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Determination of Proximate, Ultimate and Structural Properties of Elephant Grass As Biomass Material for Bio-oil Production

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ABSTRACT: Elephant grass was analyzed for its chemical properties, proximate, ultimate and structural composition and high heating value using the American Standard for Testing and Materials. (ASTM) methods. Results of proximate analysis showed moisture and ash content, fixed carbon and volatile matter content to be 0.85%, 5.86%, 14.22% and 79.24% respectively. The composition of ultimate analyses showed carbon, hydrogen, nitrogen, oxygen and sulphur content to be 54.44%, 5.59%, 0.67%, 40.95% and 0.35% respectively. The results of structural analyses showed cellulose, hemicelluloses, lignin and high heating value to be 46.26%, 29.90%, and 24.60% and 18.52MJ/kg respectively. The results of proximate, ultimate and structural analyses and high heating value showed an indication that the elephant grass is suitable for pyrolysis process because a relatively higher bio-oil yield would be expected.

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The primary source of energy that initially sparked technological development in human lives is the utilization of fossil fuels. However, significant environmental impacts and depletion of fossil fuel reserves lead relevant authorities and governments to concentrate on renewable energy resources. Recently, the quest for petroleum-derived fuels is tremendously in high increase due to economy and population growth. These conventional fuels may not be able to sustain for the next 100 years, as they are limited in nature (Safana*et. al.,* 2018). In response organizations and researchers have proposed transition to an economy of 100% renewable energy as the solution.

The conversion of biomass to bio-fuel can be achieved broadly through various means, physical, biological, biochemical and thermo-chemical means. Researchers like (Okonkwo *et al.*, 2018) (Achebe et al., 2018), used the biological process for the production of methane, which is a major constituent of biogas. However, pyrolysis among the thermochemical process for converting biomass to energy has attracted more interest in the production of liquid fuel due to its advantages on the versatility of application like turbines, boilers, combustion engines, etc., transportation, and storage (Jahirul *et al.,* 2012). In addition, managing waste and solid biomass poses a difficult task that gives impetus to pyrolysis research. The production of liquid fuel (bio-oil), biochar and non-condensable gases through the pyrolysis of several biomass species (beech wood, bagasse, straw

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and stalk of rape plant, etc.) has been widely investigated previously by researchers. Pyrolysis process is of great interest due to its adaptability to a wide range of biomass, versatility of the technology, flexibility of operation and production of a variety of products. Therefore, the pyrolysis route has been investigated to enhance the properties and qualities of bio-fuels especially bio-oil with upmost commercial potential usage. Obtaining maximum production of bio-oil entails a good understanding of the type of reactors adopted in addition to its intrinsic (heat and mass) transport process. Garret pyrolysis, dynomotive process, waterloo flash pyrolysis process and the auger reactors among others happened to be the previously used reactors by earlier researchers (Panahi *et al.,* 2019). However, most recently, the fast pyrolysis process in fixed and fluidized-bed reactors has caught the interest of most researchers like Boateng *et al.* (2007).The fast pyrolysis process is of significant interest as it directly produces high yield of bio-oil to about 75% at a short vapour residence time of less than 2 seconds and a moderate temperature(Pattiya, 2018). The objective of this work is therefore to characterize biomass of elephant grass by determining the proximate, ultimate, and structural and some chemical properties and the high heating value in order to establish its suitability for bio-oil production.

MATERIALS AND METHODS

Source of biomass: The elephant grass (*Pennisetum purpureum*) was sourced from a farm site in Oghara town, Delta State, Nigeria with a geographical location (5^o 35' North, 5^o 51' East), in early January, 2022.

Biomass harvesting and handling: The maturity period of the elephant grass is approximately three months with a height of 1.0-1.3 meters. The matured sample was collected into sack bags in January, 2022.

Biomass, sizing, sieving and storage: The raw sample was transported to Delta State Polytechnic, Otefe-Oghara for pretreatment (cleaning, crushing and sieving). The crushed sample was sun-dried indoors to prevent contamination for five working days at ambient temperature. It was then pulverized in a ball mill to particle size (0.2 mm). Prior to analyses, the sample was stored at room temperature in Ziploc bag. After which, a portion of the sample was transported to Lighthouse Petroleum Engineering Company Laboratory, Warri, Delta State for characterization.

Characterization of the biomass (Elephant grass): After pretreatment of the sample in the laboratory of the Department of Science Laboratory Technology, Delta State Polytechnic, Otefe, Oghara, the sample was transported to Lighthouse Petroleum Engineering Company Laboratory, Warri for characterization. Characterization was done before experimentation to determine the physicochemical (proximate and ultimate) properties, structural compositions (cellulose, hemi-cellulose and lignin) and other properties of the biomass feedstock. The proximate and physical analysis was to evaluate the percentages of the fixed carbon, moisture content, ash, volatile matter (VM) and pH of the feedstock following the American Standard for Testing and Material (ASTM E872-82 and ASTM D1102-84.34) standard as described by Kumar *et al*. 2020. The chemical and ultimate analysis was carried out to determine the carbon, hydrogen, oxygen, nitrogen, sulphur and heating value following the American Standard for Testing and Material (ASTMD5373 and ASTM D4239-11) standard as described by Kristin *et al.,* 2020. Furthermore, components analysis was carried out to estimate the amount of cellulose, hemi-cellulose and lignin in the substrates using also the (ASTMD1104) standard as described by Kristin *et al.,* 2020.

Characterization by proximate analysis

Moisture content: The moisture content is an important property that affects the burning characteristics of biomass material. It has influence on the energy value of the fuel (Yang *et al*., 2005; Onochie *et al.,* 2017). The ASTM standard used to analyze the moisture content is ASTM E871-80. The moisture content of the samples was determined by drying at a temperature of 105°C in an oven and expressed in percentage of oven dry mass.

1. An empty moisture dish was correctly weighed and recorded.
2. 10g of the specimen was placed inside the dish and reweighed.
3. The dish with the specimen was placed in an oven of partial vacuum (less than 100mm Hg) at a temperature of 105°C for about 5 hours.
4. After which the dish was removed from the oven and gradually allowed to cool and weighed.
5. The process was repeated for an hour until we noticed a constant weight.

Calculation:

$$\%\text{Moisture} = \frac{\text{A} - \text{B}}{\text{A}}\, x\, 100$$

Where A = weight of sample before drying; B = weight of dry sample

*Ash content***:** The ASTM standard used to analyze the ash content is E1755- II. The muffle furnace was used for this analysis. The ash fraction contains all the mineral elements jumbled together.

1. 5g of the prepared sample was placed in a crucible.
2. After which it was placed in an oven at 100ºC for 24 hours.
3. It was then removed and transferred to a muffle furnace where the temperature was increased further to 550±5ºC.
4. This increased temperature was maintained for another 8 hours
5. Finally, the crucible was removed from the furnace and transferred to a desiccator to cool, weighed and recorded.

The ash content was expressed in percentage dry basis using the expression below

$$\%\text{Ash Content} = \frac{\text{X}}{\text{Z}}\ x\ 100$$

Where X = weight of ash; Z = weight of sample

Volatile matter (VM): The VM was obtained by difference as shown in the equation below

$$\%\ \text{VM} = \frac{\text{B} - \text{C}}{\text{B}}\ x\ 100$$

Where B = initial mass of the sample; C = final mass of the sample

Fixed carbon: The fixed carbon (FC) was obtained from the equation below

% FC = 100 – (% moisture content + % volatile matter + % ash content)

In obtaining the fixed carbon ASTM D3174 – 76 standard method was used. The crucible cover used in performing the volatile matter last analysis was removed after which the crucible heated over Bunsen burner to allow all the carbon to burn. The difference in weight of the residue from the previous weight is the fixed carbon.

Extractives content in the biomass: In obtaining the weight of extractives in biomass solvent extraction. 60 mL of Aceton added to 1 g biomass sample (elephant grass) (A). The temperature 90 °C controlled by using a hot plate for 2 h. 80 After 2 h, the sample dried in an oven at 105 – 110 °C until constant weight was obtained (B). By using the below equation, the weight of extractives was identified.

$$\%\ (A - B) = \%\ weight\ of\ extractives\ (g)$$

Where A, is initial biomass sample and B, % weight of the biomass after extraction.

Hemicellulose Content in the Biomass: In obtaining hemicellulose in biomass. 150 mL of Sodium Hydroxide (NaOH) solution (0.5 mol/L) was added to 1 g of the biomass sample (elephant grass) with extractives free (B). The temperature 80 °C controlled by using a hot plate for 3.5 h. After that, the sample was washed with deionized water until it is free from Na+. The Na+ was detected by using pH paper and the reading should be closed to 7. The sample was dried in an oven at 105 – 110 °C until constant weight was obtained (C). the amount of hemicellulose was identified by using the below equation.

$$\%\ Hemicellulose = \%\ B - \%\ C$$

Where, B = initial weight of content after extraction and C, = weight of content after heating

Lignin Content in the Biomass: In determining the content of lignin in biomass. 30 mL of 98 % Sulphuric Acid was added to 1g of the biomass sample (elephant grass) with extractives free (B). The sample left at ambient temperature for 24 h before boiled at temperature 100 °C controlled by using a hot plate for 1 h. The mixture was filtered and the solid residue was washed by using deionised water until sulfate ion undetectable. Detection of sulfate ion was done via titration process with 10 % of Barium Chloride solution. The sample dried 81 in an oven at 105 – 110 °C until constant weight was obtained (D). The final weight of residue is recorded as lignin content.

$$(D) = \%\ \text{wieght of Lignin (g)}$$

Cellulose Content in The Biomass: An assumption of the total lignocellulosic component inside the biomass. 1 g is referred to the total amount of biomass sample used in the experiment. By calculating the difference between the initial weight of the sample with the three others component weight calculated from the experimental process, the content of cellulose (E) will be identified.

$$\%(A - B) + (B - C) + D + E = 1g$$

RESULTS AND DISCUSSION

The results of the proximate analysis of the physical and chemical properties of the elephant grass shown in Table 1 contained the lowest moisture content (MC) at 0.85%, the lowest fixed carbon (FC) content at 14.22%, the highest volatile matter (VM) at 79.24%. High volatile matter content is an indication that biomass would ignite easily during pyrolysis process and this has influence in the thermal behavior of fuels.The lowest ash content at 5.86% between temperature of 350^0C and 400^0C, While VM between temperature of 500^0C to 600^0C, high moisture content entails logistic issues as it increases the tendency to decompose which results in energy loss and tend to reduce energy and cost balances (Javier and Jesus, 2019).

Table 1: Results of proximate analysis

Sample	Moisture Content (%)	Fixed Carbon Content (%)	Volatile Matter Content (%)	Ash Content (%)
Elephant Grass	0.85±0.3	14.22±0.4	79.24 ± 0.4	5.86±0.1

Table 2: Results of ultimate analysis of elephant grass

Sample	C (%)	H (%)	N (%)	O (%)	S (%)
Elephant Grass	45.44 ±0.1	5.59± 0.3	0.67± 0.2	40.95±0.1	0.35± 0.1

Table 3: Results of bio mass structural composition and heating value

Sample	Cellulose (%)	Hemicellulose (%)	Lignin (%)	Heating Value (MJ/Kg)
Elephant Grass	46.28	29.90	24.60	18.520

Table 2 showed results of ultimate analysis of elephant grass. Ultimate analysis mainly focused on carbon, hydrogen, nitrogen, oxygen and sulphur content. These elements are the major component of biomass that determine the fuel efficacy and the possible pollutant behavior (Singh *et. al.*, 2017). The results of ultimate analysis gave the chemical composition of the elephant grass as shown in Table 2.0 favoured the production of bio-oil, it influences the heating value and if the heating value is high it favour bio-oil production, the characteristics of the samples were consistent with values found in literature. (He *etal*., 2018)reported a gradual increase in C content from 300°C to 600°C.

$$Heating\ Value = HHV - (0.218\ x\ H$$

Where, H = weight % of Hydrogen obtained via ultimate analysis

The High Heating Value of the biomass (elephant grass) is 18.52 MJ/kg

Moisture content: Moisture content of biomass is the quantity of water existing within the biomass, expressed as a percentage of the total material's mass. Moisture content of biomass in natural conditions depends enormously on the type of biomass. Moisture content affect biomass decay rate, so dry biomass can be stored for a longer length of time whereas wet biomass will decay quickly (Boyle 2012). This is an important parameter when using biomass for energy since it has a marked effect on high moisture content which entails logistic issues as it increases the tendency to decompose which results in energy loss and tend to reduce energy and cost balances (Javier and Jesus, 2019). The results of moisture content of the biomass with elephant grass is 0.85±0.4. The heating value of a biomass feedstock represents the energy amount per unit mass or volume released on complete combustion. The heating value is seen in two different ways, the higher heating value (HHV) and low heating value (LHV). The HHV includes the latent heat contained in the water vapor that in practice cannot be used effectively, while the LHV excludes the heat of evaporation of the water formed from the hydrogen contained in the biomass feedstock and its moisture content. Thus, the LHV is the appropriate value to assess the energy available for subsequent use.

Fixed carbon: Fixed carbon is the solid combustible residue that remains after a sample is heated at 900ºCfor a period of 7 minutes and the volatile matter is expelled. The fixed carbon content result is 14.00 ±0.4for this study. The fixed carbon can influence the biological conversion of fuel due its high chemical energy level.

Volatile matter: The volatile matter of biomass is composed of condensable vapor and permanent gases (exclusive of water vapor) released when the biomass is heated to 925°C for few minutes. During this heating, the biomass decomposes into gases and solid matter is left out as char. The volatile matters of this study were found to be 79.24 ± 0.4. High volatile matter content is an indication that biomass would ignite easily during pyrolysis process and this has influence in the thermal behavior of fuels. It is worthy to state that the presence of volatile matter in biomass influences fuel reactivity, it has been observed that an increase in the volatile matter content of the biomass sample causes, as a general tendency, an increase in the peak temperature.

The peak temperature is the point on the burning profile at which the rate of weight loss due to combustion is maximum. The burning profile peak temperature is usually taken as a measure of the reactivity of the sample. The volatile matter content is also an important parameter for evaluating anaerobic digestion for biogas production (Cai *et. al.*, 2017).

Ash: Ash is generally considered to be the residue remaining after the material has been incinerated. It therefore has no energy value and, being made up of the inorganic elements in the biomass, is of no direct value in hydrolysis technologies. The ash content of this study for elephant grass is5.86 $\pm$0.1.The primary components of biomass ash are oxides of silica, aluminium, iron, calcium, magnesium, titanium, sodium and potassium. For example, knowing the exact composition of the ashes of a biomass aids in predicting both its tendency to form deposits in the boiler components and the composition of the char produced during pyrolysis and gasification processes, which in turn also influence the combustion rate. The percentage and composition vary according to the type of biomass (Gianluca *et. al.,* 2020).Structurally, the elephant grass sample comprised of cellulose, hemicelluloses, and lignin (Table 3.0). It could be showed that the value of lignin (24.60 %) was lower than that of cellulose (46.28%) and hemicelluloses (29.90%). The cellulose and hemi-cellulose content obtained in this work is greater than the cellulose (40.50%) and hemicelluloses (23.00%) contents of elephant grass reported by Wu *et. al.,* 2021. This is due to high carbon content (14.22$\pm$0.4) and volatile matter content (79.24 $\pm$ 0.4) of elephant grass which is the chemical energy of the biomass. This is an indication that the elephant grass is suitable for pyrolysis process because a relatively higher bio-oil yield would be expected. The HHV of 18.52 MJ/kg obtained here is due to the low ash and high carbon contents which are main sources of heat.

Conclusion: Proximate examination and structural investigations revealed that the elephant grass could be a potential biomass for bio-oil production.

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