

The Production of Activated Carbon from Nigerian Mineral Coal *via* Steam Activation

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(received July 19, 2010; revised April 1, 2011; accepted April 11, 2011)

Abstract. Activated carbon was produced from Okpara sub-bituminous coal and Ogwashi brown lignite coal of Nigeria through steam activation at 900 °C and 960 °C each for 30 min and 60 min. Okpara and Ogwashi precursor coals had carbon content of 67.41 and 64.47%, respectively, whereas the bulk density and the ash content were 0.59-0.68 g/mL and 2.56-9.91%, respectively. The former exhibited up to 901.0 mg/g iodine number and Brunauer Emmett Teller (BET) surface area of 604 m²/g while the latter, iodine number of 998.0 mg/g and 669 m²/g BET surface area. Both showed adequate porosity indicative of their potential for utilization for commercial production of active carbons.

Keywords: Nigerian coal, activated carbon, steam activation, adsorption capacity

Introduction

The advent of oil exploration in Nigeria as well as import and operation of heavy trucks and trains with diesel engines led to total neglect of large deposits of mineral coals found in the most parts of Nigeria. This state of affairs gave impetus to investigate the possible conversion of two Nigerian coals, namely Okpara sub-bituminous coal and Ogwashi lignite coal to useful products like activated carbon.

Total recoverable coal reserves around the world have been estimated to be 930 billion tonnes, about 76% of which are located in USA (28%), Russia (19%), China (14%), Australia (9%), India (7%), while the remaining 23% is distributed among other 65 countries (EIA, 2008; USDA, 2008; IEA, 2007). The only African country is South Africa that produces 5.75 quadrillion Btu and consumes 3.81 quadrillion Btu, (Coal Association, 2007; EIA, 2005).

Some of these imported coals are utilized by Japan and China in production of activated carbon. The global consumption of activated carbon has been put as over 350,000 tonnes per annum and due to its high market demand and applications related to the environmental policies worldwide, a projected (7%) annual increase has been forecasted. Thus, by the year 2020, a total of 600,000 metric tonnes of activated carbon might be needed globally (EMS Energy Institute, 2001). Activated carbons are used in industry, medicine, agriculture, and

in almost all areas of human activities. No single active carbon has universal application. Thus, commercial active carbons could be classified into four groups on the basis of their physico-chemical properties, pore structure and applications (Choudhury *et al.*, 1985; Hassler, 1963). (i) Decolourizing carbon grade which are soft, finely powdered with high porosity and large surface area; (ii) gas/vapour adsorbent grade which are granular with high density, porosity and strength, used for industrial gas/vapour adsorption; (iii) metal adsorbent grade used as catalyst and catalyst support and (iv) medicinal grade adsorbent.

Active carbon is versatile and indispensable adsorbent especially in removal of colour, taste and odour from municipal water, industrial waste water and from food products. It is also used for recovery of gold in mineral industries and recovery of toxic organic solvents in chemical industries. Their use in pharmaceutical industries and medicine cannot be over emphasized such as its use in kidney dialysis machine (Lozano-Castello *et al.*, 2001; Zanzi *et al.*, 2001; Teker *et al.*, 1999). In gas applications, active carbon acts as gas filters, in general air conditioning and in storage of natural gases.

Coal and lignocellulosic materials are two main sources utilized for commercial production of active carbons. Low ash content is desirable in commercial active carbon and is prepared either by acid leaching or by a suitable selection of precursors (Bansal *et al.*, 1998).

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Some well known low ash precursors are lignocellulosic materials (Rodriguez-Reinoso and Linares-Solano, 1988); some brown coals (Woskoboenko *et al.*, 1991) and peat (Wigmans, 1983). Some of these precursors have lower yield of char that originates during carbonization, while coals from low rank lignite to high rank anthracite exhibit higher carbon yield on carbonization but have higher ash level (Spreigh, 1994; Van Krevelen, 1993). Lozano-Castello *et al.* (2001) used Spanish anthracite to prepare active carbon *via* chemical activation process with the aid of KOH as activating agent and obtained a high micropore volume of 1.45 cm³/g and BET surface area of 3290 m²/g. Buczek *et al.* (2000) produced two types of active carbons by steam activation of carbonized hard coal and tar, allowing burn offs of the carbonized coals to be 41% in one instance and 54% in another. Carrasco-Marin *et al.* (1996) used demineralised Spanish bituminous coal as raw material using CO₂ activation and combination of phosphoric acid and steam activation processes yielding microporous carbon that exhibited Type 1 adsorption isotherm and BET surface area of 1074 m²/g. Linares-Solano *et al.* (2000) compared the effect of mineral content in active carbon prepared from bituminous coal from Puertollano basin (Spain) on porosity development using steam activation and CO₂ activation. They observed that active carbon produced using CO₂ activation seemed to create new narrow micropores as well as widening the existing ones developed during carbonization process whereas steam appeared to widen only the narrow microporosity of the char, giving a more open microstructure.

In preliminary studies of inorganic constituents of Nigerian coals, Oderinde (1989) reported variability in the levels of concentration of inorganic metals present in Okaba, Okpara, Obi/Lafia coal mines. Earlier works on some Nigerian coals by Afonja (1975) and Oderinde (1989) showed that Nigerian coal produced large quantities of ash. Furthermore, Oderinde (1989) pointed out that the relatively large differences in elemental concentrations of the samples might be attributed to the areas, where the coals are mined or the size of material used. However, in a recent report, the Federal Ministry of Solid Mineral Development, Nigeria and the Raw Material Research Development Council of Nigeria (RMRDC, 2006) revealed Nigerian coal to be one of the most bituminous in the world owing to its low sulphur and ash content which makes it environmental friendly. Thus, the use of Nigerian coals as precursor

for production of active carbons is of interest especially due to the large coal reserves (RMRDC, 2006). About 3 billion tonnes of coal reserves have been identified in 17 coal fields of Nigeria Federal Ministry of Solid Minerals Development, Nigeria, 2006 and the Raw Material Research Development Council of Nigeria (RMRDC, 2006). The Nigerian coals have mainly been used locally as fuel and underutilized. Though, some physico-chemical properties of Nigerian coals have been reported, studies are not available exploring the possibility of production of active carbon *via* steam activation of coals. The aim of this study is to examine the physico-chemical properties of two Nigerian coals *viz* Okpara sub-bituminous coal and Ogwashi brown lignite coal and prepare their corresponding active carbons using steam activation process and compare their performance with commercial active carbons (BDH).

Materials and Methods

Collection of materials. Okpara coal and Ogwashi coal samples were obtained from Enugu, Enugu State in the eastern Nigeria and Ogwashi, Delta State in mid-western Nigeria, respectively, through Nigerian Coal Cooperation, Enugu, Nigeria. They are designated as sub-bituminous and lignite brown coal, respectively.

Preparation of active carbon. Four steps were used for preparation of active carbon : pretreatment of precursor raw coals, oxidation, carbonization and activation processes. Figure 1 shows the flow chart for manufacture of active carbon from some Nigerian precursor coals.

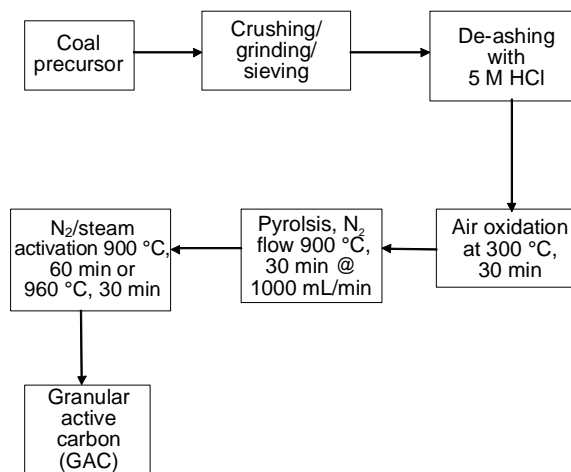


Fig. 1. Flow chart for manufacture of activated carbon from selected Nigerian coals.

Pretreatment of coal precursors. Each coal sample was ground and sieved (0.5-1.0 mm size), 50 g of each was treated with 150 mL of 5 M HCl and boiled in a 250 mL conical flask for 5 min. The solution was then filtered and the residue was washed several times with distilled-deionized water until the pH the filtrate was between 6.5 and 7.5. The samples were then dried at 60 °C in an air circulating oven for 2 h.

Oxidation, carbonization and activation processes.

The coal samples were then individually heated at ambient temperature to 300 °C and oxidized with air for 30 min. Carrasco-Marin *et al.* (1996) method of activation of demineralised precursor was used. A known quantity (9.0-10.5 g) of the acid-treated oxidized coal was loaded in hollow quartz glass tube (100 × 2.5 cm) and placed in activation horizontal tubular reactor furnace (Carbolite tube furnace, CTF 12/65/550 Model, Italy). The reactor was first evacuated with the aid of N₂ flowing at 750 mL/min for 30 min. The treated-oxidized coal samples were then pyrolysed at 900 °C for 30 min in the presence of N₂ at flow rate of 1000 mL/min. The activation process involved separate heating of the coal samples in the presence of nitrogen/steam mixture that was achieved *via* nitrogen supply with flow rate of 750 mL/min through a heated water bath kept at constant temperature of 92 ± 2 °C to specified final temperatures of 900 or 960 °C for either of 30 min or 60 min. Figure 2 shows the steam activation laboratory reactor coupled with horizontal tube furnace. A heating belt was used to cover the quartz glass in order to avoid condensation of vapours and thus prevent breakage of the glass. The heating rate

was maintained at 20 °C/min. The active carbon samples were allowed to cool down in the quartz tube in the presence of nitrogen/steam mixture. The yields of active carbons were determined gravimetrically, while those of volatile (burn off) were calculated by difference using the following equations:

$$(i) \text{ Active carbon yield (\%)} = \frac{\text{mass of active carbon}}{\text{mass of char}} \times 100$$

$$(ii) \text{ Volatile yield (\%)} = \frac{\text{Loss in mass of active carbon}}{\text{Initial mass of char}} \times 100$$

Physicochemical properties of precursor coals. The bulk density and moisture content of precursor coals were also determined following Ahmedna *et al.* (1997) and AOAC (1990), respectively, while the method of AWWA (1991) was used for determination of ash content. The ASTM (1996) was used for determination of pH and conductivity of the precursor coal samples.

Elemental analysis of coal samples. The precursor coal samples were subjected to elemental analysis to determine their carbon-hydrogen-nitrogen and sulphur (CHNS) composition. The analysis was carried out in duplicate by the Institute fur Organische Chemie (Universitat Tübingen, Germany) using a Carlo Erba elemental analyzer. The higher heating values (HHV) for each of the biomass types were calculated from the values of CHNS and ash content of the precursor materials (Graboski and Bain, 1981). The equation is given as:

$$\text{HHV (kJ/kg)} = 2.3236 [(141C + 615 H - 10.2 N + 39.95 S) - (1 - \text{Ash})(17244H/C) + 149]$$

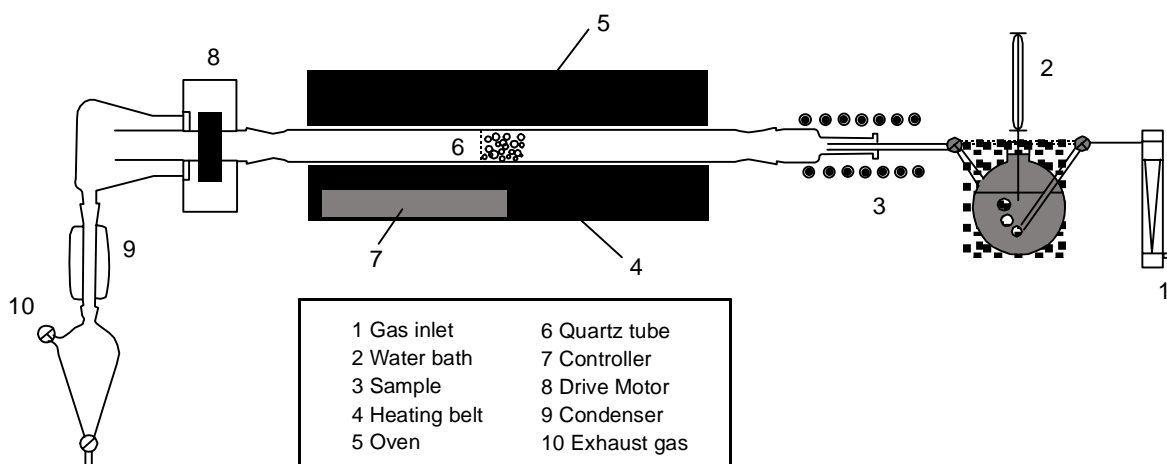


Fig. 2. Schematic diagram of the horizontal tube furnace for steam activation process.

Analysis of active carbon. The Brunauer Emmett Teller (BET) surface areas of steam activated carbons derived from the coal precursors were measured by nitrogen adsorption isotherm at 77 K using a Coulter SA 3100 surface analyser, U.K. The *n*-hexane adsorption method (Bayer *et al.*, 1995) was adopted for determination of micropore and mesopore volumes of the prepared coal-based active carbons types.

Adsorption of iodine and methylene blue. The active carbon produced from the two coal types were characterized by iodine and methylene blue adsorption. Methylene blue number and iodine number are measures of adsorption capacity of any type of active carbon. Iodine number indicates the extent of micropore distribution in the carbon (Kirubakaran *et al.*, 1991). It is a measure of iodine molecules adsorbed in the pores and indicates pore volume capacity. The iodine number, which gives an idea of the microporosity, was determined by the modified AWWA procedure (AWWA, 1974). The modification involves centrifugation of active carbon-iodine mixture for 5 min prior to titration with sodium thiosulphate solution in the presence of starch indicator. The methylene blue (MB) adsorption capacity of the active carbon, that depicts the extent of mesoporosity of the adsorbent, was measured in accordance with ASTM standards for active carbons (ASTM, 1989).

SEM analysis of active carbon. The physical surface morphology of the steam-based active carbon derived from Nigerian coal was conducted using scanning electron microscopy instrument (model DSM 982, Germany). A thin layer was mounted by a double sided tape. It was coated with Au/Pd to a thickness of about 30 nm. The scanning was carried out *in situ* at $\times 500$ magnification.

Results and Discussion

The physico-chemical properties of selected Nigerian coals: Okpara sub-bituminous coal (OKP) and Ogwashi brown lignite coal (OGW), are presented in Table 1. The OKP coal OGW brown lignite coal exhibited, in order, bulk density, moisture content, ash content, pH values and conductivity values of 0.68 g/cm³, 4.86%, 9.91%, 4.06 and 0.16 ms/sec, respectively, and 0.59 g/cm³, 8.10%, 2.56%, 3.62, 0.39 ms/sec, respectively. The variability in the values of various parameters of the two coals could be attributed to different location of the sites, where they were found. This report agreed with the report of Energy Information Administration

(EIA, 2010; 2006) about significant variability in heat content, ash and sulphur content by region and even at times within the same seam. The sulphur content range (0.59-0.66%) of OKP and OGW coals are lower than 0.9% of the United States coking coals. Table 1 also shows carbon content of precursor OKP and OGW coals as 67.41% and 64.47%, respectively, which are high enough for their utilization as raw material for production of active carbon. It had been noted that carbonization and controlled activation increased carbon content of the precursor materials (Adebowale and Adebowale, 2008); and consequently increase BET surface area of the active carbon. Low ash content in the precursor material is desirable in production of active carbon as high ash content does not contribute to surface area of the active carbon. The ash content of OKP and OGW Nigerian coals were 9.91 and 2.56%, respectively, which are low and desirable for production of active carbon.

Table 1. Physico-chemical properties of precursor Nigerian coals

	Okpara sub-bituminous coal (OKP)	Ogwashi lignite coal (OGW)
<i>Proximate analysis</i>		
Bulk density (g/cm ³)	0.67±0.01	0.59±0.01
Moisture (wt %)	4.86±0.27	7.43±1.20
Ash (wt %)	9.91±0.83	2.56±0.09
pH	4.06±0.01	3.62±0.01
Conductivity (ms/sec)	0.16±0.01	0.39±0.00
<i>Ultimate analysis</i>		
C	67.41±0.31	64.47±0.65
H	5.13±0.01	6.57±0.08
N	1.70±0.10	0.66±0.01
S	0.66±0.01	0.59±0.03
*O	14.88±0.23	25.15±0.76

*O = oxygen determined by difference.

The higher heating value (HHV) or heat content of precursor coals reported for various countries are comparable with the values of the two investigated Nigerian coals (Table 2), such as for United States coking coal (26.30 million Btu/ton), Greece lignite coal (4.4 million Btu/ton) and Canada lignite coal (12.4 million Btu/ton), which are higher than 0.31 and 0.19 million Btu/ton values for OKP and OGW precursor Nigerian coals, respectively. The heat content value is used for judging the capability of particular precursor coal for utilization for electricity production or

preparation of industrial product like active carbon. It is interesting to know that United States utilized about 90% of its coal to generate about 55% of the electricity and also produced active carbon from the unburnt carbon obtained as by-product of coal combustion (EMS-Energy Institute, 2001). From Table 3, it is evident that the yield and burn off values (volatile matter) resulting from separate activation of OKP coal at 900 °C for 60 min and 30 min are similar i.e. 47.39 % and 48.57%, respectively. However, when temperature of activation of OGW coal was raised from 900 °C to 960 °C, the yield was reduced from 27.84% to 22.15% 60 min. These findings are in line with the reports of Mameri *et al.* (2000) and Mc dougall (1991) that temperature affects yield of chars or active carbon more than variation in duration of activation process.

Total pore volumes of OKP coal active at 900 °C for 60 min and 30 min were 0.256 cm³/g and 0.089 cm³/g, respectively, while those of OGW active carbon prepared

Table 2. Higher heating value (Heat content) of some coal from selected countries

Countries	Type of coal	Amount of higher heating value (HHV) units	
		(MJ/kg)	(Btu/ton) × 106
*United States	Coking coal	61170	26.30
*Greece	Lignite	10230	4.40
*Canada	Lignite	28840	12.40
Nigeria (OKP)	Sub-bituminous	711.74	0.31
Nigeria (OGW)	Lignite	430.57	0.19

* = values obtained from Energy Information Administration, DOE/EIA-0121(2006/4Q); Washington DC (2007).

Table 3. Activation time, temperature, and burn off of active carbons

Coal types	Activation temperature (°C)	Activation time (min)	Yield (wt %)*	Burn off (wt %)*
Okpara subituminous	900	60	47.39	52.61
	900	30	48.57	51.43
Ogwashi lignite	900	60	27.84	72.16
	960	60	22.15	77.85
Okaba lignite	900	60	33.10	66.90
	900	30	40.68	59.32

*standard error is within the 5% error limit.

separately at 960 °C and 900 °C were 0.170 cm³/g and 0.120 cm³/g, respectively, for 60 min (Table 4). Thus the value of OKP active carbon is greater than that of 0.199 cm³/g for commercial powder carbon (BDH, England) and 0.206 cm³/g of commercial granular active carbon (BDH, England). The values, determined by *n*-hexane adsorption are ranked in increasing order of extent of porosity as follows:

OKP 900 °C, 30 min < OGW 960 °C, 60 min < active carbon (powder BDH) < active carbon (granular BDH) < OKP 900 °C, 60 min.

The micropore volume, V_{mi} ranges were found to be 0.048 - 0.066, 0.060 - 0.067 and 0.006 - 0.079 cm³/g while mesopore volume, V_{me} fell within the range of 0.041 - 0.190, 0.060 - 0.103, 0.127 - 0.193 cm³/g for OKP, OGW and commercial active carbons, respectively (Fig. 3). These values are lower than those reported elsewhere by N₂ adsorption method. It has, however, been recommended by IUPAC that N₂ should be used as standard adsorbate for pore volume measurement (Sing *et al.*, 1985). The micropore filling nature, unusual shape and molecular size of N₂ gas makes its use as adsorbate in pore volume measurements to give higher values than values obtained through other molecular probes like *n*-hexane and benzene that exhibit higher reliability (Sing, 1989). In consideration of the foregoing explanation, the micropore volume range (0.048-0.066 cm³/g) and mesopore range (0.041-0.190 cm³/g) for OKP active carbon is lower than micropore volume range of 0.192-0.40 cm³/g obtained for active carbon with 40-60% burn off (Linares-Solano *et al.*, 2000) and 0.281 and 0.315 cm³/g miropore volumes for 41% and

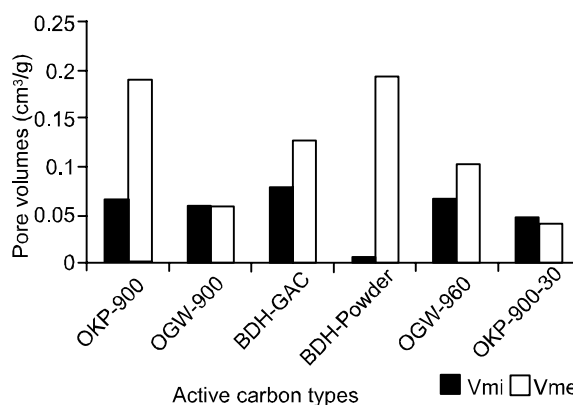


Fig. 3. Comparison of micropore/mesopore volumes of various active carbons with commercial active carbons.

Table 4. Textural characteristics of prepared active carbons from selected Nigerian coal

Coal types	Activation temperature (°C)	Activation time (min)	Iodine number (mg/g)	Methylene blue number (mg/g)	BET surface area (m ² /g)	*V _{total} (cm ³ /g)
OKP-900-60	900	60	901	130.6	604	0.256
OKP-900-30	900	30	801	27.4	537	0.089
OGW- 900-60	900	60	998	< 0.1	669	0.120
OGW-960-60	960	60	920	70.7	616	0.170
Commercial coal powder (BDH)	–	–	785	54.3	526	0.199
Commercial coal granular (BDH)	–	–	873	70.1	751	0.206

*V_{total} = represents total pore volume.

54% burn off, respectively (Buczek *et al.*, 2000) from granules prepared from hard coal and tar. The same applies to the micropore and mesopore volumes of OGW active carbons.

Total pore volume 0.256 cm³/g for OKP-90-60 active carbon was higher than those of the commercial powder active carbons (0.199 cm³/g) and granular active carbon, GAC (0.206 cm³/g) (Table 4). The values of pore volumes and BET surface area for OKP-900 active carbon prepared *via* steam activation for 60 min was somewhat better than those prepared with contact time of only 30 min (OKP-900-30). Steam activation of oxidized pre-treated OGW precursor coal at 960 °C for 60 min resulted in lower yield of 22.15% and higher burn off percentage of 77.85% as compared to steam activation at 900 °C for the same contact time, which yielded 27.84% active carbon and 72.16% burn off. Porosity of the OGW active carbon derived from steam activation at higher temperature (960 °C) exhibited higher values (0.170 cm³/g) than that activated at 900 °C with value of 0.120 cm³/g. This result is in agreement with the reports of Buczek *et al.* (2000). The iodine number of 920 mg/g and 998 mg/g were obtained for OGW active carbons prepared for 60 min at temperature of 960 °C and 900 °C, respectively, while those for OKP carbons activated at 900 °C for 60 and 30 min are 901 and 801 mg/g, respectively. The BET surface area were found to be within 537-669 m²/g for all the prepared active carbons and are comparable with the commercial BDH powder and granular active carbons (Table 4). These values are within the range of

values obtained by Bacaoui *et al.* (2001) for adsorption of methylene blue (115-490 mg/g), adsorption of iodine (741-1495 mg/g) and BET surface area (514-1271 m²/g) for series of active carbons prepared by physical activation with steam. Interestingly, the minimum molecular size of MB is 0.8 nm which could pass through minimum pore diameter of only about 1.3 nm and therefore, enters the largest micropore diameter. However, the most mesoporous carbons adsorb methylene blue, molecules while iodine molecules are greatly adsorbed into micropores due to its small size.

The FT- IR spectra (spectra not shown) show that there exists similarity in the functional groups present on the surface of OGW (900 °C, 60 min.), OKP (900 °C, 60 min.) and commercial BDH granular active carbons. The OH functional group, C=O found in COOH acids and C-O functional groups present in simple ethers, alcohols and acid anhydrides show stretch vibrations within the range of 3449-3456 cm⁻¹, 1635- 1637 cm⁻¹ and 1050-1360 cm⁻¹, respectively (Petrov *et al.*, 2000). These stretch vibrations are common to both the prepared active carbons and the commercial active carbon (BDH). The existence of 449-673 cm⁻¹ band range in the active carbons depicts carbon-heteroatom bond (Duran-Valle *et al.*, 2006). The presence of C=C functional group in the OKP-900-60 active carbon as well as the commercial granular active carbon (BDH) is additional similarity in functionality. However, it was observed that C=C functional group was conspicuously absent from the spectra of the OGW-960-60 active carbon. It might be that the carbon atoms burnt off and broke the C=C

double bond functional groups, widening the existing pores at the carbonization stage; the high burn off value may thus be a confirmation of destruction of C=C bond.

The SEM micrographs of Fig. 4a-4c revealed that there are pores and crevices all over the surfaces of the active carbons prepared from OKP and OGW Nigerian coals as well as that of the commercial powder active carbon. It was observed that OGW carbon active at 960 °C for 60 min exhibited the highest burn off percentage

(77.85%) and also exhibited high methylene blue number (70.7 mg/g) and iodine number (920 mg/g) and thus could be utilized for de-colourization purposes as well as for treatment of municipal water while the OKP active carbon prepared *via* steam activation at 900 °C for 60 min with insignificant methylene blue number (< 0.1 mg/g) and high iodine number (901 mg/g) may be used for adsorption of gas molecules or small molecules of volatile organic compounds.

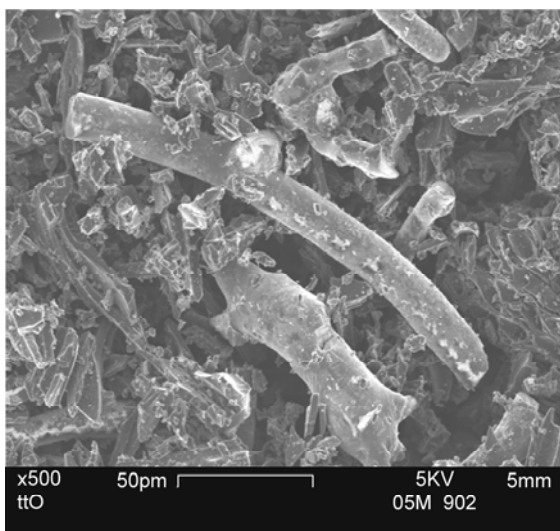


Fig. 4a. Micrograph of OGW active carbon prepared by activation at 900 °C for 60 mins.

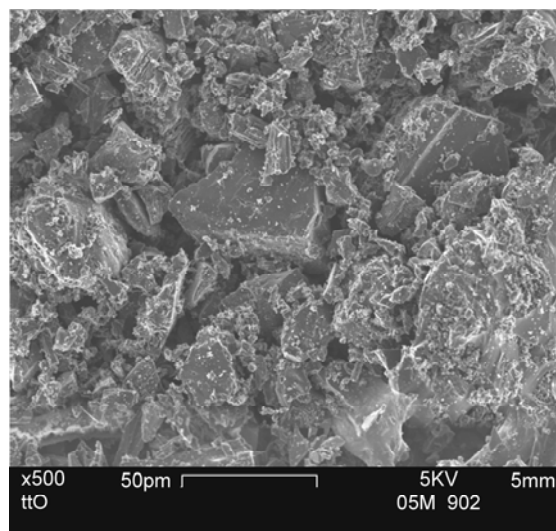


Fig. 4c. Micrograph of commercial powder active carbon.

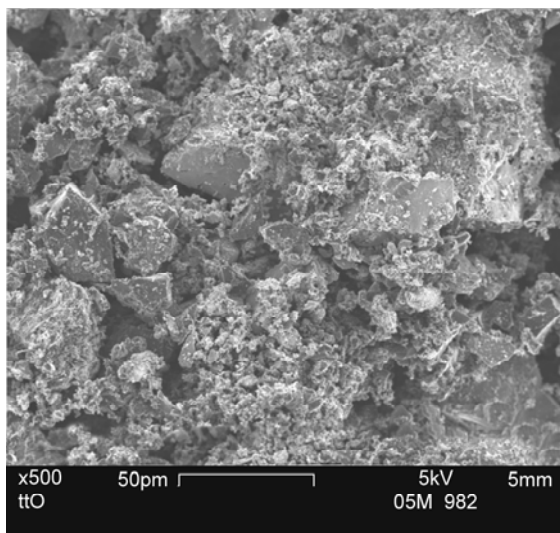


Fig. 4b. Micrograph of OKP active carbon prepared by activation at 900 °C for 60 mins.

Conclusion

The active carbon prepared *via* steam activation from Nigerian brown lignite and sub-bituminous coals compared favourably with commercial BDH active carbons in terms of porosity and surface area properties of active carbons. The effect of temperature on development of porous properties using steam activation process during production of active carbon is greater than the effects of duration of contact period. More so, the investigated Nigerian coals may contain less heat content compared to United States bituminous or Canada lignite coals and may only allow its full utilization in production of industrial product such as active carbon.

Acknowledgements

The authors thank Nigerian Coal Cooperation of Enugu that supplied the coal samples. We are also grateful to Prof. Thomas Henle and Dr. Uwe Schwarzenbolz for

running the BET surface area and Scanning Electron Microscopy (SEM) analysis.

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