



PHYSICOCHEMICAL COMPOSITION OF LIGNOCELLULOSE BIOMASS FROM *Gliricidia Sepium* AND *Cola Gigantea*

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ABSTRACT

Background: Majority of lignocelulosic biomass considered as waste materials create environment nuisance, because they were either allowed to decay or burnt-off, contributing to global warming. **Objective:** This work aims to evaluate the structural and compositional features of *Gliricidia sepium* stalk and *Cola gigantea* wood saw-dust for the purpose of industrial utilisation. **Methods:** The proximate and physicochemical constituents of *Gliricidia sepium* stalk and *Cola gigantea* wood saw-dust were investigated using standard method. The main functional group present in the biomass was study with the aid of Fourier transform infrared spectroscopy. **Results:** Results obtained indicated that the biomass contained varying cellulose, lignin and holocellulose contents. Proximate analysis showed that *Gliricidia sepium* stalk recorded higher value in all parameter examined except NaOH extractive, ash and silica content. It was equally higher in moisture content, true density and powder porosity while *Cola gigantea* had higher swelling capacity, compressibility power and Hausner index. The biomass examined had poor flowability and near true density value. **Conclusion:** The high cellulose content (43.79 % 41.44%), lignin content (30.75 % and 27.23 %) and holocellulose content (79.87% and 76.75 %) of *Gliricidia sepium* stalk and *Cola gigantea* respectively present the biomasses as good raw materials for biorefinery.

Keywords: Biomass, Cellulose, flowability, Fourier Transform Infrared Spectroscopy.

1. INTRODUCTION

Close to human existence everywhere is the vegetation referred to as lignocellulose materials, they are natural organic polymers that are non-toxic, renewable, abundance, biocompatible and biodegradable anionic polysaccharide materials on the earth [1, 2, 3, 4]. Lignocellulose waste is referred to as lignocelulosic biomass when in dry form, it accounts for about 70% of the world's biomass which has been estimated at 3×10^{11} tonnes [5]. Lignocelulosic waste major constituent are cellulose 35-50%, hemicellulose 20-35%, lignin 5-30%, and extractives 0-10% [6, 7].

The lignocelulosic biomass includes agricultural by-products (e.g. sawdust) from agricultural activities and agro-based processing which constitute environmental nuisance because they either burnt or allowed to decay, contributing to the global warming [8, 9, 5-10]. Lignocelulosic biomass is synthesis by photosynthesis through available atmospheric CO₂, water, and sunlight. It consists of three major polymers constituent –cellulose; a polymer of glucose monomer having β (1-4) glycosidic linkages, It makes up 35-50% of plant biomass, cellulose molecules arrange regularly, gather into bundles, and determine the framework of the cell wall; hemicellulose; an acetylated arabinoxylan with minor amounts of galactose and mannose 20-35%, Fibers are filled with hemicellulose and lignin; lignin; a complex phenolic polymer 5-30% that is strongly intermeshed and chemically bonded by non-covalent forces and by covalent cross-linkages. Lignocelulosic also consists of a minor component such as an extractives 0-10%, [10-6, 7]. The constituency of the extractives is cutin, suberin, wax, and other fatty substances. Lignocelulosic can be obtained from wood (soft and hard), non-wood (grass, agricultural residues), and municipal solid wastes (used paper, cardboard etc.). Turning lignocelulosic waste material into a useful product is attracting increased interests around the world, particularly for the production of environmentally friendly industrial materials either in its raw (biomass) form, isolation of its constituent (cellulose, Lignin, hemicellulose and extractives) to serve as precursors for other usable industrial materials through chemical modification [11, 12]. The biomass could also be used as a filtration medium for wastewater treatment: removal of heavy metal and organic pollutant [13, 14, 15]. Cellulose, being the most abundance of agricultural waste and its modified products find useful in any human endeavors. It is of great value in many industrial sectors; including food, textiles, paper, adhesives, paints, pharmaceuticals, cosmetics, petroleum and mineral processing [1-4]. Cellulose products such as cellulose acetate, cellulose triacetate, cellulose propionate, cellulose acetate propionate (CAP), nitrocellulose (cellulose nitrate), cellulose acetate butyrate (CAB) Microcrystalline cellulose (MCC), carboxymethyl cellulose (CMC), potassium cellulose succinate, Cellulose acetate trimellitate, cellulose acetate resin and sheet etc., have been produced and used for various industrial purposes.

Hemicellulose which is a sugar component of biomass is readily converted to yield fermentable sugars [16], biopolymers and biofuel [17], hydrogels [18], or thermoplastic xylan derivatives and chemical such as succinic, levulinic, glucaric, aspartic, sorbitol, xylitol and glycerol [17, 18].

Lignin is removed from wood pulp as liginosulfonates, for which many applications have been proposed. They are used as dispersants, humectants, emulsion stabilizer, and sequestrants (water treatment). Lignin and lignin derivatives biopolymers have several properties, such as high thermal stability, antioxidant, biodegradability, antimicrobial actions, adhesive properties, etc., and thus they can be extensively used in wide range of areas [19].

The quest for a cleaner environment, economic revitalization and societal wealth led to the choice of wood biomass such as *Gliricidia sepium stalk* and *Cola gigantea saw-mill dust*. A heap of saw-mill produce after logging is commonly turned to ashes by burning leaving the environment polluted as a result of the process. Also, *Gliricidia sepium stalk* turns out to be a nuisance as used as fencing material, yam stalk, and shade for coffee and cocoa plantations. In an attempt to utilized the biomass, this research aimed at address characterization of the material as bases of their industrial utilization.

2. MATERIAL AND METHODS

2.1 Material

The Lignocellulose bio-materials used in this study are *Gliricidia sepium stalk* and *Cola gigantea saw-mill dust*. These materials were sourced from Akure metropolis in Ondo State. All the samples were authenticated at the Department of Crop, Soil and Pest Management, Federal University of Technology, Akure, Ondo State, Nigeria. *G. sepium* stack materials were harvested at 20 cm above the ground level and chipped to 0.5 cm – 1 cm while *C. gigantea* was collected at a specialized Saw-mill (Daramola saw-mill, Ondo Road, Akure. All the samples were screened to remove dust, sand, dirt, and contaminations and dried at ambient temperature to have uniform moisture content before milled to a 0.25 mm particle size [20]. The milled samples were stored separately in labeled polyethylene bags for subsequent experiments at room temperature.

2.2 METHODOLOGY

2.2.1 Determination of moisture content : An empty crucible with its cover was dried in the oven at 105 °C until constant weight was obtained, cooled in a desiccator, and weighed. Exactly, 2 g plant sample was added to the pre-weighed crucible, dried for 3 h in an oven at 105 °C, cooled in a desiccator, and weighed. The cooling and weighing were repeating until constant weight was reached [21]. The experiment was done in triplicate and the moisture content calculated as follows:

$$\text{Moisture content (\%)} = \left(\frac{w_1 - w_2}{w_1} \right) \times 100 \quad (1)$$

Where:

w_1 = initial weight of the sample (g),

w_2 = the weight of the oven-dried sample (g).

2.2.2 Determination of ash content: An empty crucible was ignited in the muffle furnace at 750 ± 25 °C, cool in a desiccator, and weigh to the nearest 0.1 mg. exactly; 2 g oven-dried sample was weighed into the crucible. The crucible with its contents was then placed in the muffle furnace and ignited until all the carbon was eliminated. This was then removed from the furnace and transferred into the desiccator, cooled and weighed. The heating was repeated for 30 min periods until the weight after cooling was constant to within 0.2 mg. The ash content was calculated on the basis of the dry weight of the original sample after the sample was ignited at a 750 ± 25 °C (Technical Association of the Pulp and Paper Industry [22] for 6 h. The experiment was done in triplicate. The following formula was used to obtain the ash content:

$$\text{Ash content (\%)} = \left(\frac{A}{B} \right) \times 100 \quad (2)$$

Where:

A = weight of ash, g

B = weight of moisture-free test specimen (g)

2.2.3 Determination of silica content: To find the silica content from the ash, the [23] method was used. Ash obtained from the above method was digested with 5 mL of 6 M HCl and evaporated on a steam bath to dryness. After evaporation, another 5 mL of 6 M HCl was added and evaporated. The addition of another 5 mL of 6 M HCl to the residue was followed by heating, and then the solution was diluted with 20 mL of distilled water. Hot distilled water was used to wash the residue on ashless filter paper. After sufficient washing to remove chloride, the ashless filter paper along with

residue was placed in the crucible and ignited at 575 °C for 6 h, this was cooled and the silica weight was taken (C). The experiment was done in triplicate. The following formula was used to obtain:

$$\text{The silica content:} \quad \% = \frac{C}{D} \times 100 \quad (3)$$

Where:

C = weight of the silica (g)

D = weight of moisture-free, test sample (g)

2.2.4 Determination of water-soluble matter: The water solubility procedure removed a part of extraneous components, such as inorganic compounds, tannins, gums, sugars, starches, and coloring matter in the sample [24].

2.2.5 Determination of hot water solubility: About 2 g of oven dried plant specimen (w_3) was transferred to a 250 mL Erlenmeyer flask, 100 mL of hot distilled water was added. This was reflux for 3 h using water bath. After refluxing, the contents of the flask was transferred to a tarred filtering crucible which has been previously dried to a constant weight at 105 ± 3 °C, and wash with 200 mL of hot water, dried to constant weight (w_4) at 105 ± 3 °C. The experiment was done in triplicate [24]. The percentage of matter soluble in hot water, on oven dried basis was calculated as follows:

$$\text{Hot-water solubility (\%)} = \left(\frac{w_3 - w_4}{w_3} \right) \times 100 \quad (4)$$

Where:

w_3 = initial weight of moisture-free test sample (g),

w_4 = weight of oven-dry test extracted test sample (g).

2.2.6 Determination of cold-water solubility: Exactly 2 g oven-dried sample (w_5) was placed in a 500 mL beaker and covered with 300 mL of distilled water. This was kept at a temperature of about 25 °C for 48 h with frequent stirring. At the end of the experimental time, the sample was filtered, washed with cold distilled water and then dried to a constant weight (w_6) in oven at 105 °C. The experiment was done in triplicate [24]. The percentage of matter soluble in cold water on oven dry basis was calculated as follows:

$$\text{Cold-water solubility (\%)} = \left(\frac{w_5 - w_6}{w_5} \right) \times 100 \quad (5)$$

Where:

w_5 = weight of oven dried sample prior to treatment with cold water (g),

w_6 = weight of oven dried sample after extraction with cold water (g).

2.2.7 Determination of 1% sodium hydroxide solubility : Exactly 2 g of oven dried sample (w_7) was placed in 200 mL beaker and 100 mL of 1 % sodium hydroxide solution added. The beaker was covered and placed in boiling water bath and the content stirred at an interval of 10, 15, and 25 min for 1 h, after which the content of the beaker was filtered. The residue was washed with 100 mL of hot water , 50 mL of 10 % acetic acid and then thoroughly with hot water again. The residue was dried in the oven to a constant weight (w_8) at 105 °C [25]. The weight percentage of the dry matter soluble in one percent caustic soda solution was calculated as follows:

$$\text{1\% NaOH solubility (\%)} = \left(\frac{w_7 - w_8}{w_7} \right) \times 100 \quad (6)$$

Where:

w_7 = oven-dry weight of test specimen before extraction (g),

w_8 = oven-dry weight of test specimen after extraction (g).

2.2.8 Determination of ethanol- Toluene solubility: Soxhlet apparatus was used and a two-gram oven-dried sample was placed in an extraction thimble and refluxing solvent was 1: 2 solution of ethanol and toluene mixture. The extraction was conducted for 8 h. When the extraction was completed, all of the remaining solutions were transferred to the boiling flask which was heated on a heating mantle until the solution was evaporated. The flasks were oven-dried at 105 °C, cooled in a desiccator, and weighed repeatedly until a constant weight was obtained [26]. The experiment was done in triplicate. The following formula was used to obtain the alcohol-toluene solubility content of sample:

$$\text{Alcohol-toluene solubility matter} = \left(\frac{w_9 - w_{10}}{w_9} \right) \times 100 \quad (7)$$

Where:

w_9 = weight of oven-dry test specimen (g),

w_{10} = weight of oven-dry extracted residue (g).

2.2.9 Determination of lignin content: The determination of lignin content was carried out using a sample which had first been extracted with 1:2 (ethanol: toluene), in accordance with TAPPI standard method [27, 28].

2.2.10 Determination of acid-insoluble lignin ("Klason Lignin"): In this method one gram of the ethanol- toluene extracted sample was placed in 100 mL beaker, 15 ml of 72 % sulphuric acid was added gradually in small increments while stirring and macerating the sample with a glass rod. After the sample has dispersed, the beaker was covered with a watch glass and kept in a bath at about 20 °C for 2 h while stirring. At the end of the 2 h, the content was diluted to a total volume

of 575 ml in a 1-liter flask and then boiled for 4 h at a constant volume by frequent addition of hot water. It was then left overnight. The insoluble material (lignin) was filtered and the filtrate kept for the determination of acid-soluble lignin. The acid-insoluble lignin obtained was washed free of acid with hot water and dried to a constant weight at 105 °C in the oven [28]. The experiment was done in triplicate. The acid-insoluble lignin was calculated as follows:

$$\text{Klason lignin, (\%)} = \frac{Y \times 100}{W} \quad 8$$

Where:

Y = oven-dry weight of Klason lignin (g),

W = oven-dry weight of initial specimen (g).

2.2.11 Determination of acid-soluble lignin: The acid-soluble lignin was determined using a portion of the filtrate obtained from the determination of acid-insoluble lignin. The determination was from ultraviolet absorbance measured at 205 nm using 3 % tetra-oxo-sulfate acid (VI) (H₂SO₄) as a reference. The filtrate was first diluted with 3 % H₂SO₄ in order to obtain absorbance in the range of 0.2-0.7. The experiment was done in triplicate [27]. The lignin content B in the filtrate was calculated as follows:

$$B = \frac{AD}{110} \quad (9)$$

Where:

A = Absorbance

D = Dilution factor of the filtrate

110 = Absorptivity or extinction coefficient

The acid-soluble content in the test sample was determined as follows:

$$\text{Lignin (\%)} = \frac{110 \times B \times V}{100 \times W} \quad (10)$$

Where:

B= Lignin content in the filtrate in g /1000 ml

V= Total volume of the filtrate (575 ml)

W= Oven dry weight of the test sample (1g)

2.2.12 Determination of Holocellulose content: Accurately weighed 2 g oven-dried extractive-free sample was weighed (w_{12}) and placed into a 250 mL flask with a small watch glass cover. The sample was then treated with 150 mL of distilled water, 1 mL of cold glacial acetic acid, and 20 mL 15 % NaClO₂ and then placed in a water bath maintained at 75 °C. Every 1 h, for 5 h, 1 mL of cold glacial acetic acid and 20 mL 15 % NaClO₂ was added and stirred constantly. At the end of 5 h, the flask was placed in an ice water bath until the temperature of the flask reduced to 10 °C. The content of the flask was filtered into a coarse porosity fritted-glass crucible of known weight. The residue was washed free of ClO₂ with 200 mL ethanol and 25 mL acetone (the residue changed color from yellow to white). The residue was oven-dried at 105 °C, then cooled in a desiccator, and weighed until a constant weight was reached (W_2). The experiment was done in triplicate [29]. The following formula was used to determine the holocellulose content in sample:

$$\text{Holocellulose content (\%)} = \frac{w_{12}}{w_{11}} \times 100 \quad (11)$$

Where:

w_{11} = weight of oven-dried extractive-free sample (g).

w_{12} = Weight of holocellulose obtained (g).

2.2.13 Determination of cellulose content: Cellulose content was determined using Kurshner- Hoffer method [30]. Accurately weighed 1 g of oven dried sample was placed in a 250 mL round bottom flask fitted with a reflux condenser. 15 mL of 80 % acetic acid and 1.5 mL of concentrated nitric acid (HNO₃) was added. The mixture was boiled for exactly 20 min. About 20 mL of 95 % cold ethanol was added. The resultant mixture was cooled and filtered. The residue was then washed successively with hot benzene, hot alcohol, and diethyl ether. The residue was dried overnight to a constant weight and then ashed in a muffle furnace at about 500 °C for 5 h. The loss in weight upon ignition was taken as a measure of the cellulose content.

3. RESULT AND DISCUSSIONS

The result of proximate analysis of the lignocellulosic materials *Gliricidia sepium* and *Cola gigantea* was presented in Table 1. *Cola gigantea* biomass recorded higher cellulose value of 43.79 % against 41.44 % of *Gliricidia sepium*. The samples could be a promising raw material for pulp and paper manufacturer as a result of higher percentage yield, Nieschlag, (1960) stated that any plant materials with equal or higher than 34 % cellulose content would be a good pulp and paper industrial raw material [31]. While Samariha and Khakifirooz (2011), Iqbal, et al., (2011) and Kumar, (2009) recommended

lignocellulosic feedstocks contain $\geq 40\%$ cellulose for pulp production [32, 33, 34]. Values of 43.79 % and 41.44 % in this research works was incomparable with those of other authors, Iqbal, et al., (2013) obtained 40-45 % cellulose from hardwood [35]; 45-50 % for softwood [36]; 44 % found in Carmagnola Hemp according to Gandolfi et al., (2013) [37]. The different in cellulose content of lignocellulose biomass might be as a result of type, species, ages, and source of the biomass as well as the process used to isolate the cellulose from the lignocellulosic materials [38, 39].

Table 1: Proximate Composition of *Gliricidia sepium* biomass and *Cola gigantea* biomass.

Parameter (%)	<i>Cola gigantea</i>	<i>Gliricidia sepium</i>
Cellulose content	43.79 \pm 0.28	41.44 \pm 0.51
Lignin content	30.75 \pm 0.72	27.23 \pm 0.17
Acid soluble lignin	29.91 \pm 0.72	26.51 \pm 0.17
Acid insoluble lignin	0.84 \pm 0.04	0.69 \pm 0.07
Holocellulose content	79.87 \pm 0.20	76.75 \pm 0.71
Toluene- alcohol extractive content	04.12 \pm 0.30	02.21 \pm 0.15
NaOH extractive content	20.73 \pm 0.29	30.95 \pm 0.11
Cld water extractive content	12.89 \pm 0.14	12.37 \pm 0.03
Hot water extractive content	14.44 \pm 0.11	11.07 \pm 0.03
Ash content	12.62 \pm 0.33	15.39 \pm 0.11
Silica content	04.14 \pm 0.33	09.35 \pm 0.10

Mean values listed of three replicates with \pm standard deviation values

The Lignin content of lignocellulose biomass for *G. sepium* recorded 26.51% and *C. gigantea* 29.91 % while the acid soluble-lignin was 0.69 % (*G. sepium*) and 0.84 % (*C. gigantea*) records The lignin values obtained in this study were higher than those reported for *P. elongata* 20.5 % [40], Eucalyptus 23.30% [41], bamboo 24.5% [42], coniferous 25-32 % and bagasse 23-32 % [43]. Notwithstanding, the lignin contents of the two samples under examination are within the satisfactory level ($< 30\%$) [44]. The significance of their lignin content is that the plant biomass would require higher temperature and more bleaching chemicals to achieve the needed cellulose grade and good brightness [45]. The high level of lignin content reported here show that *Gliricidia sepium* and *Cola gigantea* may be a good source of lignin that could be utilized for industrial purposes [46].

The result presented in Table 2 revealed that holocellulose content was 79.87% *Cola gigantea* and 76.75% *Gliricidia sepium*. The result shows that the two wood samples could compete favourably with another plant as a good source of total carbohydrate. Other authors have holocellulose of 75.74 % (*P. elongata*), 70.50% (bamboo), 77.6% (*T. diversifolia* stalk), 75.20 % for pineapple leave and 67.6 % for tobacco stalk [47, 48, 49, 45, 40], The holocellulose content obtained from this study is an indication that the plant fibers would be able to withstand processing condition and good physical properties if used for paper since high holocellulose content correlates with a higher pulp yield is desirable for pulp and paper and other allied industries [50].

Lignocellulose biomass ethanol-toluene solubility

The result of the ethanol-toluene solubility showed that *C. gigantea* contained 4.12 % extractive while *G. sepium* has 2.21 %. The ethanol-toluene solubility is a measure of volatile matter (extractive), mostly, an organic matter from biomass as it forms condensable vapor (exclusive of water vapor) released from biomass when it is heated. This could be waxes, fats, resins, oils, tannins, gums and other soluble materials. The factors affecting the ethanol-toluene content of plant material are; seasoning, drying method, heating conditions (the heating rate, temperature, and residence time) and sampling method [27] and this might play a major role in the different value recorded for the extractive content. The *Gliricidia sepium* was obtained at the onset of dry season and ground with the bark; this could cause its high extractive content. The obtained result is comparable to 3.76 % for *P. elongata* [40], 9.5 % for corn stalk [48], while 8.2 % and 12.8% are reported for mid-rib and pseudostem of *M. paradisiaca*, 3.0 % and 2.3 % for stalk of *M. paradisiaca* [51].

Lignocellulose biomass Alkali and water solubility

The alkali-soluble material results revealed that 20.73 % was obtained for *C. gigantea* and 30.95 % *G. sepium*, cold water solubility was 12.89 % *C. gigantea* and 12.37 % *G. sepium*, while hot water solubility was 14.44 % and 11.07% for *C. gigantea* and *G. sepium* respectively. *C. gigantea* (20.73 %) 1 % NaOH obtained was less than 29.5 % recommended as suitability for fiber materials meant for pulp and paper production. While *Gliricidia sepium* recorded higher value, this is an indication that *Gliricidia sepium* biomass required more drying period and better storage condition to prevent fungus or microbial decay or degradations by heat and light after harvesting [52]. On the other hand, the 1 % NaOH extractives values obtained in this research are within the range of values reported for most biomass materials like *H. Cannabis*

(25.8 %), *chenopodium album* (30.00 %) [53], lemon grass 30.64 % (Harjeet and Dharm, 2013), sunflower 50.00 % [54] cotton stalks 39.60 % [44] and tobacco stalk 42.00 % ; *eucalyptus grandis* 17.9 % [55] and *Pinus nigra arnold spp* 13.0 % [44]. The 1 % NaOH values could be an indication that *C. gigantea* and *Gliricidia sepium* could be a good candidate for ethanol production because they contained a reasonable amount of low-molecular-weight carbohydrates. The cold water solubility recorded higher value 12.89 % for *C. gigantea* and *Gliricidia sepium* 12, 37 %, while the hot water soluble of *C. gigantea* was 14.44% and *Gliricidia sepium* has 11.07 %. The higher hot was solubility of *C. gigantea* could be attributed to leaching of some of its soluble sugar or carbohydrate component in the hot water [56].

Physicochemical properties of lignocellulose biomass

The ash content (Table 1) is an inorganic residue remaining after ignition at a high temperature not less than 550 °C and these represent the inorganic substances such as silicates, sulfates, carbonates, or metal ions [57]. The biomass ash content was 12.62 % and 15.39 %. The biomass silica content of *C gigantea* was 4.14 and 9.35 % was for *Gliricidia sepium*. The biomass ash content is lower than 16.6 % reported for rice straw biomass [58] but greater than 4.5 % - 9.0 % for wheat, 6.0 % to 8.0 % for Esparto [59], 6.9 %, 3.44 % and 11.8 % reported for mid-rib, pseudostem and stalk of *M. paradisiacal* [51].

The higher ash content found in *C gigantea* and *G. sepium* biomass with it corresponding silica content may be a result of transport system in the plant and photosynthetic mechanism employed which might allow easy transportation of silicic acid to the plant fiber, its folding and encapsulating nature as well as method of harvesting [60-10], soil composition, age, types of plants, silica content [61], growth environment, present of several joints and ears. The present of internodes in *C. gigantea* and *G. sepium* could also be responsible for the higher ash and silica content [10].

Density and flowability indices of lignocellulose biomass and cellulose

An important characteristic of biomass materials is their bulk density or volume; both as produced and as-subsequently processed dictate the transportation pace and store space (Table 2). The Hausner ratio and Carr's index are both measures of the flow properties of powders [62, 63]. The bulk density of *Cola gigantea* was 0.15 gdm⁻³ and 0.29 gdm⁻³ was for *Gliricidia sepium*, The biomass bulk density values were within the range reported for Sawdust (0.12 g/cm³), hardwood (0.23 g/cm³) and softwood (0.18-0.19 g/cm³) [64]. The *Gliricidia sepium* with lower moisture content (5.97 %) had about double of *Cola gigantea* bulk density in spite of the duo with same particle size, this might be as a result of differences in their biomass particle shape, particle density, surface characteristics and the degree of fill tightness [65]. The pore spaces in bulk samples can be described by the porosity. *Cola gigantea powder* porosity was 71.19 % and 41.26 % was for *Gliricidia sepium*. These show that Porosity or void fraction of the biomass account for the sharp variations that occur between the two biomass densities. Generally, the bulk density of lignocellulosic biomass is relatively low as observed (0.15 -0.29 g cm⁻³) compared with a coal (about 0.9 g cm⁻³) while hardwood biomasses ranges are 0.28– 0.48 g cm⁻³. The *Cola gigantea* biomass tap density recorded was 0.24 g/cm³ and *Gliricidia sepium* had 0.21 g/cm³. Tap density gives information on consolidation of a powder and the extent of its flowability; the high the value the more resistant to powder flow. *Cola gigantea* biomass true density was 0.52 g/cm³ and *Gliricidia sepium* was 0.51 g/cm³. The biomass could be classified as partially packed fill after tapping because the bulk density of for loose fill material ranged from 0.050 to 0.264 g cm⁻³ and from 0.068 to 0.325 g cm⁻³ for packed fill after tapping [66].

Table 2: Physicochemical Composition of *Gliricidia sepium* biomass and *Cola gigantea* biomass.

Index	Percentage report for dry matter	
Moisture content	8.35 ± 0.01	5.97 ± 0.14
Moisture sorption capacity	10.84 ± 0.15	8.21 ± 0.12
Swelling capacity	28.33 ± 2.89	63.33 ± 5.78
Bulk density (Bd) (g/cm ³)	0.15 ± 0.00	0.29 ± 0.01
Tap density (Td) g/cm ³	0.21 ± 0.01	0.42 ± 0.00
Hausner indx	1.40 ± 0.01	1.45 ± 0.01
Carr's index %	28.57 ± 1.16	30.95 ± 1.16
True density	0.52 ± 0.01	0.51 ± 0.01
Powder porosity	71.19 ± 0.02	41.26 ± 0.04

Mean values listed of three replicates with ± standard deviation values

The biomass flow indexes (Table 2) which play an important role in the transportation, storage, handling and biomass conversion [67, 68, 69] showed that *Cola gigantea* has Hausner index of 1.40 and *Gliricidia sepium* has 1.45, Carr's index was 28.57 and 30.95 for *Cola gigantea* and *Gliricidia sepium*, Hausner index greater than 1.25 and Carr index greater than

25% are pointer to poor flowability, whereas Carrs' index and below 15 signifies good flowability [70, 71]. The poor flowability recorded might be as a result of the biomass moisture sorption [72, 73] and intraparticle forces exist as a result of particles below 1 mm in size [74].

Swelling capacity and moisture sorption content

The study on the moisture sorption of biomass is important for biomass harvest, handling, transport, and storage. Therefore, proper drying and storage operations are required to preserve the quality of biomass feedstocks. The *Gliricidia sepium* gave a higher swelling capacity of 63.33% while *Cola gigantea* gave 28.33 %. The moisture sorption capacities recorded are 10.84 % and 8.21 % for *Cola gigantea* and *Gliricidia sepium*. The moisture sorption recorded in this study suggested that the biomass could not be easily attacked by microbe and could not be easily degraded, because moisture sorption of 5 to 11 % have been suggested as good for storage of biomass without degradation [75]. The swelling capacity and moisture sorption capacity could have been influenced by their moisture content, *Gliricidia sepium* with lower moisture content has the higher swelling capacity but lower moisture sorption capacity which could be an indication for its moisture attractiveness thus seems to absorb moisture faster than *Cola gigantea*. It also depends on the humidity of the environment [76].

Fourier Transform Infra-Red analysis of biomass

The FT-IR spectra of *Gliricidia sepium* and *Cola gigantea* are presented in Fig 1 and Fig 2. Absorption at 3347 cm^{-1} and 3358 cm^{-1} for *Cola gigantea* and *Gliricidia sepium* biomass could be attributed to O-H stretching vibration [77, 78]. The absorbance at 2917 and 2919 cm^{-1} was attributed to $-\text{C}-\text{H}$ symmetric and asymmetric stretching of CH_2 and CH_3 groups for *Cola gigantea* and *Gliricidia sepium* biomass, [78], The biomass CH_2 bend absorption peak was recorded at 1424 cm^{-1} and at 1421 cm^{-1} against *Cola gigantea* and *Gliricidia sepium* biomass respectively. The presence of peaks between $1620\text{--}1595$ shows the present of $\text{C}=\text{C}$ stretching of the aromatic ring (lignin), while the presence of the absorption peak at around 1420 cm^{-1} assigned to aromatic $\text{C}-\text{O}-\text{CH}_3$ stretching mode in the plane symmetrical stretching vibration of the aromatic ring of lignin [79].

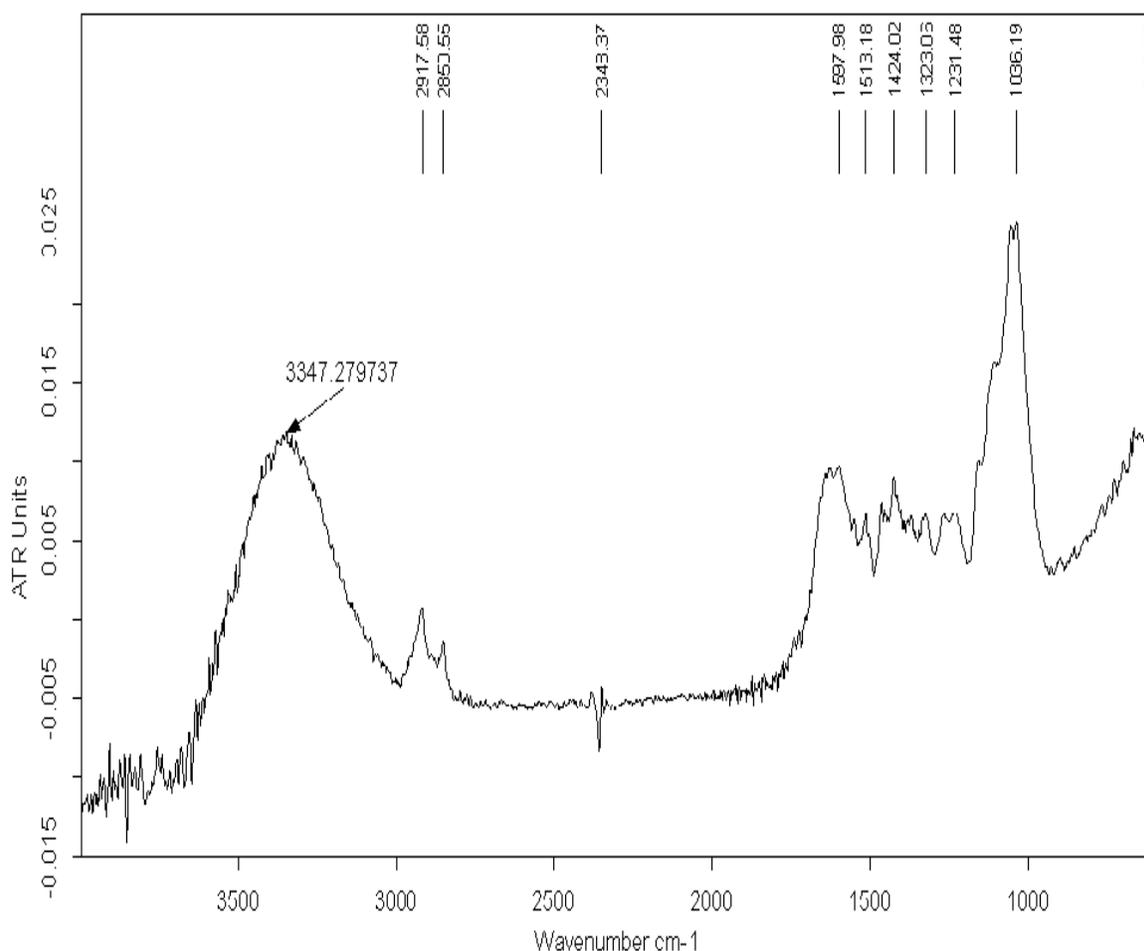


Figure 1: The figure presents the Fourier transform infrared spectra of *C. gigantea* biomass showing functional group peak.

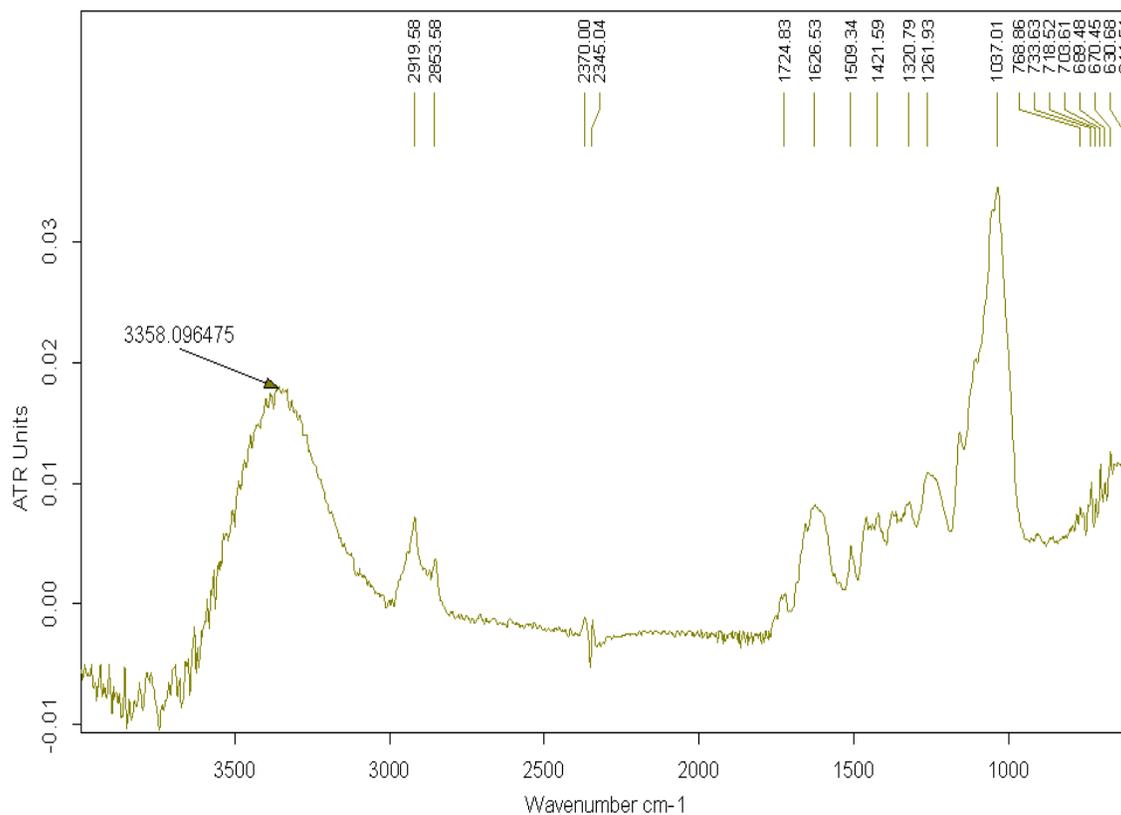


Figure 2: The figure presents the Fourier transform infrared spectra of *Gliricidia sepium* biomass showing functional group peak.

3. CONCLUSION

Lignocellulosic biomass from *Gliricidia sepium* and *Cola gigantea* offers many possibilities as feedstock for the energy sector through its high holocellulose content. Also, Chemical industry stand to benefit due to its chemical composition; the high cellulose content in *Gliricidia sepium* and *Cola gigantea* are suitable for pulp and paper industries, Carboxymethyl cellulose and microcrystalline cellulose syntheses, this are useful in food, drug and cosmetic industries. The Lignin content of *Gliricidia sepium* and *Cola gigantea* could be use in wood adhesives and aromatic chemicals industries.

4. REFERENCES

- Adinugraha, M.P., Marseno, D.W., and Haryadi. Synthesis and characterization of sodium carboxymethyl cellulose from Cavendish banana pseudo stem (*Musa cavendishii* LAMBERT). *Carbohydrate Polymers*. 2005; 62:164-169.
- Kamel. S., Ali1 N., Jahangir1 K., Shah1 S. M., and El-Gendy A. A. Pharmaceutical significance of cellulose: A review; *eXPRESS Polymer Letters*. 2008; 2(11):758-778.
- Bahramparvar, M., and Mazaheri T.M. Application ad functions of stabilizers in ice cream. *Food Reviews International*. 2011; 27(4): 389-407.
- Stanford John. Food processing technologies for reduction of fat in products" *Food and Health Innovation Serogies vice*. Scotland Food & Drink. 2012, Archived from 2014-10-23.
- Cui Y., Chang X., Zhu X., Luo H., Hu Z., Zou X. and He Q. Chemically modified silica gel with paradimethylaminobenzaldehyde for selective solid phase extraction and preconcentration of Cr(III), Cu(II), Ni(II), Pb(II) and Zn(II) by ICP-OES. *Microchemical Journal*, 2007, 87, p. 20-26.
- da Costa Sousa L., Chundawat S., Balan V. and Dale B. Cradle-to-grave assessment of existing lignocellulose pretreatment technologies. *Current Opinion in Biotechnology*. 2009; 20:339-347.
- Tian, X, Zhen F., Dan J. and Xi-yan S. Pretreatment of microcrystalline cellulose in organic electrolyte solutions for enzymatic hydrolysis. *Biotechnology for Biofuels*. 2011; 4 (53): 1-14
- Heslop-Harrison J.S. and Schwarzach T. Domestication, genomics and the future of the banana. *Annals of Botany*. 2007, 100, 1073-1084.
- Ahmed, O.H., Husni, M.H., Anuar, A.R. and Hanafi, M.M. Towards sustainable use of potassium in pineapple waste. *The Scientific World Journal*, 2004, (4):1007-1013.
- Yang S.H. Plant fiber chemistry. Beijing: China Light Industry Press, 2008.
- Pandey, A., Soccol, C.R., Nigam, P. and Soccol, V.T. Biotechnological potential of agroindustrial residues. I: Sugarcane bagasse, *Bioresource Technology*. 2000; 74(1): 69-80.
- Richardson S. and Gorton, L. Characterisation of the substituent distribution in starch and cellulose derivatives, *Analytica Chimica Acta*. 2003; 497(1-2): 27-65.
- Willmott N.J., Guthrie J.T. and Nelson G. The biotechnology approach to colour removal from textile effluent. *JSDC*; 1998, 114(February):38-41.
- Nasim A. K., Shaliza I. and Piarapakaran S. Elimination of Heavy Metals from Wastewater Using Agricultural Wastes as Adsorbents, *Malaysian Journal of Science*. 2004; 23:43-51.

15. Goletic S., and Avdic N.: The Cellulose and Paper Industry Wastewater Treatment. *Bulletin of the Chemists and Technologists of Bosnia and Herzegovina*. 2012; 39:45-50.
16. Lee S.H., Doherty V.T., Linhardt J.R. and Dordick S.J. Ionic liquid-mediated selective extraction of lignin from wood leading to enhanced enzymatic cellulose hydrolysis. 2009 102(5):1368-76. doi: 10.1002.
17. Gabrieli, I., Gatenholm, P., Glasser, W.G., Jain, R.K. and Kenne, L. Separation, characterization and hydrogelformation of hemicellulose from aspen wood. *Carbohydr. Polym.* 2000; 43:367–374. 6.
18. Sun, Y. and Cheng, J. Hydrolysis of Lignocellulosic Materials for Ethanol Production A Review. *Bioresource Technology*. 2002; 83: 1-11.
19. Anina N., Shazia T., Khalid M., Mohammad Z. and Aqdas N. Lignin-derivatives based polymers, blends and composites: A review. *International Journal of Biological Macromolecules*. 2016; 93, 296-313.
20. TAPPI- T 264 om-88: Technical Association of the Pulp and Paper Industry: Preparation method of wood for chemical analysis.
21. Technical Association of the Pulp and Paper Industry (TAPPI 550om-03 (2008): Standard for Moisture in Pulp, Paper and Paperboard.
22. Technical Association of the Pulp and Paper Industry (TAPPI- T 211om-93): Standard method for ash in wood, pulp, paper and paperboard.
23. Technical Association of the Pulp and Paper Industry (TAPPI 244om-93 (1999).). Standard for Silica in Wood, Pulp, Paper and Paperboard
24. Technical Association of the Pulp and Paper Industry (TAPPI- T 207 om-88): Water solubility of wood and pulp.
25. Technical Association of the Pulp and Paper Industry (TAPPI- T212om-98): Standard method for one percent caustic soda solubility of wood.
26. Technical Association of the Pulp and Paper Industry (TAPPI- T 204om-97): Standard method for alcohol –benzene solubility of wood.
27. Technical Association of the Pulp and Paper Industry (TAPPI- T UM250): Standard method for acid - soluble lignin in wood and pulp.
28. Technical Association of the Pulp and Paper Industry (TAPPI- T 222om-98): Standard Method for acid-insoluble lignin in wood and pulp
29. American Society for Testing and Materials ASTM D 1104-56. Method of test for holocellulose in wood, 1978, USA.
30. Tehnologija hrane : Pravilnik o metodama analiza kaka. 1987 "Sl. List SFRJ", br. 41/87.
31. Nieschlag H.J., Nelson G.H. and Wolff I.A. A search for new fibre crops. *Technical Association of the Pulp and Paper Industry Journal*. 1960; 44(7): 515-516.
32. Samariha A. and Khakifirooz A. Application of NSSC pulping to sugarcane bagasse. *BioResources*. 2011; 6(3):3313-3323.
33. Iqbal, H.M.N., Ahmed, I., Zia, M.A. and Irfan, M. Purification and characterization of the kinetic parameters of cellulase produced from wheat straw by *Trichoderma viride* under SSF and its detergent compatibility. *Advances in Bioscience and Biotechnology*. 2011; 2: 149-156.
34. Kumar, P., Barrett, D.M., Delwiche, M.J. and Stroeve, P. Methods for pretreatment of lignocellulosic biomass for efficient hydrolysis and biofuel production. *Industrial & Engineering Chemistry Research*. 2009; 48:3713-3729.
35. Iqbal, H.M.N., Kyazze, G. and Keshavarz, T. Advances in valorization of lignocellulosic materials by biotechnology: An overview. *BioResources*. 2013; 8: 3157-3176.
36. Malherbe S., and Cloete T. E. Lignocellulose biodegradation: fundamentals and applications: A review. *Environ. Sci. Biotechnol.* 2003; 1:105-114.
37. Gandolfi Stefano, Gianluca Ottolina, Sergio Riva, Guseppe Pedrocchi fantoni and Ilabahan Patel. Complete chemical analysis of carnagole hemp hurds and structural features of its components. *BoiResources*. 2013; 8(2): 2641-2656.
38. Carere CR, Sparling R, Cicek N, Levin DB. Third generation biofuels via direct cellulose fermentation. *International journal Molecula Science*. 2008; 9:1342–1360.
39. Chandra R.P., Bura R., Mabee W.E., Berlin A., Pan X., and Saddler J.N. Substrate pretreatment: the key to effective enzymatic hydrolysis of lignocellulosics? *Adv Biochem Eng Biotechnology*. 2007; 108:67–93.
40. Ates S., Ni Y., Akgul M., and Toziouglu A. Characterization and Evaluation of *Poulownia elongatoas* a raw material for paper production, *African Journal of Biotechnology*. 2008; 7(22):4153-4158.
41. Ayata U. A research on *Eucalyptus maldulensis* and *Eucalyptus grandis*) wood properties and their use in the paper industry, Kahramanmaras Sutcu Imam University, Institute of natural and Applied Sciences, MSc dissertation, Kahramanmaras, Turkey. 2008.
42. Deniz I. and Ates S. Determination of optimum kraft pulping conditions using bamboo (*Pyllcachys bambusoides*) 2nd. National Black Sea Forestry Congress Proceeding, Artvin, Turkey, 2002; 1072-1084.
43. Rowell R.M. Paper and composites from agro-based resources, CRC, press, 1997, 7-427
44. Kamoga, O.L.M., Kirabira, J.B. and Byaruhanga, J.K. Ververis Characterisation of Ugandan selected grasses and tree leaves for pulp extraction for paper industry. *International Journal of Scientific and Technology Research*. 2013; 2(9): 146-154
45. Ververis C, Georghiou K. Christodoulakis N. Santas P., Santas R. : Fiber dimensions, lignin and cellulose content of various plant materials and their suitability for paper production *Industrial Crops and Products*. 2004; 19: 245–254.
46. Andersson-Gunneras S, Mellerowicz EJ, Love J, Segerman B, Ohmiya Y, Coutinho PM, Nilsson P, Henrissat B, Moritz T, Sundberg B. : Biosynthesis of cellulose-enriched tension wood in *Populus*: a global analysis of transcripts and metabolites identifies biochemical and developmental regulators in secondary wall biosynthesis. *Plant Journal*. 2006; 45: 144–165.
47. Kirci H., Usta M. and Ero-Lu H. Soda- Air pulping of Phragmites reed, the 5th meeting of Pulp and Paper industry of Balkan Countries, 1998 October 7-9. Constanta, Mamaia, Romania.
48. Usta M., Kirci H. and Eroglu H. Soda-Oxygen pulping of corn (*Zea mays idurata* Stuar). In: Tappi pulping conference: Toronto.onitario, Canada, proceeding booklet. 1990; 1: 307-312.
49. Mokhter M., Hassan A., Rahmat A and Abd Samat S. Mokhter M., Hassan A., Rahmat A and Abd Samat S. Characterization and treatments of Pineapple leaf fibre thermoplastic composite for construction application. Report. Universiti Teknologi Malaysia. 2005; Unpublished.
50. Crowell E.P. and Burnett B.B.: Determination of the carbohydrate composition of wood pulps by chromatography of the alditol acetates. *Analytical Chemistry Journal*. 1967; 39, 121.
51. Ogunbile B. O., Omotoso M. A. and Onilude M. A. Comparative soda pulps from mid- rib, pseudostem and stalk of *Musa paradisiaca*. *Journal of biological science*. 2006; 6(6), 1047-1052. DOI: 10.3923
52. Agarwal N. K., Khanna N. K., Chechi S. K., Patel K. I., Pawar S. L. and Patil, R. G. Evaluation of Banana pseudostem based fibre as pulp and papermaking. *Indian Pulp and Paper, Technical Association Journal*. 2011; 23(3):119-124.
53. Dutt, D., Arvind, K.S., Swarnima, A. and Archana, G. Characterisation of dogs tooth grass and its delignification by soda pulping process. *Journal of Science and Technology*. 2012; 1(8):434- 447.
54. Lopez F., Eugenio M. E., Diaz M. J., Nacimiento J. A., Garcia M. M. and Jimenez. Soda pulping of sunflower stalks. Influence of process variables on the resulting pulp; *Journal of Industrial and Engineering Chemistry*. 2005; 11(3): 387 – 394.
55. Shakhes, J., Morteza, A.B., Zeinaly, F., Saraian, A. and Saghafi, T. Tobacco fibre for paper. *BioResources*. 2011; (6)4:4481-4493.
56. Justin B. Sluiter, Raymond O. Ruiz, Christopher J. Scarlata, Amie D. Sluiter, and David W. Templeton : Compositional Analysis of Lignocellulosic Feedstocks. 1. Review and Description of Methods. *J Agric Food Chem*. 2010; Aug 25; 58(16):9043–9053.
57. Rydholm S. A. Pulping process, 1st ed., Interscience Publishers, New York, 1965.
58. Tutus A., Deniz, I. and Eroglu H. Rice straw pulping with oxide added soda-oxygenanthraquinone. *Pakistan Journal of Biological Sciences*. 2004; 7(8): 1350-1354.
59. Finell, M.: The use of reed canary-grass (*Phalaris arundinacea*) as a short fibre raw material for the pulp and paper industry. Doctoral Thesis Swedish University of Agricultural Sciences Umea. 2003.

60. Rafi M. M. and Epstein E. Silicon absorption by wheat (*Triticum arstivum* L). *plant and soil*. 1999; 211(2): 223-230.
61. Brunow G. Lundquist K. and Gellersted G. Lignin, In Sjostrom and R Allen (edu), Analytical method in wood chemistry, Pulp and Papermaking, Springer series in wood science, New York. 1998.
62. Azubuike C. P. and Okhamafe A. O. Physicochemical, spectroscopic and thermal properties of microcrystalline cellulose derived from corn cobs. *International Journal of Recycling of Organic Waste in Agriculture* 2012, 1:9
63. Oluwasina O., Lajide L. and Owolabi B. Microcrystalline cellulose from plant waste through sodium hydroxide-anthraquinone-ethanol pulping. *Bio-Resources*. 2014; 9(4):6166-6192.
64. Peter McKendry. Energy production from biomass (part 1). *An overview of biomass, Bioresource Technology*. 2002; 83; 37–46.
65. Bhagwanrao SV, Singaravelu M. Bulk density of biomass and particle density of their briquettes. *International Journal Agric Engineering*, 2014; 7: (221)–4.
66. Lam PS, Sokhansanj S, Bi X, Mani S, Lim CJ, Womac AR. : Physical characterization of wet and dry wheat straw and switchgrass - Bulk and specific density. ASABE meeting. Minneapolis Convention Center, Minneapolis, Minnesota, USA; 2007, Paper Number: 076058.
67. Ueki Y, Torigoe T, Ono H, Yoshiie R, Kihedu JH, Naruse I. Gasification characteristics of woody biomass in the packed bed reactor. *Proc Combust Inst*; 2011; 33:1795–800.
68. Dahlquist E. 2013, Biomass as energy source: resources, systems and applications. CRC Press.
69. Miao Z, Grift TE, Hansen AC, Ting K. Flow performance of ground biomass in a commercial auger *Powder Technology*. 2014; 267: 354-361 8 p
70. Garcia R.A., Flores R.A. and Mazenko C.E. Factors contributing to the poor bulk behavior of meat and bone meal and methods for improving these behaviors. *Bioresource Technology*. 2007; 98(15):2852-2858.
71. Thalberg K., Lindholm D., and Axelsson A. Comparison of different flowability tests for powders for inhalation. *Powder Technol*. 2004; 146-206-213.
72. Miccio F, Silvestri N, Barletta D, Poletto M. Characterization of woody biomass flowability. *Chem Eng Trans*; 2011; 24: 643–648
73. Lumay G, Boschini F, Traina K, Bontempi S, Remy J-C, Cloots R. Measuring the flowing properties of powders and grains. *Powder Technology*. 2012; 224:19–27.
74. Seville J. P. K., Tüzün, U., and Clift, R. Processing of particulate solids', Powder Technol. Ser., IInd Edition, 1997
75. Medic D., Darr M., Shah A., Potter B., Zimmerman J. Effects of torrefaction process parameters on biomass feedstock upgrading. *Fuel* 2012; 91: 147–154.
76. Bridgwater AV, Boocock D.G.B. Developments in thermochemical biomass conversion, 1. Netherlands: 2013, Springer.
77. Muna H.A.: Extraction of Cellulose from some Industrial and Plant's Waste and its hydrolysis using new heterogeneous catalyst at thesis Submitted to the College of Science University of Baghdad In Partial Fulfillment of the Requirements of the Degree of M.Sc. In Chemistry 2013-63.
78. Granstrom M. Cellulose derivatives: synthesis, properties and application. An academic dissertation submitted to the faculty of Science, University of Helsinki. 2009.
79. Owen N. L. and Thomas D. W. Infrared studies of "hard" and "soft" woods, *Applied Spectroscopy*. 1989; 43: 451–455.

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