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Production and characterization of activated carbon from selected local raw materials

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Quality indices of activated carbons from cattle bone (CB), coconut-shell (CS) and wood carbon (WC) as influenced by mode of activation (heat and acid) were studied using the non-activated carbon samples as control. Preliminary investigation was carried out to determine optimum condition for carbonization and activations. The iodine values, ash, bulk densities, moisture, hardness and porosity analysis of the samples were evaluated using standard methods. Heat (H) activation was at 950°C for 3 h to produce heat activated bone carbon (HCB), and 850°C for 2¹/₂ h for heat activated coconut-shell (HCS) and wood carbon (HWC) while concentrated phosphoric acid (1:4) carbon: acid (w/w) was at 600°C for 2 h to produce acid activated wood (AWC), acid activated coconut-shell (ACS) and acid activated bone (ACB) samples. The activation yield for the acid activation ranged from 40.40 to 46.21% while heat activation ranged from 36.30 to 41.10%. Iodine values for heat activated carbons ranged from 828.39 (HCB) to 995.38 (HWC) mg/g. That of acid activated carbons ranged from 834.94 (ACB) to 914.17 (AWC) mg/g as compared with 66.2 to 222.7 mg/g for non-activated carbons (controls). Hardness of heat activated carbons ranged from 88.5 to 93.5% while that of acid activated carbons ranged from 62.0 to 89.5%. Bulk densities ranged from 0.40 to 0.79 g/cm³. The moisture for heat activated carbons ranged from 4.7 (HCB) to 8.4% (HWC) while that of acid activated carbons ranged from 5.0 (ACB) to 9.4% (AWC). The volume activities for the activated carbon samples ranged from 228.7(HCB) to 324.9 (HWC). The ash for bone carbons ranged from 80.6 to 85.9% while those of coconut shell and wood ranged from 1.1 to 3.5%. The carbons presented Brunauer, Emmett and Teller (BET) surface areas between 718 and 1018 m²/g, and micropores and mesopores with volumes between 0.004 and 0.776 cm³/g. This study indicated that efficient activated carbons could be obtained from local raw materials by controlled activation with acid or heat for home and industrial purposes.

Key words: Quality indices, optimum condition, carbonization, activation.

INTRODUCTION

The importance and relevance of activated carbon to an ever growing society cannot be overemphasized considering its enormous uses. Its uses range from liquid phase to gaseous phase applications. Activated carbons

are highly porous and adsorbent materials. They have wide applications in domestic, commercial and industrial settings (Mendez et al., 2006). In the food industry, activated carbon is used in de-colourization, deodorization and taste removal. It is used to remove heavy metals and organic contaminants from liquids. Activated carbon is used in water de-chlorination and processing of foods. It is also used in medicine for adsorption of harmful chemicals and drugs. In gas cleaning applications, activated carbon is extensively used in air filters at industrial level as well as in general air conditioning application (Oyo and Igbokwe, 2001; Diets, 1990), (Inamullah et al., 2008), (Elliot et al., 1989).

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ABBREVIATIONS: ACB, Acid activated bone carbon; ACS, acid activated coconut shell carbon; AWC, acid activated wood carbon; HCB, heat activated bone carbon; HCS, heat activated coconut shell carbon; HWC, heat activated wood carbon; CAC, commercial activated carbon.

Basically, there are two different processes for the preparation of activated carbon, the so-called physical or thermal and chemical activation. The first step is carbonization of the raw material and is performed in an oxygen free environment at temperature of about 700°C (Dileck and Oznuh, 2008). Carbonization means obtaining charcoal from the raw material and the produced charcoal having low surface area is not an active product. During the process of carbonization, volatile components are removed and the residual carbonaceous material is then activated in air at temperatures higher than 800°C, or in the presence of an activating agent at lower temperatures. According to Sang et al. (1997), activation can be physical or thermal with the use of air, carbon dioxide or water vapour at high temperature or a chemical process employing activating agents such as phosphoric acid (H₃PO₄) or zinc chloride (ZnCl₂). As reported by several authors, activation temperature significantly affects the production yield of activated carbon and also the surface area of activated carbon. The temperature used as low as 200°C (Haimour and Emeish, 2006). In chemical activation, the raw material or carbonized sample is impregnated with an activating agent and heated in an inert atmosphere (Srinvasakannan and Mohammad, 2004). During the activation process, the spaces between the elementary crystallites become cleared of less organized, loosely bound carbonaceous material. The resulting channels through the graphitic regions, the spaces between the elementary crystallites, together with fissures within and parallel to the graphitic planes, constitute the porous structure, with large internal surface area (Rodriguez and Solano, 1989). Sang et al. (1997) reported that at temperatures above 1000°C, the reaction in the pores become prohibited and limiting the reactions to the outer layer of the carbon particle. Guo and Lua (2001) mentioned that the characteristics of activated carbon depend on the physical and chemical properties of the raw materials as well as activation method used.

The physical properties of carbon are important in determining which form is best suited for specific application. For instance, abrasion resistance or hardness of activated carbon will be important if the carbon is to be used in application where frequent back-washing is required. Density characteristics can also be a major consideration for specific applications. Densities vary with the raw materials. A fewer quantity of carbon with a lower density will fit into a given container as compared to a carbon with high density. This is significant because while a container may require less carbon weight of low density to make a fill, its contaminant removal performance may be severely reduced as compared to a high-density carbon. Iodine value is an important characteristic of activated carbon. The iodine value gives a measure of the micro pore volume of carbon and it approximates the total internal surface of the carbon. The ash level reflects the purity of

the carbon. The ash content can be important in water filtration applications. Activated carbon with high phosphate ash can bring about cloudy water during use because the ash could combine with metal ions to form magnesium or calcium precipitates (Ecologix systems, 2008). The performance of activated carbon is indicated by its adsorptive characteristics, which is derived from the specific surface area, porosity of the carbon (Akinyemi and Taiwo, 2004). Micro pores are formed in the interlayer spacing with widths in the range of 0.34 – 0.8 nm. It is the micro pores in activated carbon which have the greatest influence upon gas adsorption while macro pores and mesopores are important in transport of fluids to and from the micro pores (Hu and Srinivasan, 2001). The knowledge of the physical and activity characteristics of activated carbons will help in determining where each can be better applied. The goal of the study was to produce and characterize activated carbon from cattle bones; coconut-shell and wood.

MATERIALS AND METHODS

Source/Sample preparation

Coconut shells, waste pieces of wood *Prosopis africana* were respectively collected from coconut and wood sellers in Idah, Kogi state, Nigeria while cattle bones were collected from an abattoir opposite Federal Polytechnic, Idah, Kogi state, Nigeria. Each material was crushed manually to small chips of approximately equal sizes (1.0 to 0.8 cm). A commercial activated carbon (Chemivon, Sussex, UK) were purchased from Charltech Laboratories, Makurdi, Nigeria.

Carbonization and activation

Carbonization was as described by Gimba et al. (2001). The carbonization time was varied between 0.5 and 3 h at 600°C to determine optimum carbonization for each raw material by fixed carbon measurement without ash formation. Acid activation was carried out as described by Tao and Xiaoqin (2008). The final carbons were weighed after drying to determine the final product yield:

$$\% \text{ Final product yield} = \frac{\text{weight of carbon from furnace after drying}}{100} \times 100 \quad (1)$$

Analytical methods

Iodine values were determined as described by Okuo et al. (2008). This involves the titration of 0.1 M sodium thiosulphate solution against 60.0 ml of activated carbon sample free aliquot solution (prepared by centrifuging 0.5 g of activated carbon sample in 75.0 ml of 0.086 M iodine solution using 5 ml of freshly prepared starch indicator. The iodine value (IV) was calculated using the formula:

$$IV \text{ (mg/g)} = \frac{Y - X}{V} \times W \times M \text{ (126.9)} \quad (2)$$

Where Y = volume of thiosulphate for blank, X = Titer value, V = volume iodine solution used, W = Weight of sample. M = molarity of iodine solution.

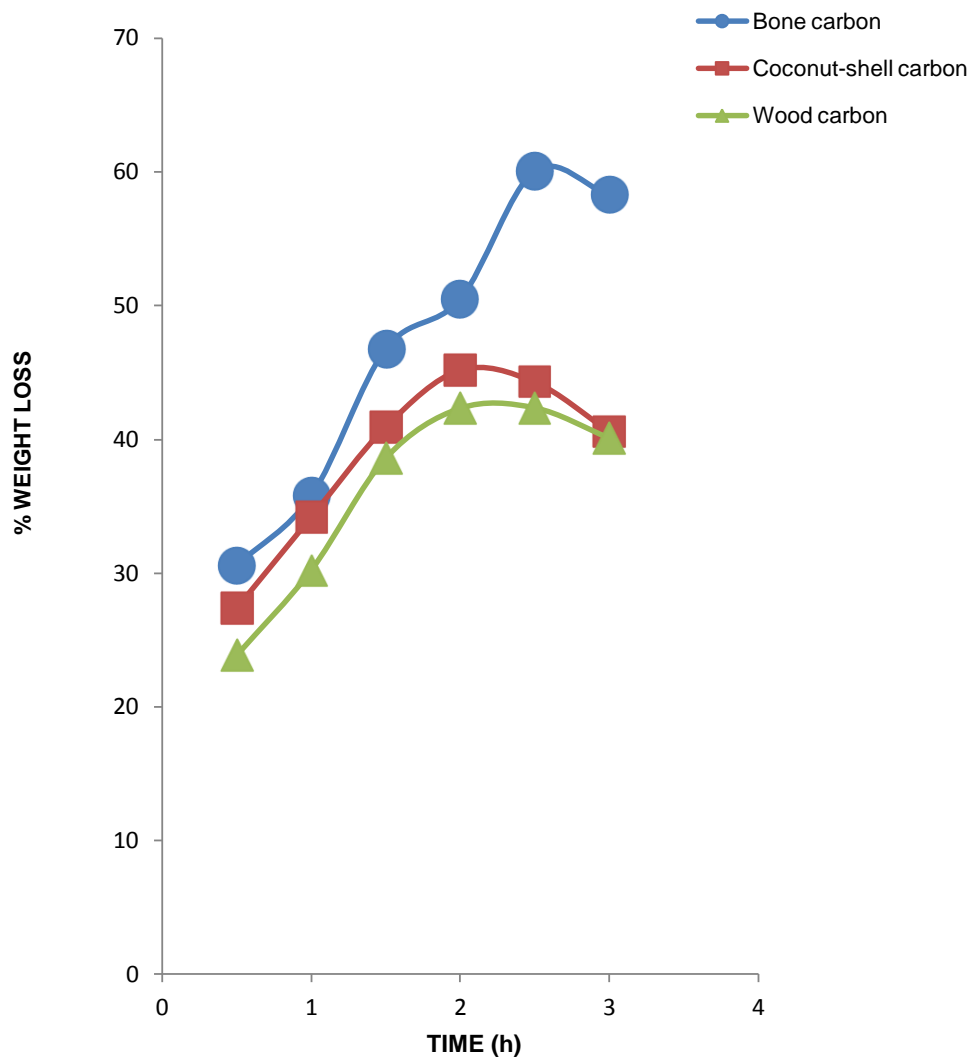


Figure 1. Effect of carbonization time on carbon weight loss.

Carbon hardness was determined according to ASTM (2002) method. This involves measuring 2 g of carbon sample in a standard sieve no 40. The sieve was shaken for 1h using mechanical shaker (Millano Italia-type) at 160 strokes per minutes with four steel balls (6.0 mm diameter each) inside the sieve. The amount of activated carbon retained in the sieve after 1h was expressed in percent. Bulk density was determined according to the method of Okaka and Potter (1976). Five-gram quantity of each activated carbon was weighed in a graduated glass cylinder and tap until a constant volume is noted. The density was calculated in g/cm^3 . Moisture content was determined by the air-oven method as reported by AOAC (1994). Ash content was determined according to AOAC (1994).

Surface area and porosity characterization

This was performed by the adsorption of N_2 at 77K using micrometric surface area analyzer (ASAP2010) Micrometric Inc. USA. Before the adsorption of N_2 , the samples were subjected to degasing for 2 h at final pressure of 133.32×10^{-4} pa. The total pore volume (V_T) was estimated from the volume of N_2 (as liquid)

adsorbed at relative pressure of (p/p°) of 0.95. The microspore volumes were calculated from adsorption isotherms and mesopore was calculated as the difference between macro pore volume and micro pore volume. Barrett, Joyner, Halenda (BJH) method was employed for the evaluation of the pore size distribution (Results not shown).

Statistical analysis

Experimental results were analyzed statistically. Means were compared using test of significant difference (Steel and Torrie, 1980).

RESULTS

Carbonation/activation effects

Figure 1 shows the results of percentage weight loss for activated raw materials during carbonization at 600°C in

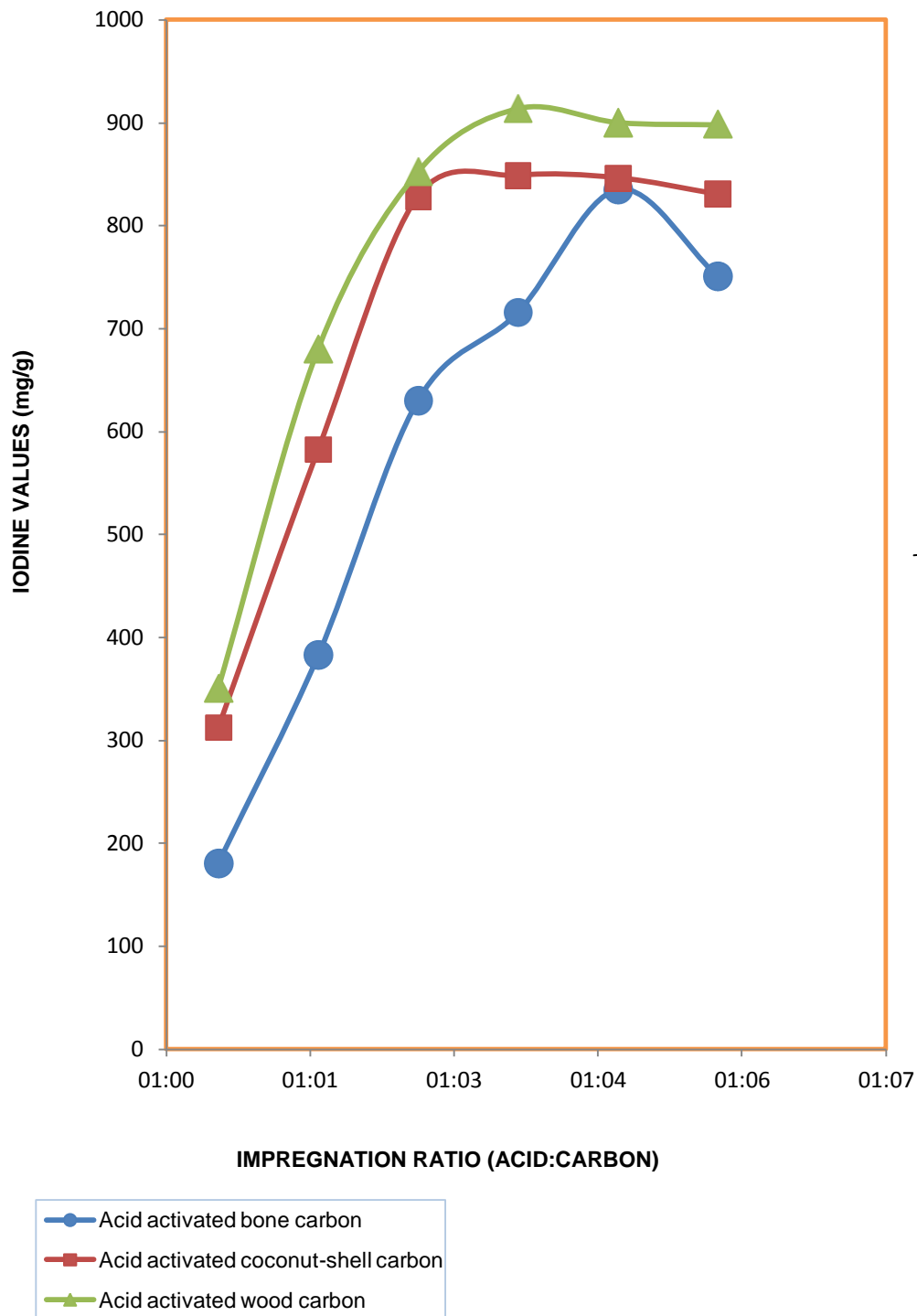


Figure 2. Effect impregnation ratio on iodine values of acid activated carbon.

a furnace. Increase in carbonization time resulted increased in weight loss for all the carbon samples. The increased in weight loss reached a maximum value at a carbonization time of 2 h for coconut-shell and 2.5 h for wood and for bone. Figure 2 shows the results of iodine values for activated carbons using phosphoric acid

activation. Increase in impregnation ratio resulted increased in iodine values for all the carbon samples. The increased in adsorptive capacity reached a maximum value at an impregnation ratio of 1:3 for coconut-shell and wood and 1:4 for bone carbon. Figure 3 shows the variation of iodine values with time using heat (thermal)

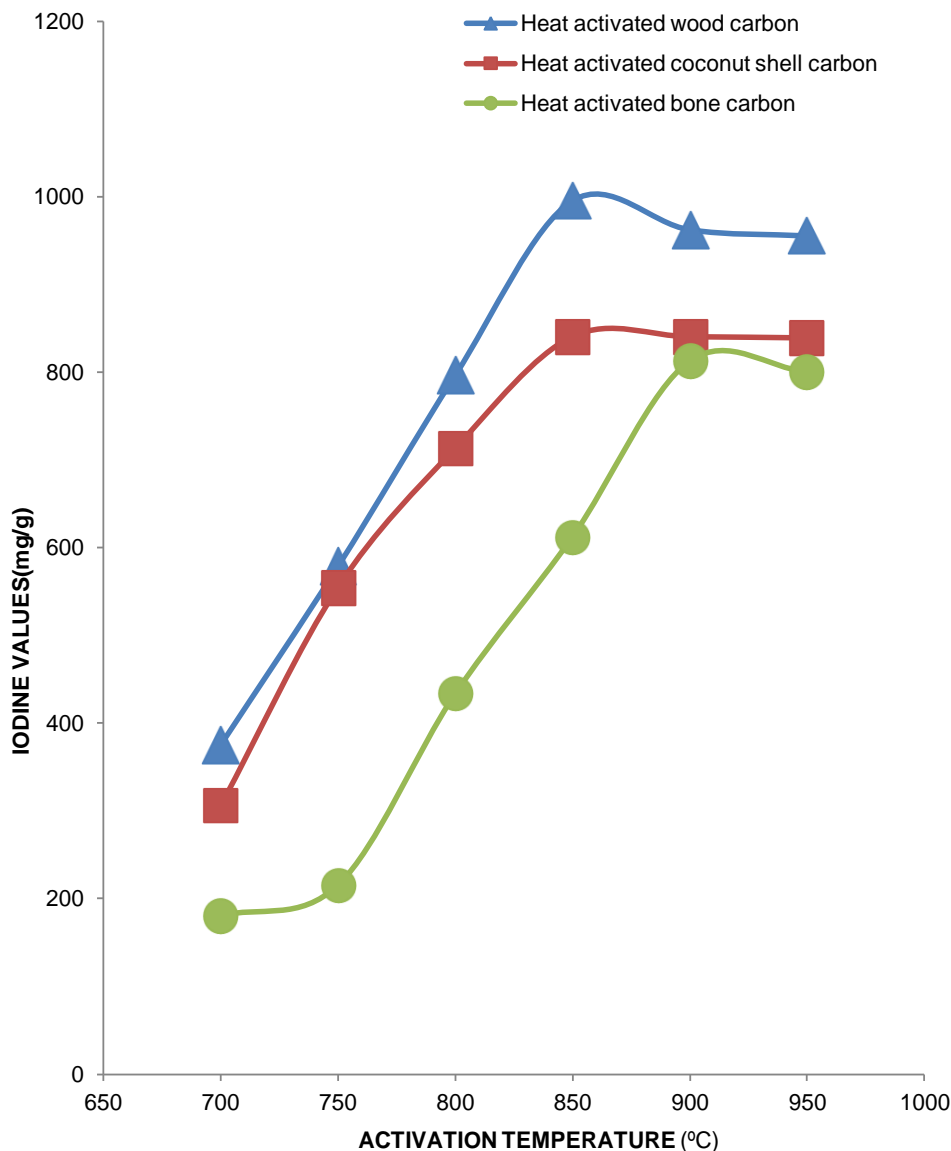


Figure 3. Effect of activation temperature on iodine values of heat activated carbon.

activation. Increase in carbonization temperature resulted in increased iodine values for all the carbon samples. The increased iodine values reached a maximum value at activation temperature of 850°C for coconut-shell and wood carbon and 900°C for bone. The results of final activation yield and volatile constituents of carbon raw materials after activation processes are shown in Table 1. The percent carbon yield for heat activated bone, coconut-shell and wood were 40.10, 39.30, and 36.30% while for acid activation percent fixed carbon were 46.21, 40.60 and 40.4, respectively. The volatile constituents were 60.2, 58.9, 63.1, 53.8, 59.2 and 58.6% respectively. The fixed carbon content of bone and wood were significantly affected by method of activation. The volatile constituents were not significant by carbon source and

mode of activation.

Iodine values and ash

The iodine values and ash content of acid and heat activated carbon are shown in Table 2. The iodine values were significantly ($P \leq 0.05$) affected by heat and acid activation process. The non-activated carbons had iodine values (66.16 to 222.69 mg/g) which is significantly lower than (828.39 to 995.38 mg/g) and (834.94 to 914.17 mg/g) for heat and acid activated carbons respectively. The heat activated wood carbon had significantly higher iodine value than the acid activated wood carbon. There is no significant difference between iodine values of bone

Table 1. Activated carbon yield and volatile losses.

Material	Treatment	Constituent (%)	
		Carbon	Volatile
Bone	Acid	46.21a	53.76a
	Heat	41.10b	60.20a
Coconut shell	Acid	40.62b	59.20a
	Heat	39.30b	58.91as
Wood	Acid	40.40b	58.60a
	Heat	36.30c	63.10a

Values are means of triplicate determinations. Means within a column with the same letters are not significantly ($P \leq 0.05$) different. Acid : Carbon ratio was varied between 1:1 and 1:6 to determine the optimum activation for each of the raw material by iodine value measurement. Heat : Heat activation temperature was varied between 700 to 950°C to determine optimum activation by iodine value measurement.

Table 2. Physico-chemical characteristics of carbon samples.

Material	Treatment	Physicochemical characteristics				
		IV	Ash (%)	Hardness (%)	BD	Moisture (%)
Bone	Acid	834.94a	82.4b	89.5b	0.79a	5.0a
	Heat	828.39a	80.6c	93.5a	0.78a	4.7a
	Non	66.15b	85.9a	90.0b	0.78a	4.6b
Coconut shell	Acid	848.69a	2.8b	81.0c	0.68a	6.4a
	Heat	840.18a	2.4b	96.5a	0.66a	5.1
	Non	125.67b	3.5a	90.5b	0.60a	4.4b
Wood	Acid	914.17b	1.8a	62.8b	0.47a	9.4a
	Heat	995.38a	1.1b	88.5a	0.48a	8.4b
	Non	222.69c	2.1a	87.5a	0.40a	7.3c

Values are means of triplicate determinations. Means within a column with the same letters are not sig. ($P \leq 0.05$) different. IV = Iodine values, BD = bulk density, ash, hardness and moisture were determined using standard methods.

and coconut-shell carbons in both activation processes. The total ashes for bone, coconut-shell and wood carbon were 89.9, 3.5 and 2.0%, respectively. The total ash for acid activated carbons was higher than those of heat activated carbons. For both activation methods, the ash for the bone carbon was higher than those of coconut-shell and bone wood carbon.

Hardness, density and moisture

Hardness, carbon density and moisture values are presented in Table 2. The hardness of the non-activated carbons was 90.0, 90.5 and 87.5% for bone, coconut-shell and wood respectively. The hardness of the carbons increased during heat activation and decreased during acid activation. The increase was significant for bone and coconut-shell carbon. The hardness of heat activated carbons decrease in the order: coconut shell < bone <

wood and the acid activated carbon in the order: bone < coconut-shell < wood. The density of the non-activated was 0.78 g/cm³, 0.60 g/cm³ and 0.40 g/cm³, respectively for bone, coconut-shell and wood carbon. Generally, activations increased densities of the carbons and there was no significant ($p \geq 0.05$) difference in densities.

The moisture content was 4.6, 4.4 and 7.3% for bone, coconut-shell and wood, respectively. The moisture content increased during activation processes. There was no significant difference between bone carbon activated with acid and bone carbon activated with heat. Acid and heat activated wood carbon had significantly higher moisture than coconut-shell and bone carbon.

Activated carbon porosity

The results of BET surface area, macropore, mesopore and micropore volumes of the produced activated carbon

Table 3. Porosity analysis of produced activated carbons and a commercial activated carbon.

Samples	S _{BET} (m ² /g)	Porosity parameter		
		V _P (cm ³ /g)	V _{micro} (cm ³ /g)	V _{meso} (cm ³ /g)
ACS	912c	0.711a	0.746a	0.025a
HCS	895d	0.768a	0.728a	0.040a
AWC	1018a	0.679a	0.663a	0.016a
HWC	1015b	0.779a	0.771a	0.008a
ACB	782e	0.662b	0.641b	0.021a
HCB	718f	0.656b	0.652b	0.004b
CAC	1022a	0.782a	0.770a	0.012a
LSD (0.05)	5.175	0.115	0.113	0.083

S_{BET}= BET surface area, V_P= Macropore, V_{micro}= Micro pore, V_{meso}= Mesopore volume.

and the commercial activated carbon are presented in Table 3. The surface area for the carbons ranged from 718 to 1018 m²/g. The activated carbons differed significantly in surface area. There were no significant difference in surface area of CAC and AWC. The macropore and mesopore volume were statistically the same for wood and coconut shell carbon. The activated carbon from bone presented significantly lower macropore and micropore volumes. The mesopore volume of heat activated carbon was significantly lower than mesopore volume for all the carbons.

DISCUSSION

Characterization of activated carbons

The results of percentage weight loss for activated raw materials during carbonization presented in Figure 1 shows that increase in carbonization time resulted in increased weight loss for all the carbon samples. The increased in weight loss is as a result of increase volatilization (loss of volatile matter) of heat labile substances which reached a maximum value at a carbonization time of 2 h for coconut-shell and 2½ h for wood and bone. The reduction in weight loss after the maximum values is an indication of the beginning of activation of the already formed carbon. Similar observations were reported (Oyo and Igbokwe, 2001), Sang et al. (1997) for other materials. According to these authors, during carbonization, a primary pore structure is developed followed by development of porosity and reduction in weight losses.

The results of iodine values for activated carbons using phosphoric acid activation at 600°C (Figure 2) indicated that increase in impregnation ratio resulted in increase in iodine values for all the carbon samples. The increase in iodine values is a result of development of porosity in the carbons which reached a maximum value at material to acid ratio of 1:3 for coconut-shell and wood and 1:4 for bone. The dropping of adsorptive capacity of the carbons

after the maximum values is an indication of the beginning of structural deformation of the already formed carbon. In a related study, Thanitha (2005) reported an optimum condition of activation of bone: zinc chloride (1:3 w/w) equivalent to 120% of impregnation ratio at 800°C with 3 h of holding time which gave a yield of 44.8% with iodine number of 901 mg/g.

Heat activation (Figure 3) shows that increase in carbonization temperature resulted in increase in iodine values for all the carbon samples. The increase in iodine values is due to development of pores and increased porosity in the carbons, which reached a maximum value at activation temperature of 850°C for coconut-shell and wood carbon and 900°C for bone. The reduction in adsorptive capacity of the carbons after the maximum values is an indication of the beginning of structural deformation of the already formed activated carbons. This observation is in conformity with those of Ajayi and Olawale (2009) who observed that in the production of activated carbon from *Canarium schweinfurthii* Nutshell using chemical process, when the temperature increased from 600 to 900°C, the iodine values increased. It is evident that the activation temperature had a significant influence on the iodine number. The iodine number was observed to increase at higher activation temperatures. However, at activation temperature around 950°C and above the iodine number practically decreases. Thus it is deduced that the optimum activation temperature is around 850 - 900°C for H₃PO₄ activation which is also considerable in terms energy saving. The optimum condition were similar to those suitable for corn cob (Tsai et al., 2001), various nutshells, chickpea husk and cork waste (Jun'ichi et al., 2002a, b; Carvalho et al., 2004).

The activated carbon yield (Table 1) for heat activated bone, coconut-shell and wood were 40.10, 39.30, and 36.30%, respectively while the yield for acid activation were 45.21, 40.60 and 40.40% respectively. These results are in agreement with the findings of other workers (Palnut, 2000; Savova et al., 2001; Aygum et al., 2003) who reported carbon yield of 39.99 to 55.44% for most activated carbon raw materials. It can be seen that

the local raw materials studied in this present work have high carbon yield. Product yield is an important measure of the feasibility for preparing activated carbon from a give precursor. According to Guo and Rock straw (2006), Guo and Lua (2011), significant product yield differences can be observed depending on the origin of carbon material and activation process.

The results shown in Table 2 indicated that iodine values were significantly affected by activation for all the carbons. The results indicates that acid is the best activating agent for both bone and coconut-shell carbon while heat is the best activating agent for wood carbon. In all cases, the iodine values of the carbons decrease in the order: wood < coconut-shell < bone. The iodine values reported in this study is in conformity with iodine values 600 to 1100 mg/g for water treatment carbons reported by Thanittha (2005). The observed analytical differences in iodine values could be attributed to differing adsorptive characteristics, which is derived from the specific surface area, pore size and pore volume of the carbon (Akinyemi and Taiwo, 2004). When appropriate activation conditions are provided, the oxidizing action of the oxidizing agent does not consists of indiscriminate removal of successive layers of atoms from the carbon matrix. Instead, the oxidation selectively erodes the surface in order to increase the surface area, develop greater porosity and leave the remaining atoms in the carbon matrix arranged in configurations that have specific affinity for particular adsorbents (Hassler, 1988).

The ash results show that activation reduces the ash levels of carbon. The reduction in ash could be attributed to volatilization of some inorganic constituents at higher temperature during activation that could lead to a reduction in ash and also acid could react with the mineral constituents of carbon and thereby reducing the ash during activation. Acid treatment was reported to have been used to reduce high ash levels of sub-bituminous and lignite carbon (Calgon carbon corporation, 2008). Acid activated carbons had higher total ash than the heat activated carbons. These could be attributed to acid residuals, which may remain with the carbon as part of the ash. This is in consonance with the results reported for sub-bituminous and lignite carbons treated with acid (Calgon Corporation, 2008). In both activation methods, the bone carbon had higher ash than the wood and coconut-shell. The higher ash content of bone carbon could be attributed to high inorganic component of bone charcoal (Ebonex Corporation, 2006).

The heat activated carbons shows higher hardness than the acid activated carbons (Table 2). This suggests that higher temperatures treatment had significant effect on hardness of carbon materials. The hardness ranged from 62.00 to 96.5%, indicating high abrasion resistance and could be applied where frequent back-washing is required. The result of hardness for coconut-shell activated carbon is in consonance with the hardness of 95.0 to 99.0 reported for granular coconut - shell

activated carbon (Ecologix systems, 2008). The densities of the carbons increase in the order: bone; coconut shell; wood. The difference in densities of the activated carbons can be attributed to differences in the carbon origin. The densities observed in this work are in close agreement with values of 0.35 to 1.2 g/m³ reported for other agricultural raw materials by Oyo and Igbokwe (2001), Bansal et al. (1988).

The activated carbons exhibited different moisture uptake abilities following activations. Moisture uptake increased in the order: bone; coconut-shell; wood irrespective of activation method. This observation is in agreement with studies by Bansal et al. (1988) who reported moisture contents of 10% for acid activated wood and 5.0% for granulated acid activated coconut-shell carbon. The porosity analysis presented in Table 3 shows that the surface area for the activated carbons fall within the range of 600 to 1200 m²/g reported for most widely used commercial activated carbons (Ng et al., 2002). The high surface area of the carbons is important since it limits the amount of adsorb ate which can be adsorbed.

The surface areas were higher for acid activated carbons and this is expected because chemical activation normally develops more porosity and gives high surface area when compared with thermal activation (Ajayi and Olawale, 2009). The increase BET surface areas and micropore (0.746 to 0.641 cm³g⁻¹) structure of acid activated carbons was brought about by oxidation of the carbons by phosphoric acid. The surface areas (718 to 1018 m²/g) reported for this study was higher than surface areas (305 to 539 m²/g) reported for dew and enzyme retted flax shives activated with phosphoric acid (Marshall et al., 2007). The macropore volume of the carbons ranged from 0.779 for HWC to 0.656 cm³g⁻¹ for HCB and the mesopore volume ranged from 0.008 for HWC to 0.040 cm³g⁻¹ for HCS. The micropore and mesopore volumes of the carbons are comparable to micropore of 0.058 cm³g⁻¹ and mesopore 0.453 cm³g⁻¹ reported for activated carbon from brewer's spent grain (Mussatto, 2009).

Conclusion

This study indicates that efficient activated carbons can be obtained from bone, wood and coconut shell by controlled activation with phosphoric acid or heat for a number of industrial and residential applications.

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