2002; Subhashree, 2011). Zinc chloride is a strong dehydrating agent which alters the structure of carbon materials producing porous surface with higher reaction sites for chemical reactions. However chemical activation is preferred over physical activation since it requires lower temperature and shorter time.

Heavy metals such as zinc, nickel and cadmium are trace element that plays essential roles in biological processes but at higher concentration they may be toxic to the environment. They are elements with variable oxidation states and co-ordination numbers. They also form complexes with organics in the environment and increase their mobility in the biota to manifest toxic effects (Sekaran et al., 1995). Kuniawan and Chan (2006) showed that inorganic effluent from the industries contains toxic metals such as Ni, Cu, Zn and Cd which tend to accumulate in the food chain, due to high rate of solubility in water and aquatic environment. Once in food chain in large concentration, they lead to bio-accumulation in the human body and can be associated with diseases such as cancer, hypertension and/or kidney diseases (WHO, 2020).

Cassava (*Manihot esculenta* Crantz) and Bamboo (*Bambusa vulgaris*) are abundant in Nigeria. Cassava is a major staple food and its production process leads to a large volume of waste which pose environmental nuisance. Bamboo is used in ornamental designs, but mainly used as support material in construction site most of which are left as constructional waste after project is completed

The research was aimed at investigating comparative efficacy of adsorbents produced from cassava peel and waste bamboo in the adsorption of heavy metals (Zn^{2+}, Ni^{2+} and Cd^{2+}), obtained from construction site as a precursor. The study also verifies the feasibility of activating carbon with $ZnCl_2$ at different percentage as it applies to the material in the adsorption of heavy metal (Zn^{2+}, Ni^{2+}, Cd^{2+}).

2. MATERIALS AND METHODS

2.1. Sample Collection and Preparation

Waste bamboo and cassava that were utilized as precursors to produce the adsorbents used in this study were collected from a construction site located within the University of Ibadan campus and from a cassava processing industry in Ibadan, Nigeria respectively. Waste bamboo was inspected, reduced to 20 cm size and washed to remove any form of impurities, after which the samples were soaked for 24 hr to remove traces of impurities that may still adhere onto the surface during process of its use on the construction site. The material was then subjected to sun drying until a moisture content of 8-10% wet basis was attained. The dried biomass was then carbonized using a muffle furnace (Carbolite, England model AAF 11/18) at a temperature of 350 ºC for 2 hr after which samples were cooled in an inert environment overnight (Ijaola et al, 2013). The cassava peels were inspected washed and sundried to a moisture content of between 8–10% wet basis. The dried cassava peels were then carbonized in a muffle furnace (Carbolite, England Model AAF 11/18) at a temperature of 420 ºC for a period of 90 mins as described by Omotosho and Sangodoyin (2013). The resulting carbon material was allowed to cool overnight under inert conditions. Carbonized samples (Plate 1 and 2) were then weighed and the yield percentages were calculated as follows:

$$Yield\ (\%) = \frac{w_1 * 100}{w_2} \quad (1)$$

Where:

W_1 = Mass of activated carbon.

W_2 = Initial mass of sample.

Plate 1: Carbonized cassava peels

Plate 2: Carbonized waste Bamboo

2.2. Activation of Samples

The charred samples were further reduced with the aid of mortar and pestle and sieved to 500 µm particle size. Three samples of cassava peel carbon were impregnated at 0, 33.3 and 99.9% respectively while impregnation of waste bamboo carbon was done at 0, 10 and 20% weight of $ZnCl_2$. Required concentration of $ZnCl_2$ was obtained by dissolving the required weight of zinc chloride in 100 ml of deionized water. After mixing the solution of $ZnCl_2$ with carbon (impregnation), complete dissolution of each weight percentage was done separately for each biomass by adding 150 ml of distilled water into the mixture to form slurry. These were left to stand for about 45 minutes to allow the $ZnCl_2$ gets enough contact time with the carbon. The samples were put in aluminum containers, covered with aluminum foil to prevent loss of $ZnCl_2$ and later placed in a hot air oven. Impregnated carbonized cassava peels (ICCP) were placed in oven for 50 min at 150 ºC while impregnated carbonized waste bamboos (ICWB) were placed in the oven for 2 hours at 200 ºC. The oven temperature for ICCP and ICWB were increased to 200 ºC for 60 minutes and 450 ºC for 4 hours respectively to ensure complete impregnation of $ZnCl_2$. After cooling, each sample was washed with slightly boiled deionized water to remove residual zinc chloride. This was then followed by washing with distilled water to pH of 6-7. ICWB and ICCP were then placed in the oven at 90 ºC for 6 hours and 110 ºC for period of 8 hr respectively. They were removed to be cooled at room temperature. The final product was kept in airtight containers

2.3. Characterization of Prepared Activated Carbon

The average particle size, bulk density, ash content, moisture content, pH of the activated carbons were determined according to previously described methods (Akpapunam and Matkakis 1981; Azim and Washab 1990). The pore space and surface area of the activated carbon were determined by scanning electron spectroscopy (SEM). The SEM was used to view the surface of the carbon at very high magnification of the carbon surface in order to view the pore space development and to reveal other information such as texture (external morphology), structural orientation and to some extent chemical composition.

2.4. Adsorption Column Experiments

Three fixed bed adsorption filters used were constructed using transparent plastic (Perspex) material of 3-4 mm thickness. The dimensions were 0.18 m length, 0.18 m breath and 0.65 m height. Each of the columns had a 140 mm diameter plastic tap fixed to its bottom for release of effluent. Wastewater collected from Awba dam located at the back of Faculty of Technology, University of Ibadan and at Abadina stream which is the confluence point for stream flowing from Moniya, NISSER, Orogun, Agbowo and Bodija market were applied by gravity from a pipe ending at the top of the column at different time respectively. The columns were connected to a main reservoir which was the collection point for filtrate from the column. Connection was made as shown in Figure 1. Known mass of ICCP and ICWB were placed into the filter bed at a depth of 0.16 m each alongside a bedding material at the top and bottom of the activated carbon which acts as a support and to prevent the release of activated carbon during staining process. The overlaid bedding was to prevent spattering as the water entered the column and ensure equalized flow of influent.

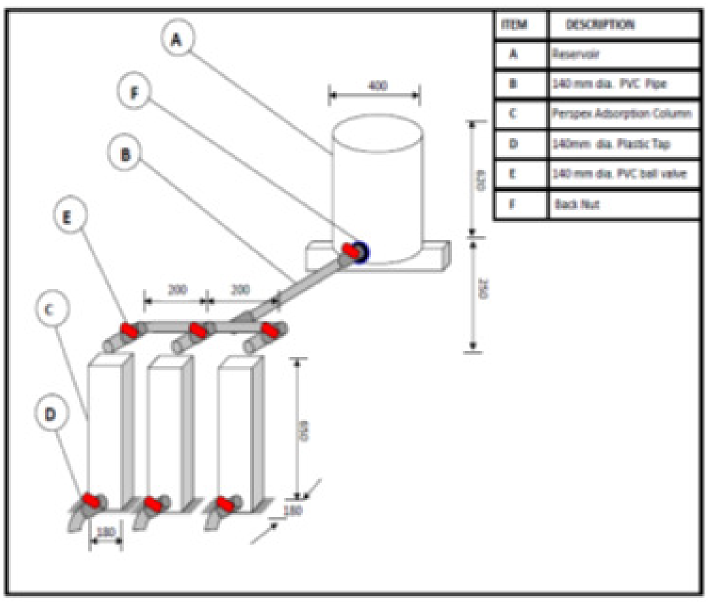

Figure 1: Adsorption bed assembly drawing for the three impregnate carbons (all units are in mm)

The valves were opened slowly such that the feed (wastewater) samples were allowed to flow from the reservoir into the top open of the filters through the beds by gravity. The feed sample was retained in each of the filters for 2 hr after which it was allowed to move down along the filtering bed. Filtration was done for 4 hours and the effluents collected at a two hour interval and then subjected to physico-chemical analysis of Zn^{2+}, Ni^{2+} and Cd^{2+} according to WHO, FEPA and EC standards. The efficacy of activated carbons in the removal of Zn^{2+}, Ni^{2+} and Cd^{2+} was determined using the difference between the initial and final concentration of the metal ions at end of each 2 hours. The percentage removal (D^i) was calculated mathematically as:

$$D^i = \left[\frac{(C_i - C_f)}{C_i}\right] x\ 100 \qquad (2)$$

Where C_i and C_f (mg/l) are concentrations of ions in wastewater before and after treatment respectively.

3. RESULTS AND DISCUSSION

3.1. Characterizations Results

From Table 1, it can be observed that for both materials, there was an increase in pH and this may be due to the fact that zinc chloride has the ability to attack metal oxide in the carbon to give a derivative. The effect of this is that ligands will be increased negatively and the binding effects of cations are raised (Parvathi et al., 2007; Ijaola et al., 2013). Inference from Table 1 also shows that as impregnations percentage is increased in both activated carbons, bulk density increased. Cassava peels at 66.6% had the highest bulk density while waste bamboo at 0% gave the least bulk density. This confirms that bulk density of carbons is affected by the raw material used and the degree of activation (Bansode et al., 2003). The trend of bulk density along the activation path shows that if percentage activation increases the bulk density will also increase this was a very important factor considered in the design of the adsorption columns as suggested by (Ijaola et al., 2013). Ash content within an activated carbon must be low for it to be a good adsorbent. High ash content will reduce the mechanical strength of the carbon and in turn affect the adsorptive capacity. Since the ash content is low, the adsorbent will show good adsorptive capacity. As temperature increases, moisture content decreases. The moisture content in each sample is small, hence the adsorption of moisture is less.

Table 1: Characterization of cassava peels and waste bamboo with different impregnation ratio

Biomass	Percentage impregnation (%)	pH	Bulk density (g/cm^3)	Ash content (%)	Moisture content (%)
Cassava peels	0	5.3	0.407	2.65	2.31
	33.3	5.4	0.410	2.38	2.21
	66.6	5.4	0.413	2.44	2.33
Waste bamboo	0	6.8	0.167	2.83	2.42
	10	6.9	0.177	2.67	2.41
	20	7.2	0.191	2.40	2.43

Results (Table 2) were obtained by subjecting 1.5 gram of impregnated carbonized waste bamboos (ICWB and) impregnated carbonized cassava peels (ICCP) to SEM. The obtained micrographs shown (Plate 3-8) were read with image J in order to get the pore area values. It can be deduced that percentage impregnation is directly proportional to increase in pore area. Comparing the two biomass materials employed, cassava peels with 66.6% $ZnCl_2$ impregnation has the highest average pore area followed by 33.3% of cassava peels, 20% $ZnCl_2$ impregnation and 10% $ZnCl_2$ impregnation for waste bamboo. The distribution of pore in an activated carbon depends on the raw material used and the activation ratio 20% activated waste bamboo is less with 46.6% when compared with cassava peels but still its average pore area is relatively high when compare to the cassava peels with high impregnation percent indicating that waste bamboo is more porous than cassava peels.

Table 2: Pore properties of the activated carbons

	Activation (%)	Average pore area (µm)	Average pore width (µm)	Average pore length (µm)	Carbon yield (%)
Cassava peels	0	166.60	10.39	13.78	34.63
	33	674.00	61.73	25.20	
	66	863.30	18.91	27.70	
Waste bamboo	0	50.45	172.30	15.00	34.10
	10	69.11	200.20	17.00	
	20	104.83	354.26	18.76	

Images obtained from SEM micrograph analysis as shown in plate revealed that the ICWB (0% $ZnCl_2$ activation) exhibited a net like structure with a thread like edge and a well open pore space. The surface configuration predicts a material which would have relatively good adsorptive characteristics see (Plate 3). However, in ICWB at 10% $ZnCl_2$ activation ratio the pore eye enlarged in length indicating an increase in pore area as a result of the activation, a closer look reveals that the netlike structure becomes uneven and unable to hold tie (Plate 5). This shows that there would be an improvement in adsorptive characteristics over ICWB at 20% (Plate 7) $ZnCl_2$ activation ratio, the ICWB still exhibited further increase in surface area resulting in a phenolic ring structure and increased reaction sites. Improvement is further observed and which is directly proportional to the adsorption intensity of the carbon material. A close observation of image from the micrograph of the ICCP surface as shown in Plate 4 reveals that pore space development on the carbon material was not very pronounced. Surface configuration is directly related to the adsorptive characteristics. Smooth adsorbent surfaces have been confirmed to offer lower adsorption characteristics when compared with adsorbents with rough configuration. At 33% $ZnCl_2$ activation, ICCP showed relatively larger pore opening characteristics when compared to the ICWB at 10% as shown in the spectrometry image (Plate 6). Thus, it can be predicted that the adsorbent ICWB would be more effective than cassava peel carbon in adsorbing materials. Micrograph analysis of the ICCP at 66% $ZnCl_2$ activation reveals that the material surface exhibited a hexagonal honeycomb structure (Plate 8). This theoretically indicates good adsorptive surface characteristics. The ICCP material surface shows a very obvious effect of chemical activation on the base carbon material. This further proves that the activation process was able open up the pores in carbons thereby increasing the adsorptive surface of each carbon tested.

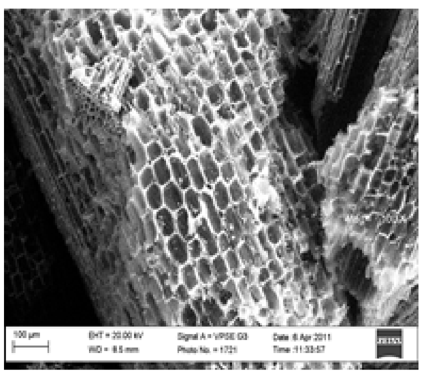

Plate 3: ICWB at 0% $ZnCl_2$ 100 MX

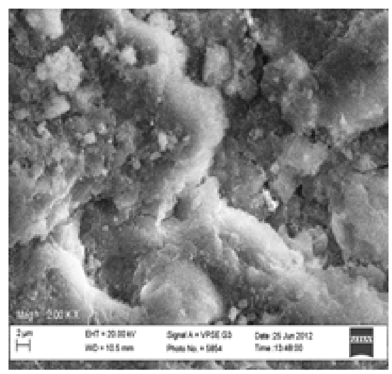

Plate 4: ICCP at 0% $ZnCl_2$ 100 MX

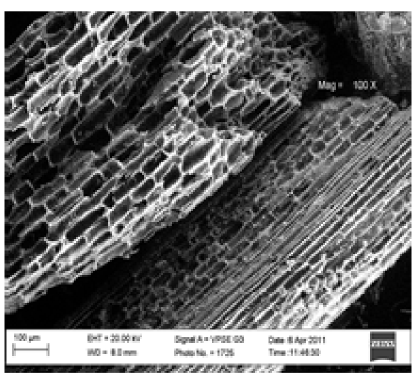

Plate 5: ICWB at10% $ZnCl_2$ 100 MX

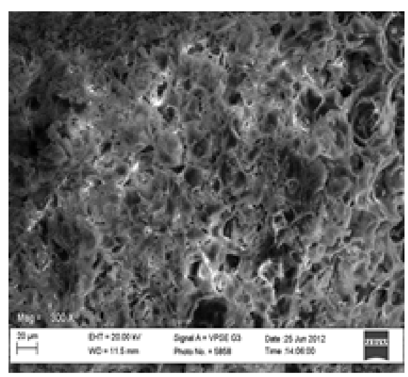

Plate 6: ICCP at 33.3% $ZnCl_2$ 100 MX

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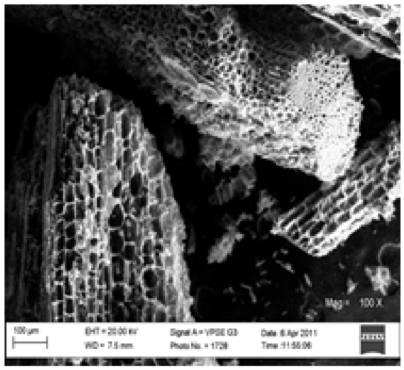
Plate 7: ICWB at 20% ZnCl2 100 MX

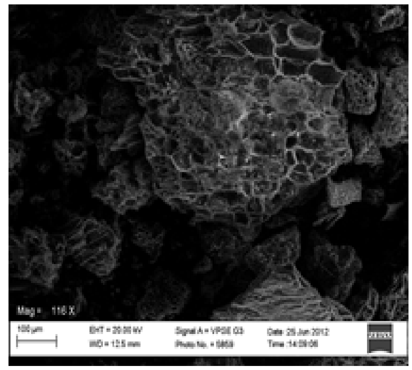
Plate 8 ICCP at 66.6% ZnCl2 100 MX

3.2. Adsorption Removal of Metal ions by $ZnCl_2$ impregnation Biomasses

Tables 3 and 4 showed the percentage removal efficiency of metal ions by ICCP and ICWB at different percentage of activation of $ZnCl_2$ for 2 and 4 hours. Unit of pollutant before and after treatment with activated carbon were recorded. Comparing the adsorption rate of Zn^{2+}, Ni^{2+} and Cd^{2+} in Table 3 and 4, the removal efficiencies of each biomass differ on the level of pollution, pore opening, raw material and $ZnCl_2$ impregnation level.

Table 3: Percentage removal efficiency of metal ions by ICCP and ICWB at different percentage of activation of $ZnCl_2$ for 2 hours

Biomass	Heavy metal	Initial concentration (mg/l)	Percentage removal at 0% $ZnCl_2$ (ICCP&ICWB)	Percentage removal at 10% $ZnCl_2$ ICWB	Percentage removal at 20% $ZnCl_2$ ICWB	Percentage removal at 33.3% $ZnCl_2$ ICCP	Percentage removal at 66.6% $ZnCl_2$ ICCP
Cassava peels	Zn^{2+}	0.14	28.51	-	-	56.4	66.2
	Ni^{2+}	0.03	30.8	-	-	92.3	95.4
	Cd^{2+}	0.02	28.51	-	-	92.5	95.3
Waste bamboo	Zn^{2+}	0.05	50	75	85.2	-	-
	Ni^{2+}	0.02	50	100	100	-	-
	Cd^{2+}	0.01	100	100	100	-	-

All parameters are in percentage (%) except for initial concentration (mg/l)

Table 4: Percentage removal efficiency of metal ions by ICCP and ICWB at different percentage of activation of $ZnCl_2$ for 4 hours

Biomass	Heavy metal	Initial concentration (mg/l)	Percentage removal at 0% $ZnCl_2$ (ICCP&ICWB)	Percentage removal at 10% $ZnCl_2$ ICWB	Percentage removal at 20% $ZnCl_2$ ICWB	Percentage removal at 33.3% $ZnCl_2$ ICCP	Percentage removal at 66.6% $ZnCl_2$ ICCP
Cassava peels	Zn^{2+}	0.14	33.6	-	-	89.4	92.3
	Ni^{2+}	0.03	72.4	-	-	93.4	92.5
	Cd^{2+}	0.02	65.0	-	-	99.3	99.5
Waste bamboo	Zn^{2+}	0.05	73.2	90.2	94.3	-	-
	Ni^{2+}	0.02	50	100	100	-	-
	Cd^{2+}	0.01	100	100	100	-	-

All parameters are in percentage (%), except for initial concentration (mg/l)

Waste bamboo biomass has affinity for Ni^{2+} and Cd^{2+} with 100% removal efficiency at 10% and 20% $ZnCl_2$ impregnation level for all contact time while 0% has removal efficiency of 50% for Ni^2 at all contact time and about 50-73.2% for Zn^{2+} at 2-4 hours contact time respectively. Cassava peel carbon material showed appreciable removal efficiencies for all tested metals ions with Zn^{2+} at 0% ICCP having 28.51% and 33.6% for 2 and 4 hours respectively. Ni^{2+} and Cd^{2+} had removal efficiencies ranging between 56.4-95.99.5% at contact time of 2-4 hr. Activation levels affect the pore enlargement and removal efficiency as more pollutant are removed from the wastewater when activated biomass is used as compared to non- activated biomass at the same contact time. Comparing the biomasses when activated, waste bamboo shows higher adsorption rate irrespective of the activation rate and this implies that waste bamboo has more chemical active sites than cassava peel carbon material. Another factor that may have contributed to waste bamboo having greater adsorption may be the bulk density as waste bamboo has a lower density as compare to cassava peels. Ademiluyi and David-West, (2012) reported that low density of bamboo carbon will aid fast adsorption of gas and liquid phase systems.

4. CONCLUSION

The study revealed the effectiveness of $ZnCl_2$ activation levels in pore opening for cassava peel and waste bamboo carbons in the adsorption of Zn^{2+}, Ni^{2+} and Cd^{2+} from wastewater. The opening of pore area by $ZnCl_2$ was based on raw materials used as carbon and the percentage activation level. The increase in bulk density and pH is due to increase in percentage activation while ash content reduces as activation levels increases; the trend for moisture content is not conclusive. The cassava peels with 66.6% activation shows greater enlargement of pore area while cassava peel carbon showed the least pore enlargement. The adsorption experiment shows that waste bamboo with 20% activation levels gave the best adsorption efficiency at 100% in adsorbing Ni^{2+} and Cd^{2+} as compare to cassava peels activated at 66.6% which shows the highest pore enlargement but having an adsorption efficiency ranging between 92. 5-99.55 % for Ni^{2+} and Cd^{2+} for all contact time. The result of adsorption experiment can be attributed to the fact that waste bamboo has high chemical active site than cassava peels.

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6. CONFLICT OF INTEREST

There is no conflict of interest associated with this work.

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