Ultimate and Elemental Analysis of Some Common Charcoal in Southwestern Nigeria

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Abstract: Common charcoals from different wood species were collected, identified and prepared for analysis. Ultimate and elemental analyses were carried out. In this study, identified charcoal were characterized. The result showed that in the ultimate analysis, Carbon, Hydrogen and Oxygen are the major content of the charcoal samples and are of great importance for the heating value. Sulphur and Nitrogen content were resent in low in quantity. XRF was used in the elemental analysis and the following elements were detected (Na, Cr, Ni, Cd, Al, K, Mn, P, Ca, Si, Mg, Ti, Fe) In the charcoal samples, the percentages of Si and Al are high while the percentages of (Cd, Fe, Na, Ti, Ni, Mg, P, K, Cr) were low. Percentage of silicon was between 50% to 65% in Albizia zygia, Milicia excelsa, Terminalia avicennioides, Funtumia elastica, Milletia thonningii and Hevea brasiliensis with Hevea brasiliensis having the highest which suggested high slagging propensity. Anogeissus leiocarpa, Afzelia bipindensis, Vitellia paradoxum, and Burkea africana have high Na/K. The ratio of Ca/ Mg is greater than 2 in all the samples. LOI ranges from 54.56 – 7.88% with a mean of 25.97±22.909%.

Keywords: Charcoal, Analysis, Concentration, Ultimate, Elemental

1. Introduction

The use of fossil fuels has led to serious greenhouse gas emissions and environmental hazards. It is necessary to replace fossil fuels with alternative energy that meet energy demands. As a result of this, because it is a cheap, diversified and abundant resource biomass is of greater importance. Biomass energy can be further advanced, with sustainable development of new biomass resources and improvement of technologies for effective conversion of them [1]. Elements that are present in charcoal include carbon, nitrogen, hydrogen, oxygen, sulphur and other elements. This elements are either beneficial or harmful or both to the ecosystem. Charcoal is a solid biomass.

X-ray fluorescence (XRF) spectroscopy is one of the major well-grounded tools for the determination of the elemental composition of rocks, sediments and earth material samples ([2, 3, 4, 5]). For several years, it has been used for the determination of geochemical composition of several trace and important elements at a very high levels (ppm). This has also find its application in the successful investigation of samples from geological, archaeological and industrial site [5]. The principle used by XRF shows that every atom give off a wavelength feature or a relative abundance of X-ray photons of energy that can be calculated [4]. This method has been used in the investigation of Earth materials from a range of settings and environments ([6-11]).

Lignocellulosic materials contains metals such as Mg, Ca, Na, and K that are essential for plant growth. The composition of metals in charcoal may be increased by environmental growth with heavy metal contamination. Studies have shown that the presence of metal species, such as Mg, Ca, Na, and K, could greatly affect the character of lignocellulosic materials during pyrolysis ([12, 13])

Inorganic elements, like Fe, K, Na, Si, Mg, Al, and Ca, are widely present in biomass and they can significantly affect the pyrolysis techniques and products distribution [14]. The
essence of using biomass as a source of energy is closely linked with the problem of high environmental hazard as a result of their disposal. This cannot be excluded because biomass comprises of other elements aside C, H, and O that may be naturally hazardous [15]. In the process of biomass pyrolysis, these other elements are evenly distributed in the product like Cl, S, P and alkaline earth metals [16] and may (a) leads to critical damage to human health and the ecosystem; (b) cause serious operational problems like deposition, agglomeration, heated side corrosion and slagging; and (c) serve as catalyst in the pyrolysis process. ([17, 18]). Some heavy metal are also present in the biomass which can be harmful to human health and the environment at large such as Cd, Ni, Cr, As, Pb, etc. This study analyses the elements present in the charcoal samples and gives an overview of the influence and impact of these elements.

2. Materials and Methods

Ten charcoal samples were collected in southwestern Nigeria. The samples were identified and prepared for analysis. The charcoal species includes: Anogeissus leiocarpa (Ayin), Afzelia bipindensis (Ayan), Vitellaria paradoxa (Emi), Burkea Africana (Asapa), Albizia zygia (Ayunre), Hevea brasiliensis (Rubber), Milicia excelsa (Iroko), Terminalia avicennioides (Idi), Funtumia elastica (Ire), Milletia thonningii (Ito).

2.1. Study Area

The study area is south-western Nigeria and these includes of Lagos, Ogun, Oyo, Osun, Ondo and Ekiti states. It is also called the south west geographical zone of Nigeria and the map is shown in figure 1. The longitude of the area lies 2°31’ and 6°00’ East and Latitude 6°21’ and 8° 37’N [19] with about 77,818 km² land area and the population is about 32.5 million in 2006 [20]. South western, Nigeria is bounded in the North by Kogi and Kwara states, in the South by the Gulf of Guinea, in the East by Delta and Edo states and in the West by Benin Republic. The study area had a forest cover of 842,499 ha and 85 constituted forest reserves.

2.2. Ultimate Analysis

This analysis basically involves the determination of the percentage of C (Carbon), H (Hydrogen), O (Oxygen), N (Nitrogen), S (Sulphur).

2.2.1. Determination of Carbon and Hydrogen

A solid of a known mass is subjected to burning in dry oxygen so as to convert the hydrogen and carbon to water (H₂O) and carbon (IV) oxide (CO₂) respectively. After burning, the
combustion products are passed over weighed tubes of anhydrous calcium chloride (CaCl₂) and potassium hydroxide (KOH) which absorb H₂O and CO₂ respectively. The weight of water formed is represented by the increase in weight of CaCl₂ tube while the weight of carbon dioxide produced is represented by the increase in the weight of KOH in the tube. Percentage of Hydrogen (H) and Carbon (C) in the sample was estimated.

A is the weight of sample taken.
B is the increase in the weight of CaCl₂ tube.
C is the increase in the weight of KOH tube.

\[
\% \text{ of carbon in the sample} = \frac{2B}{18A} \times 100 \tag{1}
\]

\[
\% \text{ of hydrogen in the sample} = \frac{2C}{4A} \times 100 \tag{2}
\]

2.2.2. Determination of Nitrogen

Kjeldahal’s method was used in the determination of nitrogen in the specimen. 1.00g of the prepared solid waste specimen was weighed and recorded as \( w_s \) (g). The specimen was heated with concentrated tetraoxosulphate (VI) acid (H₂SO₄) in the presence of Potassium tetraoxosulphate (VI) salt (K₂SO₄) and Copper (II) tetraoxosulphate (VI) salt (CuSO₄) in a long necked flask called Kjeldahal’s flask so as to convert the nitrogen in the specimen into ammonium sulphate (NH₄)₂SO₄. The formation of a clear solution showed that all the nitrogen has been converted to ammonium sulphate (NH₄)₂SO₄, 50% of Sodium hydroxide (NaOH) was used for treating the resulting solution.

The ammonia formed was distilled over and absorbed in a certain amount of standard H₂SO₄ solution. Titration against standard solution of NaOH was used to determine the volume of un-used acid. The quantity of acid neutralized by released NH₃ from the specimen was then evaluated.

(Titre Value) \( V_t = V_1 - V_2 \) (cm³)
Where \( V_1 \) = volume of H₂SO₄ neutralized (cm³)
\( V_2 \) = volume of H₂SO₄ neutralized in determination (cm³)
\( w_s \) = weight of the specimen (g)

\[
\text{Nitrogen in the specimen} = \frac{0.00455 \times V_t}{w_s} \times 100 \tag{3}
\]

2.2.3. Determination of Sulphur

A specimen of 25 ml were weighed, dissolved and pipetted into 50ml standard flask observed through 20 ml gelatine BaCl₂ solution and scaled up to 50 ml mark. The solutions were permitted to stay for 0.5 hour. The Spectrophotometer was used to study the absorbance of the standard solution at 420 nm. The specimen concentration was estimated by plotting the graphs of absorbance against concentration of standards. Percentage of sulphur in the specimen was evaluated using the following equation. The average value for each specimen was calculated when the processes were repeated twice.

\[
\% \text{ of Sulphur in the Specimen} = \frac{R \times V \times Df}{\text{weight of sample used}} \tag{4}
\]

Where: \( R \) = Reading from the graph
\( V \) = 25 ml (total volume)

\( Df \) = Dilution factor

2.2.4. Determination of% of Hydrogen and Oxygen in the Sample

The addition of the % of sulphur, total carbon and nitrogen subtracted from 100 will give the % of oxygen and hydrogen in the specimen.

\[
\% H + \% O = 100 - (\% of C + N + S) \tag{5}
\]

Determination of Elemental Composition

Samples were prepared at the Environmental Laboratory of the Department of Chemical Engineering, Obafemi Awolowo University Ile-Ife, Osun state. The samples were dried and the dried samples were pulverized into a fine powder with an agate mortar and pestle. During preparation cover both faces of the compression die to avoid contamination and the mixture of samples. Each pulverized ground sample (powder pellet) was then weighed to be about 5g prior to analysis. These samples were analyzed with a Shimadzu EDX-720HS which allows the quantitative analysis of the elements and gives high X-ray intensity. To perform the loss of ignition (LOI) test, a crucible of dried sample 1.0 g was placed in a furnace and ignited at 900°C for 30min to obtain a constant mass. The LOI was estimated using the formula below:

\[
\text{LOI} = \frac{M_0 - M_1}{M_0} \times 100 \tag{6}
\]

3. Results and Discussion

The result of ultimate analysis which includes carbon content, hydrogen content, oxygen content, sulphur content and nitrogen content are shown on figure 2 – figure 6.

The result shows the variation of different element in the charcoal samples. The content of carbon is higher than any other element. It expected that high carbon content should increase the calorific value. The carbon content ranges from 76.50% for Afzelia bipindensis (Ayan) to 86.05% for Hevea brasiliensis (Rubber). The hydrogen content varies from 5.24% for Milicia excelsa (Iroko) to 10.40% for Burkea africana (Asapa). The oxygen content ranges from 2.67% for Afzelia bipindensis (Ayan) to 17.11% for Burkea africana (Asapa). Biomass compared to fossil fuels with relatively low sulfur content in the wood 0.02 to 0.05 wt.% [22]. Sulphur in the burning process is converted to sulfur oxides which are pollutant to the environment and also be irritating to the human respiratory system. ([23, 24, 25]). The Sulphur content ranges from 0.00% for Burkea africana (Asapa), Funtumia elastica (Ire) and Milletia thonningii (Ito) to 1.03% for Hevea brasiliensis (Rubber). The nitrogen content varies from 0.15% for Burkea africana (Asapa) to 0.71% for Afzelia bipindensis (Ayan). The percentage of each content were presented in figure 2 – figure 6.
Silicon exist as silica (\( \text{SiO}_2 \)) or several silicate minerals. During combustion, usually the several metals associated with silicate remains in the silicate matrix. Combustion in fluidized beds is not affected by Silicates. The ash produced by silicate is usually high melting and majority of the silicate ashes are crystalline in nature under fluidized bed conditions and they don’t cause sintering, slagging, fouling, or corrosion problems [26]. During burning, low melting potassium silicate are formed when they react easily with each other and these results in fouling and sintering problem in the fluidized bed. Though their behaviour is rare, the combustion of their silicates and silica may affect the chemistry of other elements that can form ash majorly Ca, Na, and K by minor reaction hence causing directly the fouling- corrosion properties of fly ash [26]. The Si percentage between 72% and 80% has low slagging propensity. Medium slagging propensity when it is between 65% and 72% and high slagging propensity when the Si percentage is between 50% and 65% [27]. *Hevea brasiliensis* (rubber), *Millettia thonningii* (ito), *Funtumia
Elastica (ire), Terminalia avicennioides (idi), Milicia excelsa (iroko), and Albizia zygia (ayunre) have Si percentage between 50% and 65%.

Aluminum exists in various forms in charcoal. It form alumina, Al₂O₃, in the furnace processes which is a solid (that cannot react) and will not be involved in the chemistry of the ash in the fluidized bed system. In few solid fuels, Aluminum may occur as silicates when the fuel is contaminated with clay or other impurities. Aluminum silicates are less reactive although they can react with gaseous alkaline, hence affecting the chemistry of the flue ash alkaline. Aluminum metals have the ability to oxidize to alumina Al₂O₃(s) in combustion processes. They have low melting points. In the furnace or superheaters, it will form agglomerates easily and deposits on tube surfaces (Zevenhoven et al., 2010). Hevea brasiliensis (rubber), Milletia thonningii (ito), Funtumia elastica (ire), Terminalia avicennioides (idi), Milicia excelsa (iroko), and Albizia zygia (ayunre) have concentration ranging from 23.08 g/kg to 28.12 g/kg. Rubber and Idi have the highest while Ayunre has the lowest concentration. High values of Si: Al ratio and increased ash yield are an indication of charcoal formed under unstable conditions as shown in Figure 15. A higher Si: Al ratio that is a value above 2, shows that the ash fusion temperature changes slightly or remains constant [28]. Hevea brasiliensis (rubber), Milletia thonningii (ito), Funtumia elastica (ire), Terminalia avicennioides (idi), Milicia excelsa (iroko), and Albizia zygia (ayunre) have Si: Al ratio greater than 4 that is the ash fusion temperature is not serious affected.

Iron exist in different state in solid fuels. During burning, different form of Fe will produce iron oxides – Fe₂O₃, FeO, and lastly Fe₃O₄. It may also be formed under strongly reducing conditions. Compounds of iron can produce first deposits on furnace wall tubes in PF (pulverized fuel) boilers, when temperatures of gas in the furnace are high [29]. Iron is very useful in the body as it helps to transport oxygen to the tissue from the lungs by the red blood cell hemoglobin where it acts as a medium of transportation for electrons within the cells. For many enzymatic reactions, iron serves as one of the major enzyme systems in numerous cells where it serves as a cofactor. It is also present in oxygen storing protein myoglobin and hence leading to decrease in acid due point corrosion. The presence of Ca and Mg can be associated with calcite and dolomite minerals. When the weight percentage ratio of Ca: Mg is greater than 1, then dolomite is present as shown in Figure 16. Therefore dolomite is present in all except Afzelia bipindensis, (ayin). Usually, calcium dominates over magnesium.

Magnesium induces muscle relaxation. The combustion chemistry of magnesium is less likely when compared with calcium. Ca and Mg undergo the same reactions but for Mg, the temperature is very low that it cannot actually have importance in burning processes. In combustion systems, Magnesium oxides are usually considered as rare compound. Magnesium as an essential element represents a moderately mobile Mg component is biomass [30]. One difference is a corrosion additive of low temperature with magnesium. MgO can react when the temperature is low, selectively with SO₃ and hence leading to decrease in acid due point corrosion. Hevea brasiliensis (rubber) 3.20 g/kg, Milletia thonningii (ito) 2.17 g/kg, Funtumia elastica (ire) 1.93 g/kg, Terminalia avicennioides (idi) 1.69 g/kg, Milicia excelsa (iroko) 1.87 g/kg, and Albizia zygia (ayunre) 2.53 g/kg.

During biomass burning, Potassium is the most intriguing element. The higher its concentration, the lower ash melting temperature and therefore results to the production of deposits on the furnace surfaces, corrosion and agglomeration. From the fireside problems point of view, the most interesting ash-forming element is potassium [26]. The challenge of bed sintering, fouling and corrosion is caused by Potassium in biomass fuels. It is important to know the different forms in which potassium exist in the fuel. Here, the selective leaching analysis of the ash-forming matter is very important. Silicate contaminations of the fuel are usually the insoluble part of K. This part of K is not involved in the major chemistry of the most reactive forms of K. [26]. In solid fuels. Potassium is usually not soluble and occurs as salt in ionic form in the aqueous fluids of the plant or as directly organically bound K ions. These compounds of K is efficiently liberated in the burning process and hence mostly present for any further reactions in the flue gases therefore lowering the ash melting temperature. Potassium reacts Sodium dioxide from the flue gases into the fly ash. In this situation where there is high quantity of alkaline earth metals in the fuel this result in a noticeable decrease in the quantity of SO₃ in the flue gases. Increase in the concentration of K results in reduction in the ignition delay time [32]. Hevea brasiliensis (rubber) 13.70 g/kg, Milletia thonningii (ito) 14.44 g/kg, Funtumia elastica (ire) 13.53 g/kg, Terminalia
Sodium exist in many biomasses though with lower concentration than that of potassium as observed in this study. Moreover, the concentration of sodium is lower in magnitude when compared with potassium. Sodium and potassium have almost the same chemistry. [33] noted that when the concentration of potassium is higher than that of sodium, it leads to a low Na/K ratio which is helpful from nutritional point of view as hypertension is associated with high Na/K ratio (Figure 17). Increase in ash deposition and fouling boiler is due to lower melting point of sodium and potassium ash thereby forming deposits on the surface of the furnace. High levels of sodium found in many low-rank coals have been found to be responsible for fouling in the combustion chambers [34]. They all have low concentration of sodium which means fouling will be reduced in combustion.

Sulphur is found in biofuel as both as organic sulphur and inorganic sulfate anions. Studies showed that about 75% sulphur in biomasses (woody) is bound organically and 25% as soluble alkali sulphates [35]. In plant biomass Sulphur is absorbed through non-organic sulphate forms and transferred through the plant in which sulphate is converted to sulphide and bound with organic molecules to form cysteine (essential amino acid found in proteins [36]. In combustion, only organic Sulphur will be liberated in combustion – mainly as hydrogen sulphide and other reduced gaseous species are then converted (oxidized) to Sulphur dioxide. Sulphur can be associated to the alkali sulphate, it may also be liberated as a result of some decomposition reaction of the alkali sulphate. Majority of the sulphur in biofuels are liberated during burning and will lastly yield Sulphur dioxide which is poisonous to human health at an elevated temperature. *Hevea brasiliensis* (rubber) 15.94 g/kg, *Milletia thonningii* (ito) 13.70 g/kg, *Funtumia elastica* (ire) 22.35 g/kg, *Terminalia avicennioides* (idi) 17.98 g/kg, *Milicia excelsa* (iroko) 14.50 g/kg and *Albizia zygia* (ayunre) 15.46 g/kg.

The concentrations of Titanium are low in all the charcoal samples which show that no impurities are present in the biomass. When concentration of TiO₂ is very high, it shows that the biomass is contaminated [31]. This non-essential mostly represents less mobile components in biomass [31]. Because natural biomass does not possess such a high concentration of Ti.

During fossil fuel combustion Cr is liberated and deposited in lungs. It found in low concentrations; and participates in metabolic processes of saccharides (carbohydrates) and lipids in organism [37]. Universally, the corrosion resistance of a material depends on the concentration of Cr and Ni. The concentration of Cr and Ni is directly proportional to the corrosion resistance. *Hevea brasiliensis* (rubber) 0.79 g/kg, *Milletia thonningii* (ito) 1.02 g/kg, *Funtumia elastica* (ire) 0.85 g/kg, *Terminalia avicennioides* (idi) 0.90 g/kg, *Milicia excelsa* (iroko) 0.74 g/kg and *Albizia zygia* (ayunre) 0.85 g/kg.

Nickel (Ni) is believed to be an important element [37], but it is also significantly toxic. The most of all the nickel compounds that are released to the air will adsorb to sediment or soil particle therefore becoming immobile. In acidic ground however, nickel will become more mobile. Nickel exist in small amount as essential foodstuff in animals. But nickel is not only beneficial; it can be dangerous when the maximum tolerable amounts are exceeded [38].

However, there is a substantial difference between the concentration required for normal metabolism functioning and the toxic one. Higher Ni concentrations originate during combustion [24], where the most toxic Nickel compound – tetracarboxylynickel Ni(CO)₄ which penetrates through body system when inhaled by lungs. From toxicological standpoint, Nickel compound intoxication can cause carcinogenity of respiratory tract and allergic contact dermatitis. *Albizia zygia* (ayunre) 8.96 g/kg, *Hevea brasiliensis* (rubber) 9.59 g/kg, *Milicia excelsa* (iroko) 8.57 g/kg, *Terminalia avicennioides* (idi) 10.53 g/kg, *Funtumia elastica* (ire) 10.13 g/kg, *Milletia thonningii* (ito), 10.14 g/kg. That is, the charcoals are free from the negative effect of nickel emission.

The origin of cadmium in biomass is deposition in the dissipation of Cd by human activities, most notably fossil energy use. Cadmium (Cd) is found in biomass in low concentrations where it penetrates from polluted soils due to industrial pollution. Liberating of Cd occurs also during burning of fossil fuels and Cd can penetrate into soil ([24, 37]). Cadmium adsorbs to organic matter in the soils. It is very dangerous when it is present in the soils as the uptake through food will increase. Acidified soils increase the cadmium uptake by plants. This pose great danger on human and animals depending on such plants for survival. Cadmium can accumulate in the body when such plant are consumed often. Cows may have large amount of cadmium in their kidney through this [38].

Based on their doses, trace metals are harmful and can contaminate meats when they accumulated in the tissues [39]. Renal tube dysfunction have al so been linked to the presence of some levels of cadmium [40]. *Albizia zygia* (ayunre) 0.35 g/kg, *Hevea brasiliensis* (rubber) 0.088 g/kg, *Milicia excelsa* (iroko) 0.70 g/kg, *Terminalia avicennioides* (idi), 0.35 g/kg, *Funtumia elastica* (ire) 0.35 g/kg, *Milletia thonningii* (ito) 0.44 g/kg. High loss on ignition suggested that temperature were not high enough and heating is not uniform due to the presence of undecomposed CaCO₃. The descriptive statistics for the elemental composition are presented in Table 1. Figures 7 -14 showed the elemental composition of the charcoal samples and figures 7-17 showed the elemental ratio.
Figure 7. SiO$_2$ content (%) in the Charcoal Species.

Figure 8. Al$_2$O$_3$ content (%) in the Charcoal Species.

Figure 9. Fe$_2$O$_3$ content (%) in Charcoal Species.
Figure 10. MgO content (%) in the Charcoal Species.

Figure 11. CaO content (%) in the Charcoal Species.

Figure 12. Na₂O content (%) in the Charcoal Species.
Figure 13. $K_2O$ content (%) in the Charcoal Species.

Figure 14. $MnO$ content (%) in the Charcoal Species.

Figure 15. Charcoal Si/Al ratio in the samples.
Figure 16. Charcoal Ca/Mg ratio in the samples.

Figure 17. Charcoal Na/K ratio in the samples.

Table 1. Descriptive Statistics of Elemental Composition of the Charcoal.

| Element | Mean   | Standard Deviation | Maximum  | Minimum  | Range    |
|---------|--------|--------------------|----------|----------|----------|
| SiO₂    | 179.75 | 142.19             | 294.16   | 11.92    | 282.24   |
| Al₂O₃   | 47.73  | 32.58              | 74.25    | 9.63     | 64.62    |
| Fe₂O₃   | 97.73  | 94.53              | 213.61   | 23.08    | 190.53   |
| MgO     | 5.14   | 3.83               | 10.43    | 1.69     | 3.74     |
| CaO     | 7.49   | 4.78               | 13.75    | 2.57     | 11.15    |
| Na₂O    | 5.185  | 0.77               | 6.16     | 4.08     | 2.08     |
| K₂O     | 12.82  | 3.14               | 17.27    | 8.80     | 8.47     |
| MnO     | 7.73   | 0.93               | 8.60     | 5.96     | 2.64     |
| TiO₂    | 3.45   | 2.43               | 6.11     | 0.48     | 5.63     |
| P₂O₅    | 2.16   | 0.66               | 3.27     | 1.13     | 2.14     |
| SO₃     | 16.66  | 3.15               | 22.35    | 13.7     | 8.65     |
| Cr₂O₃   | 0.859  | 0.097              | 1.02     | 0.74     | 0.28     |
| NiO     | 9.65   | 0.76               | 10.53    | 8.57     | 1.96     |
| CdO     | 0.38   | 0.20               | 0.70     | 0.088    | 0.61     |
| LOI     | 25.978 | 22.909             | 54.56    | 7.880    | 46.68    |
4. Conclusion

The results of the elemental composition showed that the concentration of sodium, Potassium, magnesium, manganese and titanium in all the charcoal samples are low 1.69 – 17.27 mg/kg. Concentration of chromium, nickel, and cadmium were also low 0.088 – 10.53 mg/kg, but the accumulation can cause severe damage to human health. Percentage of silicon was between 50% to 65% in Albizia zygia, Milicia excelsa, Terminalia avicennioides, Funtumia elastica, Millietia thonningii and Hevea brasilensis having the highest which suggested high slagging propensity. Anogeissus leiocarpa, Afzelia bipindensis, Vitellia accumlation can cause hypertension. The ratio of Ca/Mg is having the highest which suggested high slagging pr opensity.

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