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Research Article

CHARACTERIZATION OF INDIGENOUS BIOMASS

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Abstract- The characteristic proximate analysis of the paddy straw, sugarcane bagasse, sorghum bagasse and pearl millet straw was carried out in this paper. The properties of biomass like moisture content, ash content, volatile matter, bulk density, lignin, hemicelluloses, cellulose, sugars, and fixed carbon were determined as per standard ASTM methods. Cellulose, hemicelluloses, and lignin are the three primary components in biomass. For the analysis of cellulose, hemicelluloses lignin and sugars extractives were prepared with finely powdered biomass sample with particle size of 600µm and below with moisture below 10%. The other properties were analyzed using dried and powered biomass samples. The biomass characterization is a mandate for the selection of the most viable biomass for bioconversion processes and biofuels. The calorific value and energy densities of biomass were found in which sugarcane bagasse had maximum calorific value of 18.3MJ/kg.

Keywords- Proximate analysis, Cellulose, Hemicellulose, Lignin, Energy density, Reducing sugars

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Introduction

Biomass arrives through dissimilar resources and can be utilized for various energy purposes ranging from electricity production, producing fuels for transportation, thermal applications and almost every biomass based materials can be converted into fuel considering the current advancements in bioconversion system throughout the globe. Biomass characterization is mandatory since biomass does not flow well. Biomass particle are capable of knitting together, lacking originality or spontaneity. Biomass is vulnerable to self-heating and ignition. A research on the characterization of the properties of biomass is demanded for enabling a sustainable chain of biomass furnishing for energy production. Biomass is utilized for producing renewable sources of energy. Characterization of plant derived biomass is a challenge attributing to its heterogeneous structure and chemical composition. Substantial bottlenecks [1] clogging increased billow in biomass use for energy yield are highly dependent on the management of the supplies and transport required for biomass operation which include its costs, its complex logistics and operations in the entire chain of supplies, which are biomass harvesting and segregation from various or a single point of availability and pretreatment, storage, conveyance and coursing the ultimate energy supply source. The insufficiency [2] in sustained supply chains for biomass feedstock justifies that there is a denotative motive for more research for meliorated interpreting, developing functional and firm tools for qualifying physical and chemical properties, perils linked with handling of biomass materials. A wide installation of bio refineries could be established by this. The plant cell wall comprises of three primary portions, namely cellulose, hemicelluloses, and lignin. Cellulose is major constituent of bagasse and straws and hemicelluloses is the second major constituent, which accounts for 22 to 35 percent of dry mass of weight. Hemicelluloses comprises of acetyl and methyl substitute groups. Cellulose and most of the hemi cellulose make up the carbohydrates. Lignin is next to cellulose, which are more abundant polymeric organic substitutes in biomass. The mechanical strength of a plant is attributed to lignin. Lignin are complex polymers and phenyl propane linked together by ether bonds and carbon-carbon bonds. In hard woods 18 to 25 percent of lignin is present and in soft woods 25 to 35 percent of lignin is present.

Materials and Methods

Methods 1

Determination of moisture content in biomass

The moisture contents of the biomass samples were determined using the ASTM, E-871 [3] procedure. A powdered sample of biomass with particle size in the range of 800-850 µm, weighing 10 g was taken in a pan and placed inside a hot air oven at a temperature of 105±3°C. The sample was weighed at regular intervals and once the weight observed was constant, it was cooled to room temperature in desiccators. The percentage of total solids was determined using the formula,

$$\text{Total solids (\%)} = [(\text{weight}_{\text{of dry pan with dry solids}}) - (\text{weight}_{\text{of dry pan}})/ \text{weight}_{\text{of sample}}] \times 100$$

Methods 2

Determination of ash content in biomass

The ash content of the paddy straw biomass was determined following the [4] ASTM D 482 standard procedure. Biomass sample of 2g size from the oven dried sample was taken in crucible and placed in a muffle furnace at 575±25°C for a period of 4 h, then it was cooled to room temperature in a desiccators and its weight was recorded. Then again it was placed in aside a muffle furnace and dried to a constant weight. The percentage of ash in the sample was determined using the expression,

$$\text{Ash (\%)} = [(\text{weight}_{\text{of crucible with ash}}) - (\text{weight}_{\text{of crucible}})/\text{oven dry weight}_{\text{of sample}}] \times 100$$

Methods 3

Preparation of water and ethanol extractives from biomass

For the preparation of extractives, powdered biomass sample of 10g with particle size of 600 µm and below was taken. The sample was then mixed in 200 ml of distilled water. The solution was kept in a boiling water bath of 80°C for a period of 3 hours. Then it was filtered in a Whatman filter paper of 100 µm. The liquid filtrate was then stored in a sterile container at -4°C. The filtered biomass residue was dried at 105°C in a hot air oven until a constant dry weight was obtained [Fig-1] & Fig-2]. Similarly, for the preparation of ethanol extractives, ethanol of analytical grade was diluted to 5 % in the 200 ml of distilled water and then extractives were prepared. A biomass sample of 10 g with particle size 600 µm and below was mixed with 5% of ethanol in 200 ml of distilled water. The solution was kept in a boiling water bath of 80°C for a period of 3 hours. Then it was filtered in a What man filter paper of 100 µm. The liquid filtrate was then stored in a sterile container. The filtered biomass residue was dried at 105°C in a hot air oven until a constant dry weight was obtained.

Methods 4

Determination of hemicelluloses in biomass

For the estimation of hemicelluloses [5] present in the biomass, a 1g sample from dried extractive was taken and 10 ml of 0.5 mol of NaOH solution was added to it. Then the solution was kept in a boiling water bath for 3.5 h at 80°C. Then it was washed with distilled water until its pH was neutralized. The NaOH solution of 0.5 mol was prepared by dissolving 20g of NaOH in 1 litre of distilled water.

$$\text{Hemi cellulose} = \text{Weight}_{\text{Final}} - \text{Weight}_{\text{initial}}$$

Methods 5

Determination of acid soluble and acid insoluble lignin in biomass

ASTM D 1106-96 standard procedure [6] was adopted in the determination of lignin in biomass samples. Biomass sample of 0.3g was added to 3 ml of 72 % of H2SO4, the solution was mixed well and incubated for 60 minutes at 30°C. Then it was diluted with 84 ml of distilled water to 4% concentration. It was then autoclaved at 121°C for 1 h. The solution was then filtered and the liquid filtrate obtained was the acid soluble lignin and carbohydrates. The residue contains the acid insoluble lignin, and then it was dried until constant weight was noted after which it was placed in a muffle furnace in a silica crucible at 575±25°C. The ash obtained was weighed and the lignin was calculated using the following expressions, the oven dry weight (ODW) extractives free sample was calculated as,

$$\text{ODW} = [(\text{Weight}_{\text{airdry}}) \times (\%\ \text{Total solids})] / 100$$

The weight percent acid insoluble residue (AIR) was calculated using the following expression,

$$\%\text{AIR} = [(\text{Weight}_{(\text{Crucible+AIR})}) - \text{Weight}_{(\text{crucible})}] \times 100 / (\text{ODW sample})$$

Weight of the acid soluble lignin (AIL) on an extractives free basis was derived from the formula,

$$\%\text{AIL} = [(\text{Weight}_{(\text{Crucible+AIR})}) - \text{Weight}_{(\text{crucible})}] - [(\text{Weight}_{(\text{Crucible+ash})}) - \text{Weight}_{(\text{crucible})}] - [\text{Weight}_{\text{Protein}}] \times 100 / (\text{ODW sample})$$

Where, weight of protein was the amount of protein present in the acid insoluble residue. The amount of acid soluble lignin (ASL) on an extractive free basis,

$$\%\text{ASL} = \{[\text{UV}_{\text{abs}} \times \text{volume of filtrate} \times \text{dilution}] / [(\varepsilon) \times \text{ODW}_{(\text{sample})} \times \text{Path length}]\} \times 100$$

Where, UV_{abs} reading was the absorption reading displayed in the spectrometer, the average ultra violet visual absorbance for the sample at appropriate wave length of 560nm. ε was the absorptivity of the biomass at specific wavelength, was 25nm and the path length was the path length of ultra violet visual cell in cm, which was 1cm. The dilution was derived as,

$$\text{Dilution} = [(\text{Volume of sample}) - (\text{Volume of diluting sample})] / (\text{Volume of sample})$$

Total amount of lignin on an extractives free basis was calculated as,

$$\%\text{Lignin}_{(\text{Extractives free})} = \%\text{AIL} + \%\ \text{ASL}$$

The total lignin obtained or received from the analysis was estimated using the formula,

$$\%\text{Lignin}_{(\text{as received})} = \%\text{Lignin}_{(\text{Extractives free})} \times [(100 - \%\text{extractives}) / 100].$$

Methods 6

Estimation of cellulose in biomass

The cellulose [8] in the biomass was calculated by using the values obtained for their corresponding lignin, hemicelluloses and ash contents. The cellulose was calculated using the expression,

$$\text{Cellulose} = 100 - (\text{Lignin} + \text{Hemi cellulose} + \text{Ash})$$

Methods 7

Estimation of reducing sugars by DNS method

For the determination of reducing sugars 0.5ml of extractive solution was taken and added with 2.5ml of water, then 3ml of DNS reagent was added to it. The solution was kept in boiling water bath for 5 minutes, then 1ml of 40% Rochelle salt solution was added to it. The solution was cooled and the sample was added to it. The solution was cooled and the sample was analysed in a spectrometer at 510nm bandwidth. The DNS reagent was prepared with 2.5g of dinitro salicylic acid, 0.5g of phenol, 0.125g of sodium sulphate, and 2.5g of NaOH and was made up to 250 ml with distilled water. Rochelle salt of 40% was prepared with 40g of sodium potassium tartarate in 100ml of distilled water.

Methods 8

Bulk Density

Bulk density was determined by weighing the feedstock filled in a vessel of known volume and calculating the ratio of the weight of the feedstock to the volume of the vessel (ASTM C29 / C29M - 16). The average of five trials was reported as the volume of the bulk density of the feedstock.

$$\text{Bulk density } (\rho),\ \text{kg m}^{-3} = W/V$$

Methods 9

Volatile matter

The volatile matter was determined using muffle furnace (ASTM, E-872). To measure the volatile content, known content of dried sample was taken in a closed crucible and kept inside the muffle furnace at 650°C for six minutes and again at 750°C for another six minutes. The loss in weight of the sample was found out and the per cent of volatile matter was calculated as,

$$\text{Volatile matter, \%} = \frac{\text{Loss in weight of the sample, g}}{\text{Weight of moisture free sample, g}} \times 100$$

Methods 10

Fixed carbon

The fixed carbon (ASTM D3172 – 13) of samples was calculated by subtracting the sum of ash content (%) and volatile matter (%) from 100. The fixed carbon is the residue left after removing the volatile matter and the ash from the substance.

$$\text{Fixed carbon, \%} = 100 - (\text{volatile matter (\%)} + \text{ash content (\%)})$$

Methods 11

Calorific Value

The efficiency of a fuel can be understood by its calorific value. The calorific value of a solid and liquid fuel was determined using Bomb calorimeter [9]. The procedure involves using a bomb calorimeter for calculating the calorific value of coal, biomass samples. The biomass sample is first crushed in the pestle and mortar until it becomes a fine powder. As per the BS specification- B.S. 1016: Part 5:1967, the biomass sample was ground to pass through a 210µm sieve. The

crucible from the calorimeter was weighed. The powdered biomass sample was then pressed into a small pellet manually in a hand pellet press; 1g of this pellet sample was placed in the crucible. The crucible along with the biomass sample was weighed and the mass of the biomass sample was determined by subtracting the known mass of the crucible.

The top and bottom of the bomb are then separated and the crucible was set centrally at the base of the bomb in the support ring. A fuse is set across the support pillars and adjusted such that it touches the biomass sample in the crucible. During this process the bomb was kept up right so as not to upset its contents. The bomb is then charged with O2 to a pressure of 3 MN/m2 (30 atm), without displacing the original air content. After charging, the valves at the top of the bomb are carefully sealed. The charged bomb was then inserted into the water container vessel, making sure that the bottom of the bomb and the electrodes register correctly at the base of the vessel. A measured quantity of double distilled water (2 kg) was filled in the vessel covering the bomb. The temperature of water should be such that the final temperature reached has to be within a degree of standard reference temperature of 25°C. The cover and its stirrer are then assembled; thermometer was inserted through this cover. Stirring was commenced at a moderate pace and this stirring continued throughout experiment.

After a period of 5 minutes, the temperature was noted at intervals of every 1 minute. At the fifth minute the bomb was fired. The ammeter was observed to see if the bomb fuse has fired. This is detected by the ammeter swinging up to a maximum value and then quickly returning to zero showing that the fuse has broken. This marks the end of preliminary period and chief period commences. The temperature initiates to rise rapidly indicating successful firing of the bomb. The thermometer is read every minute with minimum accuracy of 0.01°C and was continued for the remainder of the experiment. When the maximum temperature had reached, it marked the end of chief period and the beginning of after period. The temperature fall during the after period was slow initially and when temperature decreased a steady rate was observed with the temperature recorded for 5 min. After this, the bomb was removed from the apparatus and the pressure was released to reduce the pressure inside the bomb to the atmospheric. The bomb was then opened and crucible was inspected to check a complete absence of soot deposition to ensure that the biomass sample had complete combustion. Then the calorific value of biomass sample was determined from the results obtained during the experiment. Regnault – Pfaundler cooling correction was done based on the expression furnished below

$$\text{Correction} = nv' + \frac{v'' - v'}{t'' - t'}\left[\sum_{1}^{c}(t) + \tfrac{1}{2}(t_0 + t_n) - nt'\right]$$

$$= nv + kS$$

Where, S is expression of the equation indicated inside the brackets, n is the number of minutes inside the chief period, v' is the rate of fall of temperature per minute within the preliminary period, if the temperature rises in this period, then v is negative, v" is the rate of fall of temperature / minute in the after period, t' and t" are the average temps during preliminary and after periods respectively.

$\sum_{1}^{n-1}(t)$ – sum of temperatures noted during the chief period.

½ (t0 + tn) – mean of firing temp. t0 and the first temp, tn after which the rate of change was constant.

$k = \frac{v'' - v'}{t'' - t'}$ cooling constant of calorimeter.

(If total water equivalent is not less than 2500 g and with adequate heat insulation, k should not exceed 0.0025).

$$\text{Cooling}_{\text{correction}} = nv + kS.$$

Uncorrected temperature rise was found by the expression, $t_n - t_0$

Corrected $_{\text{temperature rise}}$ is, $\text{Cooling}_{\text{correction}} + \text{uncorrected}_{\text{temperaturerise}}$

Since the water equivalent of the calorimeter is 2.6 kg/equiv,

Energy liberated by the biomass = 2.6 x Corrected $_{\text{temperature rise}}$ **x 4.1868** (Energy liberated in kJ for 2°Crise)

Calorific value (C.V.) of biomass was determined by using the following expression,

$$\text{C.V.} = [(2.6 \times 2.626 \times 4.1868) / (\text{sample (g)} \times 10^{-3})]$$

Methods 12

Energy density

Energy density was calculated using bulk density and calorific value of biomass feed stocks. The energy density was calculated based on the expression,

$$\text{Energy Density, MJ/m}^3 = \rho \times CV$$

Where, ρ is the bulk density of the biomass, Kg/m3 and CV is the calorific value of the biomass, MJ/kg.

Results

The proximate analysis of the four different biomass, namely paddy straw [Fig-3], sugarcane bagasse [Fig-5], sorghum bagasse [Fig-6], pearl millet straw [Fig-4], were carried out and the results are furnished in [Table-1]. This was carried out bring the moisture content below 10% with the intent of employing the same set of samples for all the analysis conducted.

Fig-1 Preparation of biomass extractives

The volatiles in sorghum bagasse and pearl millet straw were higher than paddy straw and sugarcane bagasse, 86.1 and 86% were recorded for the respective biomass. Similarly, paddy straw had the lowest volatile matter of 71.5% and sugarcane bagasse with 76.7% of slightly higher volatiles. Paddy straw's ash content was higher compared to the other biomass, its ash percent was 18.2, and this was nearly 15 to 16% higher than that of sorghum bagasse and pearl millet straw. Cane bagasse had ash of 10.3% which was much higher than sorghum and pearl millet biomass. The fixed carbon in paddy straw and sorghum bagasse was 10.3 and 10.2% respectively; bagasse had the highest fixed carbon of 13% followed by pearl millet straw with 11.5% fixed carbon. The bulk density of pearl millet straw, 132.4kg/m^3 was observed to be the highest, while sugarcane bagasse had the lowest bulk density of 93kg/m^3. Paddy straw and sorghum bagasse had bulk density of 128.2 and 103.4 kg/m^3 respectively.


Fig-2 Analysis of cellulose, hemi cellulose and lignin with extractives prepared

Fig-3 Paddy Straw

Fig-4 Pearl Millet

Fig-5 Sugarcane Bagasse

Fig-6 Sorghum Bagasse

The hemicelluloses in paddy straw were observed to be the lowest, 19% whereas pearl millet had the highest percentage of hemicelluloses of 29.6%. Sugarcane and sorghum bagasse had a lower percentage of 25.6 and 26.4% hemicelluloses than pearl millet straw, which was still higher than paddy straw. Sugarcane bagasse had the highest percent lignin, 19.8%, followed by paddy straw with 14%. Both sorghum bagasse and pearl millet biomass had higher lignin percentages of 17.5 and 18.8% than paddy straw. Cellulose of 42.9% was observed to be highest for paddy straw, closely followed by sugarcane bagasse with 40% cellulose, 35% for sorghum bagasse and 37.7% for pearl millet biomass. The reducing sugars analysed for sorghum bagasse, 36% was the maximum, 34.2% for paddy straw and 26% for sugarcane bagasse and 22.5% for pearl millet straw.

Table-1 *Results of proximate analysis of biomass*

Biomass Property	Paddy straw	Sugarcane bagasse	Sorghum bagasse	Pearl millet straw
Moisture (%)	6.5	9.0	5.8	7.0
Ash (%)	18.2	10.3	3.6	2.5
Hemicellulose (%)	19.0	25.6	26.4	29.6
Cellulose (%)	42.9	40.1	35.0	37.7
Lignin (%)	14.0	19.8	17.5	18.8
Reducing sugars (%)	34.2	26.0	36.0	22.5
Bulk Density, kg/m^3	128.2	93.0	103.8	132.4
Volatile matter,%	71.5	76.7	86.1	86.0
Fixed carbon, %	10.3	13.0	10.2	11.5
HHV, MJ/kg	17.5	18.3	16.8	16.4
Energy Density, MJ/m^3	2243.5	1701.9	1743.8	2171.4

The lowest sugar was observed for pearl millet biomass. The maximum calorific value was recorded for sugarcane bagasse, which was 18.3MJ/kg. Pearl millet straw had the lowest calorific value of 16.4MJ/kg closely preceded by sorghum straw which had 16.8MJ/kg calorific value. With a calorific value of 17.5MJ/kg paddy straw had a higher calorific value than pearl millet and sorghum biomass. However, the energy density of sugarcane bagasse was low at 1701MJ/m3, whereas paddy straw had the highest energy density of 2243MJ/m3 followed by pearl millet with an energy density of 2171MJ/m^3, which was higher than bagasse of sorghum and sugarcane. The lowest energy density sugarcane could be attributed to its lower bulk density on powdered form.

Based on the above proximate analysis of the biomass, the suitability of each biomass was studied with respect to the data displayed for their corresponding viability for bioconversion into alcoholic fuel, which includes storage, transport, handling, and bioconversion efficiencies for energy efficient bio-refining.

Conclusion

Biomass is a renewable reservoir, which can be a perennial supply of resources to meet the global energy demand for thermal, electrical and liquid transport fuels. The characterization of the biomass was carried out for their suitability for utilization as substrate for acetone butanol ethanol (A.B.E) fermentation. All the four biomass are locally available, paddy, pearl millet and sorghum are major food crops, of which the biomass comprising of stalks, stems, and leaves are dumped or openly burnt in fields post threshing of grains. Paddy straw or rice straw is a common cattle field. Sorghum bagasse and pearl millet straw are also used ad cattle feed. However sugarcane bagasse has a wider application like use as fuel in boilers in sugar industries, and for making paper in pulp industries. However, the other biomasses, which comparatively have a lesser significance for commercial product conversion, can readily be used as a renewable resource of lignocellulosic biomass in bio refineries for 2nd generation alcoholic fuel production.

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