

Adsorptive Property, Ash Content Analysis of Activated Carbon Derived from Three Nigerian Plants: Water Hyacinth (*Eichhornia crassipes*), Iroko (*Chlorophora excelsa*) and Gmelina (*Gmelina arborea*)

Luke N. Ukiwe* • Emeka Oguzie • Chukwuemeka Ajaero

Department of Chemistry, Federal University of Technology, P.M.B 1526 Owerri, Nigeria

Corresponding author: *luggil2002@yahoo.com

ABSTRACT

The adsorption property and ash content of activated carbon (AC; equivalent to activated charcoal) derived from water hyacinth (*Eichhornia crassipes*), iroko (*Chlorophora excelsa*), and gmelina (*Gmelina arborea*) were studied. AC prepared from water hyacinth resulted in the highest percentage of decolorisation (58%), gmelina (31%) while that prepared from iroko resulted in the lowest percentage (10%) at 360 nm using the UV/visible spectrophotometry. Overall, from 360 nm to 460 nm at 20 nm intervals, water hyacinth AC produced the highest percentage of decolorisation followed by gmelina AC, then iroko AC. Total ash content analysis indicated that water hyacinth AC was highly adulterated and impure (3.53 mg/L), followed by gmelina AC (2.68 mg/L) while that from iroko (1.82 mg/L) was the least adulterated with a lower degree of impurities.

Keywords: adulteration, activation, decolorisation, hydrophobic, oleophilic, UV/visible spectrophotometry

INTRODUCTION

Activated carbon (AC), also referred to as activated charcoal) is a natural organic material with a high carbon content such as coal, wood, peat, etc. AC is amorphous carbon that has been treated with steam and heat until it has a very great affinity for absorbing materials (George 1984). AC could be in powdered-granulated or pelleted form characterized by very large surface area per unit volume due to enormous number of fine pores, which makes it capable of collecting gases, liquids or dissolved substances on the surface of these pores.

AC first came into prominence through its use as an adsorbent in gas masks in World War I. But it was not until the fifteenth century that the knowledge that carbon produced by the decomposition of wood can remove colouring matter from solutions became appreciated. The first commercial application of charcoal filters was in 1794 when it was used in a British sugar refinery. Activation of raw materials is accomplished by two basic processes: (i) Chemical activation and (2) gas oxidation. AC is very stable due to its carbon-carbon double bonds. The carbon atoms form flat layers. It is relatively inert and insoluble in all common substances such as water, acids, petrol, etc. They have a hydrophobic and oleophilic surface (Davidson 1970). AC can be divided into two main classes: those used for absorption of gases and vapours, for which granular (~0.8-4 mm) materials are used and those used for purification of liquid for which a powdered (0.25 mm) material is best suited. AC prepared from coconut shell and lignite have similar diameter and particle length but slightly different ash content and surface area. This difference resulted from the method used to activate the starting source material and from the final application of the substance (Henning and Degel 1990). It should be noted that studies aimed at using plant materials for the preparation of AC are rare.

Ash

Ash is the substance that remains after any material has been burnt. The plural form of the term (ashes) usually applies to the minerals obtained by burning wood, coal, or other carbonaceous materials. The amount of minerals in certain substances can be determined by analyzing the ash left when they are burnt. Ash is composed chiefly of earthy mineral particles. Ash content of AC is the residue remaining after all combustible matter in the AC has been completely burnt away and the residue denotes the quality of mineral matters contained in the liquid under test.

Ash content is a chemical property relating to quality indexing. Analysis of ash content fundamentally enables the scientist to identify the degree of purity of the AC and also detect the extent of adulteration. Effective control of the application of AC is ensured if the ash content is known. High ash content signifies a high presence of inorganic matter, including silicates. Total ash is a widely used acceptable index for refinement of AC. Adequate levels of total ash are indicative of functional properties of some carbonaceous materials as starting materials for the preparation of AC.

The seasonal variations of foliar carbon isotope discriminating potassium concentration and ash content in sand-fixing plants were investigated in three dominated desert plants (*Artemisia ordosica*, *Hedysarum scoparium* and *Caragana korshinskii*) during the growth season in Shapotu, a Southeast margin of the Tengger Desert in China. Results showed that there were significant differences in ash content in *C. korshinskii* but *A. ordosica* and *H. scoparium* had similar ash content; these differences were due to the physiological features of these plant species (Zhao *et al.* 2007). In another investigation to determine the normal ash content of legume grass silage and hay samples, it was observed that some legume grass forages contained up to 18.0% ash while hay samples contained up to 10.0% ash (Hoffman and Tayson 2007). In general, it was shown that hay samples

have less ash than legume grass forages. The authors claimed that the reason for the high ash content was likely due to contamination with soil. New forage harvesting and storage practices methods may draw soil into the forage or forages that are lodged due to heavy rains and wind and these forages may be more contaminated with unwanted soil. Forages stored in silage bags on earthen bases may result in soil contamination in the loading process and this may contribute to crops with high ash content. Studies on differential distribution of ash in stems (base to tip) of six different species of the herbaceous rose plant, *Rosa multiflora*, developed from seed grown under uniform conditions for four months in a greenhouse in spring and winter showed that there was an increasing ash gradient on the basis of dry weight when samples of stem tissue were taken from the base to the tip of the plant. The increase in ash content is correlated with the gradient in direction of growth of the plant (Edgecombe 1999). Two corn plant samples, one cut in autumn while green, and the other after weathering over the winter, exhibited strikingly different combustion properties that affected their ash content. The increase susceptibility to flaming combustion of the spring-harvested sample was attributed to its decreased ash content (Broido *et al.* 1964).

The specific objective of this study was to prepare AC with good decolorizing properties from readily available local plant materials; water hyacinth (*Eichhornia crassipes*) a grass, iroko (*Chlorophora excelsa*) a hardwood, and gmelina (*Gmelina arborea*) a softwood. The ash content was determined with a view to ascertain the absorptive capacity of the AC.

MATERIALS AND METHODS

Preparation of AC (water hyacinth)

Water hyacinth plant was harvested in a local river (Otamiri River) near Owerri municipality in Imo State, Nigeria. Only the matured plant with a thick, fibrous stem and root were harvested. The leaves were removed and the plant was washed and cut into pieces and dried in an Akson Scientific Oven for 48 h at 110°C until constant weight to drive off moisture. The dried material was further ground and pulverized and 500 g of the substance was mixed with 5 g of NaCl and placed in a Hanna muffle furnace model AS60S for carbonization in limited supply of air at 200-300°C for 30-45 min. The carbonized material was removed from the furnace and placed in a desiccator to cool. After 4 h, the carbonized material was withdrawn from the desiccator and activated using nitric acid at a 1:2 ratio; 1 g of carbonized material was mixed with 2 mL of concentrated nitric acid and heated to 150-200°C for 30 min in the muffle furnace. The resultant material was placed in a desiccator for 4 h to cool and then withdrawn, washed several times with deionized water and dried in a desiccator. This AC sample was denoted AcWh (activated carbon water hyacinth).

ator for 4 h to cool and then withdrawn, washed several times with deionized water and dried in a desiccator. This AC sample was denoted AcWh (activated carbon water hyacinth).

Preparation of AC (iroko and gmelina)

Samples of these woods were collected at a timber market in Owerri, Imo State, Nigeria. The samples were cut into pieces and dried at 110°C in an oven as above for water hyacinth for 48 h. 80 g of each sample was weighed and mixed with 5 g of NaCl and carbonized separately in a muffle furnace as above at 30-35°C for 2 h. The carbonized materials were kept in a desiccator to cool.

Activation of the samples was done by mixing the carbonized materials with concentrated nitric acid in a 1:2 ratio, i.e. 1 g of sample mixed with 2 mL of concentrated nitric acid heated at 300-350°C for 1 h in a muffle furnace. The AC samples were removed from the furnace and placed in a desiccator to cool. These samples were designated AcIr (activated carbon iroko) and AcGm (activated carbon gmelina), respectively.

Decolorizing property of AC

10 mL each of palm oil (*Elaeis guineensis*) was mixed with 1 g of each AC sample (AcWh, AcIr and AcGm) in three separate boiling tubes. All three boiling tubes were heated separately at 60°C for 5 min, stirred adequately and filtered into a standard 50 ml flask using a funnel and Whatman filter paper at room temperature. To 1 mL of each filtrate 10 mL of *n*-hexane was added and stirred. The absorbance of the mixture was determined at 360 to 460 nm at 20 nm intervals using a UV/visible spectrophotometer (Varian model-21). The values are given in **Table 1**.

Preparation of *n*-hexane standard

10 mL of palm oil was heated at 60°C for 5 min. To 1 mL of this substance, 10 mL of *n*-hexane was added, stirred and filtered. The absorbance of the filtrate was determined at 360 to 460 nm at 20 nm interval using the UV/visible spectrophotometer. The values are given in **Table 1** as control absorbance.

Preparation of ash samples

10 g of each of the samples (AcWh, AcIr and AcGm) were placed in separate crucibles and placed in a muffle furnace and heated to 650°C for 2 h. The crucibles were withdrawn from the furnace and placed in a desiccator to cool and weighed to constant weight. 1 g each of the samples was each treated with 4 ml *aqua regia* (3:1 hydrochloric acid: nitric acid); the mixture was digested in a steam bath for 10 min and filtered. The filtrate was used to determine the amount of inorganic minerals using Atomic Absorption Spectrophotometry. Details of the Atomic Absorption Spectrophotometer

Table 1 Absorption properties of activated carbon.

| Wave length (nm) | Control absorbance | AcWh | Percentage removed (%) | AcGm | Percentage removed (%) | AcIr | Percentage removed (%) |
|------------------|--------------------|-------|------------------------|-------|------------------------|-------|------------------------|
| 360 | 0.592 | 0.245 | 58 | 0.460 | 22 | 0.531 | 10 |
| 380 | 0.563 | 0.237 | 57 | 0.388 | 31 | 0.479 | 14 |
| 400 | 0.502 | 0.338 | 32 | 0.315 | 37 | 0.419 | 16 |
| 420 | 0.691 | 0.349 | 49 | 0.582 | 15 | 0.640 | 7.3 |
| 440 | 0.633 | 0.312 | 50 | 0.525 | 17 | 0.585 | 7.5 |
| 460 | 0.574 | 0.351 | 38 | 0.465 | 18 | 0.520 | 9.4 |

Table 2 Ash content of activated carbon.

| Elements | Wave length (nm) | Flame type | Burner height (nm) | AsWh (mg/L) | AsGm (mg/L) | AsIr (mg/L) |
|-------------|------------------|------------|--------------------|-------------|-------------|-------------|
| Na | 589.0 | A.A.O | 9 | 0.21 | 0.35 | 0.32 |
| K | 766.5 | A.A.O | 9 | 0.20 | 0.46 | 0.34 |
| Ca | 422.7 | N.A.R | 8 | 0.97 | 0.44 | 0.26 |
| Mg | 285.2 | A.A.O | 9 | 0.20 | 0.46 | 0.26 |
| Cu | 324.7 | A.A.O | 9 | 0.99 | 0.45 | 0.31 |
| Fe | 284.3 | A.A.O | 9 | 0.96 | 0.52 | 0.33 |
| Total ash = | | | | 3.53 mg/L | 2.68 mg/L | 1.82 mg/L |

Flame types: A.A.O. = Air Acetylene Oxidizing
N.A.R = Nitrous/Oxide Acetylene Reducing

analysis (AAS, Varian model 220-2) are given in **Table 2**. 1 mL of each of the filtered ash digestate of the samples was made up to 10 mL with deionised water in a standard 25-mL flask. 5 mL of each substance was withdrawn for the AAS analysis. The concentration (mg/L) of the minerals in the samples was determined by the following method: Standards of the elements (Na, K, Ca, Mg, Cu and Fe) were prepared using appropriate concentrations of their sulphates and deionised water. The absorbance of these concentrations was determined with the spectrophotometer (AAS). The following elements (Na, K, Mg, Cu, and Fe) were determined using an air acetylene oxidizing (AAO) flame while a reducing nitrous oxide-acetylene (RNO) flame was used to determine Ca. A plot of absorbance vs. concentration was obtained. The concentration of each element of each sample was obtained by interpolating the absorbance of each sample to the appropriate concentrations on the graph.

RESULTS AND DISCUSSION

The generalized *t*-test was used for testing the significance of the difference between the means of the sets observations for the absorption property and ash content of the AC. For the absorption property $t = 3.43$ when calculated between AcWh and AcGm while that between AcGm and AcIr was $t = 1.4$. Testing these values between sets of observations with $P = 0.05$ or $P = 0.01$ (d.f. = 10), the result is significant for the observation between AcWh and AcGm at both confidence levels but not significant at both confidence levels with respect to observations between AcGm and AcIr.

Also, for ash content analysis *t* calculated with a set of observations between ash water hyacinth and ash gmelina was $t = 0.82$, while that for the set of observations between ash gmelina and ash iroko was $t = 0.92$. These values are not significant when tested at $P = 0.05$ or $P = 0.01$ (d.f. = 10) between the sets of observations.

It is generally believed that the preparation and performance of AC is governed by a characteristic material, which forms the major non-carbohydrate constituent (methoxyl constituent) called lignin. Lignin is the starting material/source for the forming of humic and fumaric acids. Lignin is a system of thermoplastic tridimeral polymers (Li *et al.* 2006) derived from coniferyl alcohol or guaiacyl propane monomers. These lignin basic units are then subjected to oxidation followed by demethylation to substituted polyphenols and further oxidation to quinone derivatives. Condensation of the quinone groups with amino acids and polysaccharides may then yield humic acid. The bulk of lignins occur in the secondary cell wall where it is associated with cellulose and hemicellulose in stems. The quantity of lignins increases when the plant's organs are young, and particularly in monocotyledons, where there is little wood. Thus, plant age and stem content becomes a factor.

In **Table 1**, it is observed that the highest percentage of

decolorisation was with AC prepared with water hyacinth (58% at 360 nm). Decolorisation (bleaching) is a process aimed at removing coloured constituents from oils. Decolorisation usually involves adsorption of the colouring matter on bleaching earths and AC. Certain colouring matter such as carotenoids and various quinonoid colouring matters can be almost completely removed from the oil by adsorptive bleaching. The lowest percentage of decolorisation was with AC prepared from iroko (7.3% at 420 nm). Suhasa *et al.* (2006) showed that lignin is relatively non-reactive and probably the component of lignocellulosic precursors primarily responsible for the microporosity of ACs. Under appropriate conditions (monocotyledons and young plant organ) activation is possible with materials with surface areas and pore volumes approaching $2000 \text{ m}^2 \text{ g}^{-1}$ and $1 \text{ cm}^3 \text{ g}^{-1}$, respectively. These materials have the capacity for aqueous phase adsorption of metallic pollutants.

In **Table 2**, it can be observed that high ash content is found with the AC prepared from water hyacinth (3.53 mg/L) while the AC from iroko has the lowest ash content (1.82 mg/L). Furthermore, even though water hyacinth can give an AC with a good surface area and pore volume (good decolorizing properties) it also gives AC with a high degree of impurity and high extent of adulteration. Nevertheless, it should be noted that the preparation, characterization and adsorption applications of commercial ACs should take into cognizance the utilization of lignin as an adsorbent material and adequate levels of ash as indicative functional properties for starting materials of AC.

REFERENCES

- Broido A, Nelson E, Maxine A** (1964) Ash content: Its effect on combustion of corn plants. *Science* **146**, 652-653
- Davidson HW** (1970) *Manufactured Carbon* (3rd Edn), Chemical Publishing Inc., New York, 123 pp
- Edgecombe EA** (1999) Differential distribution of ash in stems of herbaceous plants from base to tip. *American Journal of Botany* **26**, 324-328
- George TA** (1984) *Shreve's Chemical Process Industries* (5th Edn), McGraw Hill Publishers, New York, 130 pp
- Hoffman PC, Tayson D** (2007) How much ash are you feeding your cows? *Hoards Dairyman* **149**, 659
- Henning K, Degel J** (1990) Activated carbon for solvent recovery. *Meeting of the European Rotogravure Association Engineers Group Mulhouse, France*, pp 20-21
- Li J-B, Gunnar H, Goran G** (2006) Lignin depolymerization/repolymerization and its critical role for delignification of aspen wood by steam explosion. *Bioresource Technology* **98**, 3061-3068
- Zhao L-J, Xiao H-L, Ren J, Lu M-F, Zhou M-X** (2007) Correlations of foliar Δ with K concentration and ash content in sand-fixing plants in the Tegger Desert of China: Patterns and implications. *Environmental Geology* **51**, 1049-1056
- Suhasa PJM, Carrott AR, Beuro C** (2006) Lignin – from natural adsorbent to activated carbon. *Bioresource Technology* **98**, 2301-2312