

Nigerian Hardwood (*Nesogordonia papaverifera*) Sawdust Characterization: Proximate Analysis, Cellulose and Lignin Contents

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In order to advance research into the conversion of biomass feedstock to other forms of sustainable energy in Africa, data on the characteristics and fuel properties of the indigenous feedstock is required. These data are seldom available, resulting in the use of data on other similar feedstock from other countries for process design and calculations. This study is aimed at experimentally determining the proximate components of ash content, moisture content, volatile matter, fixed carbon, extractive content, cellulose and lignin content, as well as the Higher Heating Value (HHV) of the sawdust from an indigenous Nigerian hardwood. The results obtained showed an ash content of $0.13\pm 0.05\text{w.t}\%$, moisture content of $7.5\pm 0.13\text{w.t}\%$, and volatile matter of $81.25\pm 1.2\text{w.t}\%$. Fixed carbon was obtained as $11.12\text{w.t}\%$. Cellulose content was obtained as $41.025\pm 1.54\text{w.t}\%$, while lignin content was obtained as $19.02\pm 1.05\text{w.t}\%$. The higher heating value estimated was found to be approximately 17915 kJ/kg. These parameters suggest that the hard-cross hardwood could be suitable for bio-oil production.

Keywords: Hardwood; Nigeria; Proximate components; Cellulose; Klason lignin; Sawdust; Africa

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INTRODUCTION

Biomass has been recognized as a major renewable energy source to supplement declining fossil fuel sources of energy, and it is expected to play a pivotal role in the world's future energy supply (Ahiekpor and Kuye 2015). Researchers over the past three decades have been investigating the conversion of biomass to other sustainable energy forms that can easily be transported and upgraded. These efforts are far advanced in many industrialized and emergent countries; however, the same cannot be said for many developing countries especially in Africa. Ironically, many of these African countries are replete with biomass resources and agricultural residues. It is estimated that over 2,000 sawmills exist in Nigeria. These sawmills generate tonnes of wood waste daily, which is indiscriminately disposed, causing a menace and a major source of environmental pollution resulting in global warming (Ahiekpor and Kuye 2015).

Data on the characteristics and fuel properties of the indigenous biomass feedstock is required normally required when considering the processing of biomass to other forms of fuel. It is usually assumed that tropical hardwood species have similar properties. However, previous studies on other hardwood species from similar

geographical regions suggest that its properties can sometimes vary significantly (Turn *et al.* 2005; Adegoke *et al.* 2014; Mitchual *et al.* 2014). These studies suggested that a biomass material should be properly assessed of its properties before selecting it for a particular fuel purpose.

This work seeks to determine the fuel properties of Nigerian indigenous hard-cross hardwood (*Nesogordonia papaverifera*) sawdusts with a view to affirming if it could be used as a feedstock for bio-oil production. The properties determined include ash content, moisture content, volatile matter, fixed carbon, organic content, cellulose, and lignin composition. The higher heating value of the biomass is also calculated.

EXPERIMENTAL

Materials and Methods

The sawdust was obtained from Illobuchi sawmill, Port Harcourt, Rivers state, Nigeria. The reagents included sodium chlorite (Manufacturer: Sigma-Aldrich, assay: 80 percent), and acetic acid (Sigma-Aldrich, density: 1.05 g/cm³) and 98% sulphuric acid (Sigma Aldrich, 1 mmHg, density: 1.840 g/ml at 25 °C) and distilled water.

Major equipment used for this research included a heating element (J.P. Selecta S.A, 3003145, S/N 0548400, 230V, 50/60 Hz, Spain), muffle furnace (J.P. Selecta, S.A, 582543 S/N, 230 VAC, 00-C/2000367, 50/60 Hz, 3500W, Spain), precision balance (RADWAG electronics, 2013, Poland, Presicion: 0.001 grams), and an electro-magnetic sieve shaker (BA 200N, 01006, 230 V, 50/60 Hz, 450 VA, Spain).

Proximate Analysis

The proximate analysis was carried out as follows:

Ash content

The ash content was determined following ASTM methods (ASTM E830, 2008). In this procedure, a porcelain crucible was weighed and oven dried until its weight was constant. A sample weight of 2g was then measured and placed in the crucible. The crucible and content were then weighed to the nearest 0.001g. The sawdust sample was then ashed at 550 °C. Ashing was conducted for 4 hours at a regulated temperature range of 550±5°C. After 4 hours, the crucible was carefully withdrawn and cooled in a dessicator. After cooling, it was weighed to the nearest 0.001g. This procedure was conducted in triplicate. The ash content was calculated using Eq. 1,

$$PAC = \frac{Z}{A} \times 100$$

(1)

Where Z is the weight of ash, in grams (g), and A is the weight of the oven dried sample at 105°C, in grams (g)

Percent moisture content

To obtain the percent moisture content, 2g of the sawdust sample was placed in a porcelain crucible. The crucible was weighed. It was then dried at 105 ± 5 degrees Centigrade for 60 minutes. After 60 minutes, the crucible was withdrawn and cooled. The crucible was then weighed to the nearest 0.001g. This procedure was conducted in triplicate. The moisture content was found on a percentage basis using Eq. 2. (Akowuah *et al.* 2012),

$$PMC = \frac{D}{E} \times 100$$

(2)

where *D* is the weight change, in grams (g), and *E* is the original weight of sawdust, in grams (g).

Percent volatile matter

A weight of 2g of sawdust sample was measured into the crucible and was oven dried at 105°C for 1 hour. The oven dried sample is cooled in a desiccator and weighed. The cooled and dried sample was then heated at a temperature range of $550 \pm 5^{\circ}\text{C}$ for 10 minutes. The crucible was then withdrawn and cooled. After cooling, the crucible was then weighed to the nearest 0.001g. This procedure was conducted in triplicate. This parameter was then calculated on a percent basis using equation 3 (ASTM D2974, 2002),

$$PVM = \frac{A - B}{A} \times 100$$

(3)

Where *A* is the weight of the oven-dried sample at 105°C , in grams (g), and *B* is the weight of the sample after 10 min in the furnace at 550°C , in grams (g).

Percent fixed carbon

This proximate component was determined on a percentage basis using Eq. 4 (Akowuah *et al.* 2012):

$$\text{FixedCarbon} = 100\% - (\text{PAC} + \text{PVM} + \text{POC})$$

(4)

Extractive Content

Water soluble extractives

A paper tray was wiped and weighed to the nearest 0.001g. It was then dried at $105 \pm 5^{\circ}\text{C}$ until it assumed a constant weight. The sawdust was placed in the paper tray and weighed to the nearest 0.001g. The combined weight of the tray and the sawdust sample was then recorded. The sample was charged into a porous thimble and then placed inside the Soxhlet extractor. 500 mL of water was dispensed into a 500 ± 50 mL round bottom flask, which was then placed on a heating mantle. The Soxhlet extraction apparatus was then coupled to the 500 mL flask. And extraction was conducted for 4 hours. After the extraction, the supernatant was collected. It was evaporated until it reached a volume of about 15 ± 5 mL. Then it was dried at $105 \pm 5^{\circ}\text{C}$ until a constant

weight was reached (ASTM E1690, 2003). The residue obtained thereafter represents the water soluble extractives.

Ethanol soluble extractives

To conduct this Compositional analysis, the sample was dried until there was no variance in weight. The dried sample was then weighed and charged into a thimble and then placed inside the Soxhlet extractor. 360 mL of water was then measured using a volumetric cylinder and dispensed into a 500 mL round bottom flask, which was then placed on the heating mantle. The soxhlet extraction mechanism was then coupled to the flask. After the extraction was conducted for 4 hours using ethanol, the supernatant was collected. It was evaporated until it reached a volume of about 15 ± 5 mL. It was then dried at 105 ± 5 °C until a constant weight was reached. The final residue represents the ethanol soluble extractive of the biomass.

Total extractive content

The total extractive content represents the summation (by weight) of the water and ethanol soluble extract,

$$X_E = X_{WE} + X_{EE} \quad (5)$$

where X_E is the total extractive content, wt%, X_{WE} is the water soluble extract, wt%, and X_{EE} is the ethanol soluble extract, wt%.

Cellulose Content

The Base-Acid-Base-Acid-Base method as described by (ASTM 1721, 2009), was used for the cellulose extraction. After extraction, the wood was washed with ultrapure water to pH 4. The final product (Cellulose) was dried in a muffle furnace at a constant temperature of 105 °C for 4 hours. The extraction was done in triplicates for fine (size range: 425 - 300 µm), fairly coarse (size range: 710 - 500 µm), coarse (size range: 1mm - 700 µm), and very coarse (size range: greater than 1 mm) particle sizes. The final product suspected to be cellulose was then subjected to a tri-iodide phosphoric acid confirmatory histochemical test.

Lignin Content

The Klason Lignin was extracted using ASTM D1106. It involves the use of 72% sulphuric acid to dissolve all the carbohydrates in the extracted-free wood sample leaving lignin as the insoluble residue. The extraction was done in triplicates for fine (size range: 425 - 300 µm), fairly coarse (size range: 710 - 500 µm), coarse (size range: 1mm - 700 µm), and very coarse (size range: greater than 1 mm) particle sizes. The residue was washed with 500 mL of hot water to remove any other contacted acid. Thereafter, it was dried at 105 ± 5 °C for 2 hours. After drying, it was then weighted as the weight of the Acid Insoluble Lignin. The percent weight of Lignin was found using Eq. 6,

$$X_L = (W_{CL} - W_C) \times 100 \quad (6)$$

Where X_L is the weight percent of lignin content, W_{CL} is the weight of crucible and lignin, grams, and W_C is the weight of the crucible used to measure, grams.

Higher Heating Value (HHV)

According to White (1987), the HHV of wood species can be determined using,

$$h_o = 7696 + 32X_L + 28.4X_E \quad (7)$$

$$h_c = 7572 + 36.7X_L \quad (8)$$

Where h_o is the higher heating value of the unextracted wood, h_c is the higher heating value of the extracted wood, X_L is the klason lignin content, wt%, and X_E is the extractive content, wt%

RESULTS AND DISCUSSION

Each of the experimental procedures described in the previous section was repeated nine (9) times. The averaged results with their deviations are presented in Tables 1 to 4.

Table 1. Proximate Analysis

Proximate components	Mean, % wt (As received basis)
Ash content	0.13±0.05
Moisture content	7.5±0.13
Volatile matter	81.25±0.11
Fixed carbon	11.12±0.03

Table 2. Higher Heating Value (HHV)

Sample	HHV (kJ/kg)
Non-Extracted sample, h_o	17915.20
Extracted sample, h_c	17915.14

Table 3. Cellulose Content

Particle Size	Mean cellulose content, wt%
Very coarse ($\geq 1\text{mm}$)	41.1±0.3
Coarse ($\geq 710 \mu\text{m}$)	40.9±0.4
Fairly coarse ($\geq 500\mu\text{m}$)	41.0±0.3
Fine ($\geq 300 \mu\text{m}$)	41.1±0.2

Table 4. Lignin Content

Particle Size	Lignin content, wt%
Very coarse ($\geq 1\text{mm}$)	22.67±0.80
Coarse ($\geq 710 \mu\text{m}$)	18.12±0.15
Fairly coarse ($\geq 500\mu\text{m}$)	18.23 ±0.12
Fine ($\geq 300 \mu\text{m}$)	17.03±0.12

Ash Content

The ash which is the non-combustible constituent of the sawdust sample is shown in Table 1. The value of 0.13% is lower than the values reported in the literature as indicated in Table 5. This indicates that the *Nesogordonia papaverifera* wood has a low mineral matter. Ash is an impurity that will not burn, so biomass with low ash content are better suited for pyrolysis than biomasses with higher ash content.

Table 5. Properties of Different Wood Species

Source/Species	Ash content (w.t%)	Moisture content (w.t%)	Volatile matter (w.t%)	Fixed carbon (w.t%)	HHV (MJ/kg)	Cellulose (w.t %)	Lignin (w.t%)
Mitchual et al. (2014)	0.61 – 5.04		75.23 - 80.60		20.89 - 22.22		
Salehi et al. (2013)	1.46	5.96	81.15	11.42			
Schild et al. (2010)	0.4	11.9			17.63	42.4	22.7
Telmo and Lousada (2011)		7.3			19.09		
Ververiset al. (2003)						37.7 – 40.7	20.5 – 27.3
White (1987)					19.65		21.7
This work	0.13	7.5	81.25	11.12	17.92	41.03	19.02

According to Kim *et al.* (2001), the combustion volume and efficacy of a fuel reduces with increasing ash content. Also the ash content of any biomass has a significant influence on the heat transfer to the surface of the biomass as well as the diffusion of oxygen to the biomass surface during combustion (Kim *et al.* 2001; Mitchual *et al.* 2014). This suggest that higher ash content in biomass could antagonise the bio-oil yield of a pyrolysis process (Jahirul *et al.* 2012).

Moisture Content

From Table 1, it can be seen that the moisture content of the sample was $7.50 \pm 0.13 \text{ wt\%}$. The moisture content determines the total energy that is needed to bring the biomass up to disintegrate during pyrolysis. It also affects the internal temperature within the biomass (Holt-Giménez 2007). The higher the moisture content of the biomass, the more prolonged the pyrolysis process due to endothermic evaporation within the biomass, which will consequently prolong its residence time within the reactor. Higher biomass moisture also affects the quality of bio-oil. Moisture content of less than 10 wt% is usually recommended for feestocks intended for use for bio-oil production to minimize moisture content in bio-oil produced (Ibrahim *et al.* 2012). A higher moisture content results in phase separation of the bio-oil and also affects the quality of bio-oil in terms of viscosity and density (Kuye *et al.* 2016).

Volatile Matter

Volatile matter was obtained as $81.25 \pm 1.2 \text{ wt\%}$. Biomass generally contains high volatile matter (Mohan *et al.* 2006). Our value confirms this and it is also within the range of values reported by previous authors as shown in Table 5. This fuel property makes the biomass sample, a highly reactive fuel. Biomass with high volatile matter give

a faster combustion rate while devolatilizing during pyrolysis. Biomasses with low volatile, have been reported to result in smouldering which leads to a significant amount of smoke and release of toxic gases(Akowuah *et al.* 2012).

Fixed Carbon

The fixed carbon, calculated using Eq. 4, was $11.12\pm 0.03\text{wt}\%$. The fixed carbon fuel property represents the amount of carbon available for char combustion during pyrolysis (Mohan *et al.* 2006). Higher fixed carbon favors the production of char during pyrolysis process.

Effect of Particle Size on Cellulose Content and Lignin Content

As shown in Table 3, the cellulose content for hardwood sawdust was approximately 41wt%. Table 3 further indicated that the cellulose content was independent of the sawdust particle size. This value falls within the range of values obtained by Schild *et al.* (2010) and Ververis *et al.*(2003), as indicated in Table 5. It has also been reported that cellulose content for hardwood species fall within the range 40 to 47% (Mohan *et al.* 2006).

Table 4 shows the effect of particle size on lignin content. The maximum acid insoluble residue was obtained for the very coarse particle size of sawdust at 22.7 wt%. The larger the particle sizes the higher the lignin content for the range of particle size considered. Our sawdust ranged from 300 μm to 1000 μm .

CONCLUSION

Proximate and compositional components of indigenous Nigerian hardwood sawdust have been determined using available tools and analytical procedures. The proximate components of ash content, moisture content, volatile matter and fixed carbon were obtained as $0.13\pm 0.05\text{wt}\%$, $7.5\pm 0.13\text{wt}\%$, $81.25\pm 1.2\text{wt}\%$, and $11.13\pm 0.03\text{wt}\%$, respectively. The cellulose and lignin content of the hardwood was found to be $41.03\pm 1.54\text{ wt}\%$ and $19.02\pm 1.05\text{ wt}\%$ respectively. The cellulose content was independent of the sawdust particle size while the lignin content varied with the sawdust particle. The higher heating value (HHV) for non-extracted sample and extractive free sample were estimated at 17915.20 and 17915.14 kJ/kg respectively. The low ash content and the high volatile matter of the hardwood sawdust make it suitable for use as feedstock for bio-oil production.

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