Coal Utilization: Physicochemical Analysis of Selected Coals and Environmental Impact

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Abstract

The properties of coals influence their applications. This work studied the elemental composition, pore structure, structural morphology and thermal behaviour of coals obtained from several Nigerian coal mines. Proximate analysis was carried out following standard methods, whereas ultimate analysis was performed with a CHNS analyser. The pore structures were analysed with a Brunner-Emmett-Teller (BET) analyser. The selected elements and inorganic oxides were determined via an Energy Dispersive X-Ray Fluorescence (EDXRF) analyser, the structural morphology was assessed via a scanning electron microscope, and the effect of heat treatment was investigated via a thermo-gravimetric analyser. EDXRF analysis indicated that approximately 35% of the trace elements of environmental concern were not detected in the Nigerian coals. SEM images revealed that the coals had pores. The properties of the coals varied indicating the importance of characterization for understanding the appropriate application and the environmental impact on the utilization of the coals for various applications. The tested Nigerian coals are preferable to some international coals for thermal applications because of their lower environmental pollution potential. Nigeria could harness the rich coal resources of its sedimentary basins for thermal and other applications.

Introduction

Energy demand and supply are major indicators of the level of development and standard of living in a country. Coal is an underground solid combustible sedimentary rock that is the global largest energy source for electricity generation, and the production of iron, steel and cement. However, it is the largest single source of carbon dioxide (CO_2) emissions [1]. Coal naturally occurs in many countries including Australia, China, Nigeria, South Africa, and United States of America. Coal deposits in Nigeria are found at Benue trough and Anambra sedimentary basins. The proven coal reserve in Nigeria is over 639 million metric tons, whereas the inferred reserve is 2.75 billion metric tons [2].

The properties of coal differ because of to the nature of the dead plants that form it, the climatic conditions during coal formation and the topography of the environment [3]. The mineral matter in coal may originate from minerals in the plant and water where dead plant materials are buried [4]. The elemental composition may change vertically and laterally within a single coalbed. Therefore, coals from different locations have different properties [5, 6].

Studies have been carried out on the quality of some Nigerian coals. Benedict *et al* characterized coals obtained from Ridi-Awe and Lafia (Nasarawa State), and Garin Maiganga (Gombe State) Nigeria via X-ray fluorescence spectroscopy. The XRF results indicate the presence of some transition metals and other trace elements [7]. Ryemshak *et al* carried out an ultimate analysis of coals obtained from Garin Maiganga (top layer and base layer), Chikila, Lamza and Afuzie. The coal samples were reported as to have low elemental contents [8]. Nigerian coals from Odagbo (Kogi State), Owukpa (Benue State), Ezimo (Enugu State), Amansiodo (Enugu State), and Inyi (Enugu State) were subjected to proximate, ultimate, and thermogravimetric analysis in a study by Chukwu *et al* for the combustion of these coals [9].

In the literature, little or no information has been obtained on the vital chemical and physical properties of Nigerian coals which could impact their utilization. Therefore, the aim of this study is to assess the chemical composition, pore structure, structural morphology and thermal behaviour of coals obtained from mines at Ribadu, Ogboyega, Ogwashi-uku, Okaba, Inyi, Owukpa and Amansiodo.

The objectives of this research are (1) to carry out proximate and ultimate analysis of coals (2) to determine some elements and inorganic oxides (3) to assess the structural morphology of the coals, and (4) to carry out thermo-gravimetric analysis of the coals.

This study was carried out at the National Centre for Energy Research and Development, University of Nigeria, Nsukka, Nigeria.

Experimental

Coal samples were obtained from the Ribadu, Ogboyega, Ogwashi-uku, Okaba, Inyi, Owukpa and Amansiodo coal mines in Nigeria.

These coals were analysed on an as-received basis. The equipment used were Perkin Elmer Series II CHNS/O Analyzer 2400, Perkin Elmer TGA 4000, PRO:X: 800-07334 Phenom World, Scanning electron microscope, Thermo Fisher Scientific ARL. Quant'x. Energy dispersive X-ray fluorescence analyzer, Hewlett adiabatic bomb calorimeter model 1242, MB 35 Halogen moisture analyser, Vecstar furnace model LF3, Adventurer Ohaus top loading weighing balance and Rupson standard test sieve.

Physical characteristics of the coal samples

A. Colour: The colours of the coal samples were observed.

B. Bulk density

The bulk density of the coal samples was measured via the displacement method [10]. This was based on the formula: density = mass/volume. A solid coal sample of known weight was immersed in known volume of water in a measuring cylinder. The new volume is used in the formula.

C. Surface area and pore structure determination

The Brunner-Emmett-Teller method was used for the determinations. This test was carried out with a Quantachrome Nova 4200e analyser, via several methods including multi point BET, Barrett-Joyner-Halenda (BJH), and Density functional theory (DFT) methods.

D. Structure and surface morphology of the coal samples

This analysis was performed via a scanning electron microscope (SEM) model: PRO:X: 800-07334 Phenom World. This method provides information on the structure and surface morphology of materials. The images were taken at different magnifications.

E. Thermogravimetric analysis of the coal samples

This analysis was done using a Perkin Elmer TGA 4000. Approximately 11.257 mg of each of the samples was placed in the sample holder and put in the analysis chamber of the equipment and then covered. The mixture was allowed to cool to 15 °C before the analyzer was connected to a desktop computer which was used to operate the analyzer. The mixture was subjected to nitrogen flow at a heating rate of 20°C/min corresponding to slow pyrolysis process. A derivative thermogravimetric analysis (DTG) profile was obtained to interpret the mass losses from the samples.

Chemical composition of some Nigerian coals

A. Proximate analysis

This is an analysis of the moisture, ash, volatile matter and fixed carbon compositions of a material. The samples were crushed and ground. The ground samples were sieved with a Rupson sieve (model 250 μm). Proximate analysis of the samples was performed following the American Society for Testing and Materials (ASTM), D3174-82, standard method [11]. The ash content determination was carried out with 3 g of the as-received coal samples, which were weighed in triplicate into porcelain crucibles. The covered crucibles with contents were placed in a Vecstar, model LF3 at 600 °C until ashed for 35 hours before being cooled and reweighed. The volatile matter determination was performed with the sieved samples placed in the furnace for 7 min at 950 °C. The weight loss was the volatile matter content. Moisture determination was carried out with an MB 35 Halogen moisture analyser. The instrument was operated as directed in its operating manual and set at 110 °C. The fixed carbon content was calculated by adding the ash, volatile matter and moisture contents and subtracting the total from 100.

B. **The Calorific values** of the samples were determined via a Hewlett 1242 adiabatic bomb calorimeter.

C. Ultimate analysis using elemental analyzer

Ultimate analysis involves the determination of carbon, hydrogen, sulphur and nitrogen in samples. This determination was carried out with a Perkin Elmer Series II CHNS/O Analyzer. The thermal conductivity was measured with a detector and the results are presented as a percentage of the sample weight.

D. Determination of elements and inorganic oxides via EDXRF analyzer

This determination was carried out with a Thermo Fisher Scientific ARL Quant'x. Energy dispersive X-ray fluorescence (EDXRF) analyzer. The samples were ground, sieved and 2 g each of the sieved samples were weighed into the sample holder of the analyzer and covered with cotton wool. The sample holders containing the samples were run in a vacuum using a vacuum pump for 10 min to remove oxygen and moisture and then placed into the XRF Spectrometer for analysis. The system was calibrated with certified samples. Calibrations were performed for the elements and for the oxides. The samples were allowed to run in the EDXRF spectrometer for 10 min each.

Results and discussion

The colour of a coal is a depiction of its elemental content. The colours of the tested coals are presented in Table 1.

Table 1: Colours of the studied coals

S/No	Location of coal field	Colour
1	Amansiodo (AM)	Grey
2	Inyi (IY)	Grey
3	Okaba (OK)	Grey
4	Ogwashi-uku (OU)	Dark
5	Owukpa (OW)	Grey
6	Ogboyega (OY)	Dark
7	Ribadu (RB)	Dark

The mean values of the bulk density of the tested coals are presented in Table 2.

Table 2: Density of selected coal samples via the displacement method

Parameters	AM	IY	OK	OU	OW	OY	RB
Density (g/cm^3)	1.16	1.04	1.19	0.50	0.74	1.25	0.63

Density is an indicator of the level of packing of particles in a material. Tightly packed materials may not burn as fast as loosely packed materials. This implies that emission is lower for denser material than for less dense material of the same structure at a given time. The volatile matter content is expected to decrease with increasing coal density. The resultsof the present study as shown in Table 2 indicate that Ogboyega coal is denser than all the tested coals are, whereas Ogwashi-uku coal has the lowest bulk density. As reported by Mosharef et al [12], the density range of coals in Bangladesh and India is 1.28-2.20, and 1.18-1.80 g/cm^3 respectively, whereas that of Australian coal is 1.16 g/cm^3 and the density of South African coal is between 1.25 and 2.10 g/cm^3 [13].

A summary of the results of the analysis of the surface area, pore volume and pore size of the selected coal samples is presented in Table 3.

Table 3: BET surface area analysis, pore volume and pore size measurements with a Quantachrome analyser

Parameters	RB	OY	OU	OK	IY	OW	AM
Single point BET Surface area (m^2/g)	2.469e+02	2.017e+02	2.653e+02	1.337e+02	1.377e+02	1.496e+02	1.216e+02
Multi point BET Surface area (m^2/g)	3.985e+02	2.828e+02	3.535e+02	2.189e+02	2.264e+02	2.461e+02	1.740e+02
DFT method cumulative pore volume (cc/g)	1.151e-01	9.593 e-02	1.444e-01	6.343 e-02	6.518e-02	7.085e-02	5.749e-02
DFT method Diameter (Mode) (nm)	2.647e+00	2.647e+00	2.647e+00	2.647e+00	2.647e+00	2.647e+00	2.647e+00

The surface area affects the burning rate of solid fuels. A wider surface area will provide more exposure for contact with oxygen which facilitates combustion and thereby results in faster mass loss of the pellet [14]. With faster combustion, more emissions are expected within a shorter time, which translates to increased pollution during combustion. The present study reveals that the surface area of Ribadu coal is greater than that of coals from the other mines, whereas the Amansiodo coal has the smallest surface area, and it is expected to burn more slowly than the other tested coals.

Pores in coal can cause differences in the amount of oxygen diffused into the micropores of coal. The volume of micropores in coal may be used to assess the likelihood of spontaneous combustion occurring. A large pore volume increases the chance of coal contacting O_2 molecules which favours the absorption of oxygen and therefore facilitates combustion leading to the emission of pollutants [15, 16]. The present study reveals that the Ogwashi-uku coal has the largest pore volume while Amansiodo coal has the smallest pore volume.

The BJH method, which is a common method of pore size analysis, shows that all the tested coals are porous. Ogwashi-uku has a micropore size as it has a pore size of < 2 nm whereas the other tested coals are within the mesoporous range as their pore sizes are between 2 and 50 nm. The porosity of coal indicates its ability to absorb oxygen which can enable combustion, and therefore pollution through emissions.

Coal is an important resource in the manufacture of cement and in metallurgy. Coking coal is mostly utilized for these purposes. Coking coals are coals that when heated, soften, fuse and re-solidify to form a porous carbon-rich material called coke. Coking properties are dependent on the porous structure of coal [17].

The pore distribution structures in the coals at different magnifications were diverse. The shapes were observed. The elemental compositions at different spots were also captured via SEM-EDX. The SEM-EDX results for the Ribadu and Ogboyega coals are presented in Figures 1 and 2.

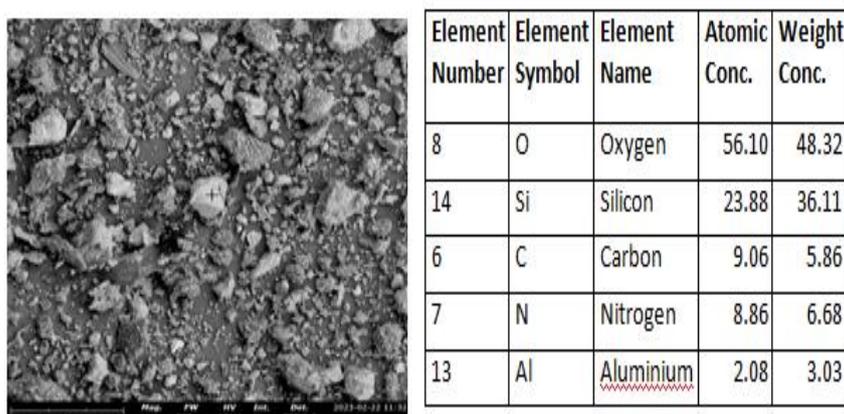| Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
8	O	Oxygen	56.10	48.32
14	Si	Silicon	23.88	36.11
6	C	Carbon	9.06	5.86
7	N	Nitrogen	8.86	6.68
13	Al	Aluminium	2.08	3.03

Figure 1: Ribadu coal 1000x SEM-EDX image

Figure 1 shows the SEM- EDX results of the Ribadu coal sample at 1000x magnification on one spot of the sample using Phenom-World-PC EDX. The elements observed at the spot are O, Si, N and C, with O having the highest concentration. The cross-section shows small, stone-like lumps, with irregular pore shapes. The pores are connected which enables the flow of fluids. Therefore, gases such as O_2 molecules can flow through to facilitate combustion.

Figure 3 shows the Ogboyega coal 1000x SEM- EDX results at one spot of the sample using Phenom-World-PC EDX. The coal sample is a long, pipe-like lump mixed with a large stonelike lump with pores and irregular holes. The flow of fluids inside the coal will be resisted; hence, the emission of pollutants will most likely be limited during utilization. The elements observed at the recorded spot are O, C, Al and Si. O was also the highest in quantity.

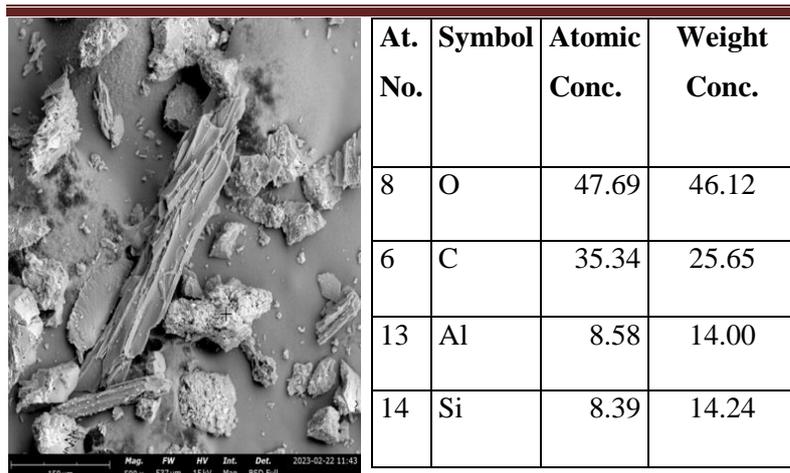| At. No.	Symbol	Atomic Conc.	Weight Conc.
8	O	47.69	46.12
6	C	35.34	25.65
13	Al	8.58	14.00
14	Si	8.39	14.24

Figure 2: 1000x SEM-EDX image of the Ogboyega coal sample at 1 spot of the sample.

Some curves from the thermo-gravimetric analysis of the coal samples are presented in Figures 3 and 4. The figures reveal the pattern of weight loss at various temperatures during the thermal treatment of the Ribadu and Ogboyega coals. The DTG profiles on the basis of the rate of weight loss at different temperatures of the coal samples are also presented.
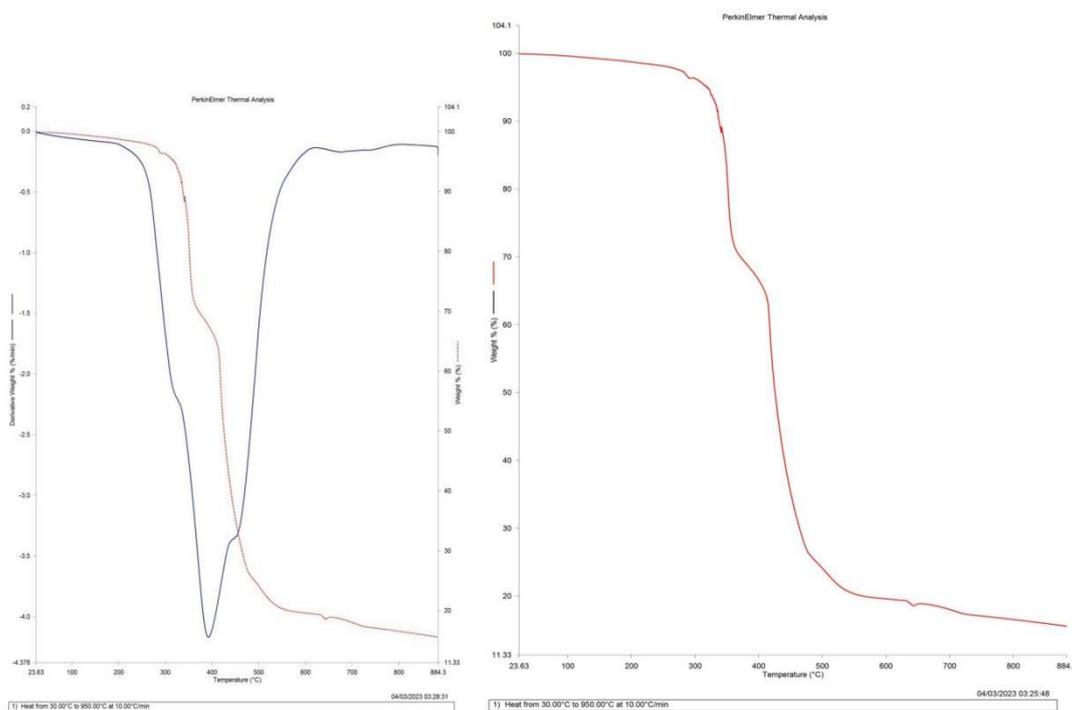

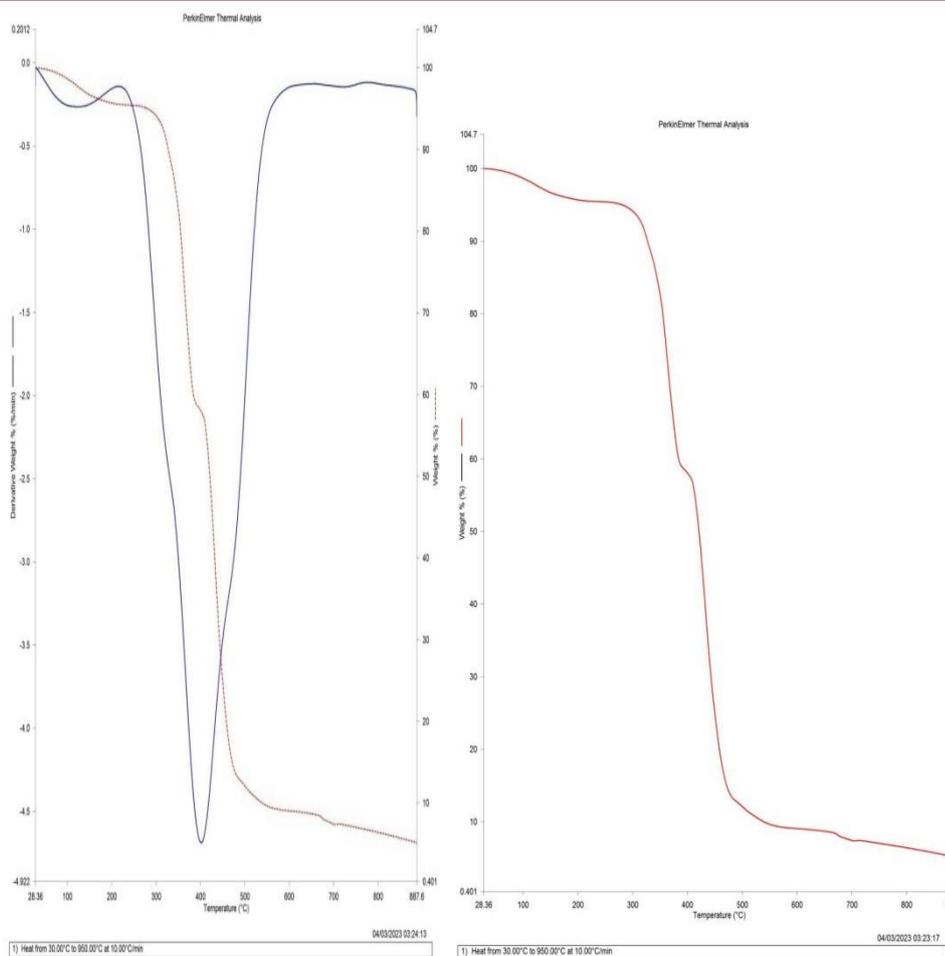Fig. 3: DTG and TG results for Ribadu coal

Fig. 4. DTG and TG results for Ogboyega coal

Table 4 shows a summary of the results of the thermal analysis of the coal samples.

Table 4: Thermal analysis of coal samples

Sample	Stages (°C)					
	1st	Wt. loss (%)	2nd	Wt. loss (%)	3rd	Wt. loss (%)
RB	26 - 350	3	350 - 480	75	480 - 740	10
OY	26 - 310	4.5	310 - 460	87	460 - 760	5
OU	26 - 300	5	300 - 440	88	440 - 780	10
OK	26 - 320	3	320 - 460	80	460 - 800	2
IY	26 - 310	6	310 - 450	76	450 - 720	6
OW	26 - 330	2	330 - 530	80	530 - 780	2
AM	26 - 320	5	320 - 440	70	440 - 700	5

As shown in Figures 3 and 4, the behaviour of the studied coals during heat treatment shows three decomposition stages. Taking RB as an example, the first part of the decomposition of the studied RB (Ribadu) coal occurs mainly below 350 °C, which corresponds to the removal of moisture in the coal (Table 4) [18]. A major decomposition path is observed between 350 and 480 °C. This significant loss may be attributed to the release of volatile matter. According to Himbane et al. [19], devolatisation occurs within this temperature range. The decomposition continues slowly above 480 °C until approximately 740 °C at which point the weight loss rates gradually flatten, indicating that the weight loss is near zero and can be neglected. A similar explanation applies to the other samples as the values are shown in Table 4. This indicates that the volatile matter in the coal will be emitted completely at 480 °C for RB, 440 °C for OU and 530 °C for OW. From the results of the TG analysis, the temperature at which volatile matter is removed from the coal samples is known. This information will be useful in the application of coal. For example, the temperature at which the coal samples are carbonized to produce smokeless briquettes is the temperature at which the volatile matter is completely released. Hence, making smokeless briquette with RB coal requires carbonization at 480 °C.

The results of the proximate analysis of the coal samples are presented in Table 5.

Table 5: Proximate analysis of the coal samples

Parameters	AM	IY	OK	OU	OW	OY	RB
Volatile matter (%)	67.99	81.30	74.57	73.76	71.24.63	77.54	67.83
Ash content (%)	10.61	6.85	4.86	10.58	9.40	13.67	16.80
Moisture content (%)	2.79	2.90	5.48	5.92	4.59	4.10	0.70
Fixed carbon (%)	18.61	8.95	15.09	9.74	14.77	8.69	15.17

Coal has good combustion quality if it has a low moisture content. The moisture in solid fuels reduces the calorific value and the flame temperature. The present study reveals that RB coal has the lowest moisture content whereas OU coal has the highest moisture content. The moisture contents of the tested coals, in descending order are Ogwashi-uku>Okaba>Owukpa>Inyi>Amansiodo>Ribadu. The moisture content of coal from the United Kingdom (UK) (Daw mill) is 4.60% [20].

The ash content is a measure of the total minerals present in a material. Metals in materials are found in ash with the exception of volatized materials. Ash does not burn and therefore constitutes an impurity in a material. It lowers the heating value of a material. The ash content in Ribadu coal is 16.80% which is higher than the 13.67% obtained for Ogboyega coal and the 4.20% obtained for UK coal [20]. The ash content is an indicator of the propensity of a material to contribute to environmental pollution.

Materials with high volatile matter ignite easily but burn with less heat intensity. Compared with materials with low volatile matter contents. Materials with high volatile matter contents emit more pollutants (volatiles or smoke) into the environment compared to materials with low volatile matter content. The present study revealed that Inyi has the highest volatile matter content. The studied UK coal has 31.30% volatile matter [20].

Fixed carbon, which is the energy stored in a material, is directly related to heat content. The percentage of carbon content increases with coal rank, as anthracite is the highest ranked coal, and lignite is the lowest ranked coal. The present study indicates that the studied coals are within the classification of low-rank coals.

The calorific values of the coal samples are presented in Table 6.

Table 6: Calorific values of selected coal samples using a bomb calorimeter

Parameters	AM	IY	OK	OU	OW	OY	RB
Calorific value (kj/kg)	29148	30282	30139	28028	28914	28940	9538

The calorific value, also known as the heating value, indicates of the quantity of a material required to produce a specific amount of energy, when burned [19]. The calorific values indicate that Ribadu coal has the lowest value, whereas Inyi coal has higher value than the other coals.

Ultimate analysis involves the determination of carbon, hydrogen, sulphur and nitrogen in samples. Figure 5 shows the results for the studied coal samples.
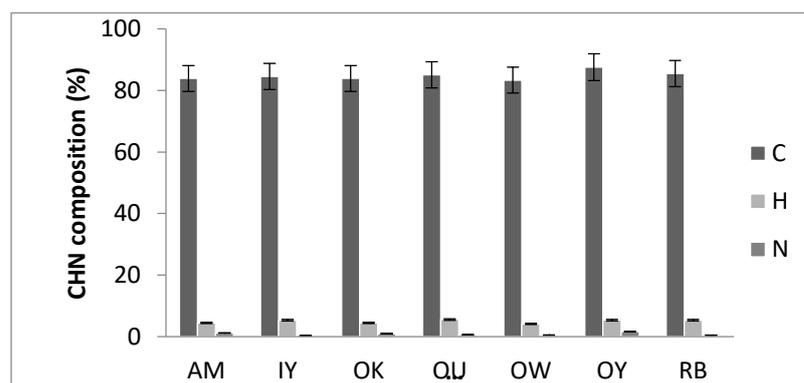

Fig. 5. CHN composition

The major environmental concern with coal is its emission of large amounts of carbon (IV) oxide which has been linked with contributions to climate change [1]. CO_2 is a greenhouse gas that may cause global warming if its amount in the environment is too high. The present study indicates that the combustion of Ogboyega coal has the greatest ability to pollute the environment with CO_2 as it has the highest

amount of carbon at 87.56% which can be oxidized to CO and then CO_2. CO is one of the criteria pollutants because of its potential harmful effects. Owukpa coal emits the lowest volume of CO_2 on the basis of its carbon content. UK coal has 74.15% carbon which is lower than the amount in the tested Nigerian coals [19].

The combustion of Ogwashi-uku coal produces more water vapour from 5.54% hydrogen, than does the combustion of Ogboyega and Ribadu coals which produce almost equal amounts of water vapour, whereas Owukpa coal has the lowest hydrogen content. Water vapour is also a greenhouse gas, which is an environmental pollutant.

More nitrogen oxides (NOx) are formed from the combustion of Ogboyega coal than from the other tested coals. This occurred because it has the highest nitrogen content at 1.65%. Inyi coal has the lowest N content at 0.29%. NOx in the environment may lead to the formation of acid rain which is deleterious to the environment.

Table 7 presents the results of EDXRF analysis for elements of selected coal samples.

Table 7: Elemental composition (mg/kg) of selected coals via Energy Dispersive X-Ray Fluorescence analysis

Coal mine	Fe	Si	Al	Mg	S	As	Ni	Cr	Cu	Cd	Pb
AM	7293.50 ± 3151.57	52875 ± 1492	31600 ± 1088.94	5000. ± 2687.01	16916.5 ± 1279.16	13.5± 19.09	49.6 ± 9.83	87.7 ± 4.24	316± 123.25	6868 ± 7964.85	738.5± 955.30
RB	5822 ± 196.58	218920 ± 4256.78	64030 ± 4044.65	13400± 3111.27	3370.5 ± 252.44	38± 48.08	35.4 ± 6.01	71.1 ± 5.73	110.8± 15.56	4200 ± 5939.70	187.5± 122.33
OY	11768 ± 8806.31	45855 ± 417.19	24345 ± 1788.98	350 ± 494.97	16858 ± 1735.24	335.5 ± 424.97	49.3 ± 8.84	66.6 ± 1.48	383.8± 232.00	4919.5 ± 5205.01	1905.5± 2621.24
IY	5052.5± 1212.69	36750 ± 3153.70	20820 ± 1202.08	2300 ± 3252.69	19889.5 ± 3751.20	0	73.2 ± 0.71	-52.95± 0.64	505.5± 68.59	5634 ± 6174.46	1354.5± 1690.69
OU	14847± 339.41	49890 ± 5953.84	16740 ± 3507.25	1800 ± 2545.58	15092 ± 1848.38	49.5± 49.5	53.5 ± 0.71	53.6 ± 16.19	326.9± 184.91	5622.5 ± 6190.72	67.4± 40.52
OW	9358± 2169.40	41070 ± 664.68	21115 ± 2184.96	2950 ± 3464.82	15808.5 ± 1198.55	0	37.2 ± 23.90	45.9 ± 8.49	333.25 ± 92.28	5000 ± 7071.07	735.5± 961.03
OK	19392.5 ± 2967.73	38250 ± 21920.31	13635 ± 5338.66	1150 ± 1626.35	17016 ± 4716.40	6.5± 9.19	30.9 ± 8.98	42.7 ± 0.28	322.2± 129.26	5643.5 ± 6161.02	1621.5± 2213.95

A suite of 23 trace elements was designated by the amendments to the United States Clean Air Act (1990) to be of greatest environmental concern [21]. Among the 23 elements, 14 were identified in

Ogwashi-uku coal, 13 were identified in Ribadu and Ogboyega coals, while 11 were found in Okaba and Inyi coals, 10 were identified in Owukpa coals and 9 were identified in Amansiodo coals.

The occurrence of sulphur in the tested coals is prominent. It is highest in Inyi, followed by Okaba coal, Ogboyega coal and Ribadu coal. Sulphur is of environmental concern because it can combine with oxygen during the combustion of sulphur-containing fuel to form oxides of sulphur (SOx). The present study reveals that the Okaba coal will form the largest amount of SOx, which is deleterious to the environment. The combination of SOx with water may lead to the formation of acid rain which acidifies soil and water, and may result in the death of plants, fishes and other animals that are exposed to contaminated acid rain environments. The tested fuels, except the Amansiodo and Ribadu coals, have sulphur levels above the acceptable limits of 1.5–1.6% [22]. The sulphur levels in the UK [19], Indiana [23], Australian, Indonesian and Chinese coals [24] are 1.2, 3.13, 0.6, 0.58, and 0.51% respectively.

Cadmium which may be toxic to the kidney, occurred at high levels in the tested coals, but was lowest in the Ribadu coal. The levels may be high compared with the maximum permissible limit (0.003 mg/l) in drinking water [25]. Pb is a toxic element that if emitted into the atmosphere, can be inhaled or ingested after it settles out of the air. Pb can cause illness among children and adults when it is absorbed in large doses. The present study reveals that the quantity of Pb is highest in Ogboyega coal and lowest in Ogwashi-uku coal. The levels may be too high compared with the maximum permissible limit of Pb in drinking water which is 0.1 mg/l [25]. The quantity of Zn is highest in the Inyi coal samples and lowest in the Ribadu coal samples. Zn, though an essential element, can be toxic if the dosage is too high. Arsenic is carcinogenic. It is present in the Ribadu, Ogboyega and Ogwashi-uku coals but not in the Amansiodo, Inyi, Okaba and Owukpa coal samples. Cu is also present in all the coals. Although Cu is an essential element, high doses may be toxic to the environment.

A comparison of the elemental composition of the samples in the present study to the compositions of coals obtained from other countries revealed that some of the elements that were not detected in Nigerian coals were present in UK, Australian, Indonesian and Chinese coals. All the detected elements in Australian, Indonesian and Chinese coals are more abundant in quantity than those in Nigerian coals [24].

Conclusions

This research characterized samples of Nigerian coals. The density, surface area, pore structure, morphology, thermal behaviours, chemical properties and chemical composition of the selected coals were investigated using sophisticated and appropriate instruments such as BET, CHN, EDXFR, SEM-EDX and thermogravimetric analysers. The tested Nigerian coals contained fewer toxic elements than some international coals did. The properties of coals vary due to different environmental factors during their formation. The present study provided valuable baseline data on the Ribadu, Ogboyega, Ogwashi-uku, Okaba, Inyi, Owukpa and Amansiodo coals. Information on quality parameters such as proximate composition and calorific values has been provided which are vital for the application of coal. Additionally, information from thermogravimetric analysis on devolatisation temperature may be

useful in the production of smokeless briquettes as it provides an indication of the temperature which the biquettes can be carbonized.

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Conflicts of Interest: The authors declare no conflict of interest.

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