Impact of Hydrolysis on Thermal Stability and Binding Capacity of Torrefied-densified Iron (Lophira Alata) Wood Dust

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Abstract

This project examined the impact of hydrolysis on the thermal stability and binding capacity of torrefied-densified wood biomass from the red iron tree (Lophira alata), with the goal of utilizing it as a feedstock for pellet fuel. Initial analysis, including proximate, ultimate, fiber, and physico-chemical analysis, were conducted on raw wood dust samples before hydrolysis and torrefaction. The wood dust sample with the highest energy value from the hydrolyzed-torrefied samples was investigated as a potential feedstock. Physico-chemical analysis of the raw biomass samples were conducted before torrefaction and hydrolysis treatments. Torrefaction experiments were conducted on the raw and hydrolyzed samples at various process temperatures (200 °C, 250 °C, and 300 °C) for 45 minutes in a custom-made laboratory-scale torrefier. The results showed that hydrolyzed and torrefied wood dust samples differed in color from the untorrefied (raw) samples, indicating the physical impact of the treatment methods. As process temperature increased, higher heating value (HHV) rose, while energy yields decreased. Proximate analysis revealed that hydrolysis significantly improved the fixed carbon content of the wood dust biomass, with a remarkable increase in hydrolyzed samples compared to their unhydrolyzed counterparts. The highest fixed carbon content (91.66 %) was achieved with the hydrolyzed sample torrefied at 283 °C, while the lowest value (74.79 %) was recorded in the unhydrolyzed sample. Similarly, hydrolysis affected fiber composition, with lignin content increasing to 35.33 % in the hydrolyzed-torrefied sample, compared to 15.43 % in the unhydrolyzed sample. Ultimate analysis showed lower H/C and O/C values (0.003 and 0.100, respectively) in the hydrolyzed sample, indicating enhanced thermal stability and energy value. These results demonstrate that hydrolysis prior to torrefaction significantly impacts lignin content, binding capacity, fixed carbon, and energy value, making hydrolyzed-torrefied wood dust biomass of Lophira alata a promising feedstock for pellet fuel production.

INTRODUCTION

The increasing need for fossil fuel-based energy in most developing countries, particularly Nigeria, has led to an astronomical rise in the price and frequent shortages of the conventional sources of energy, such as kerosene, liquefied petroleum gas, and petrol [1]. The country's dependence on these non-renewable oils has also propagated environmental and human health problems, such as air pollution, environmental ecosystem degradation, deforestation, and toxic gas emittance, which cause climate change and depletion in the ozone layer [1]. These combined economic and environmental pressures have induced a need for the investigation of alternative energy sources characterized by being renewable, green, less expensive, and more accessible that can be integrated into the energy scenario [2].

To address this challenge, there has been a growing focus on developing forest and agroresidues as alternative energy resources. Wood dust (sawdust) is one such material, which is obtained during the processing of timber and has received attention as a good feedstock for the production of solid biofuels or bio-coal [3]. Sawing mills produce tremendous amounts of wood dust every day, most of which is not

used in an effective way. Consequently, these residues are a source of waste nuisance and could also be harmful to human health when disposed of unscientifically [4].

Nevertheless, to date, this waste material is not effectively managed to harness its energy and economic value. This is mainly because of a lack of access to effective bioconversion technologies and inadequate technical skills in the conversion of this biomass into value-added energy products [3, 5]. Moreover, the utilization of sawdust via appropriate thermochemical processes could also be useful for waste management and environmental cleanliness due to a decrease in the waste/wood pile-up problem as well as a source of sustainable energy generation.

Biomass primarily consists of carbohydrate polymers with a small amount of inorganic materials and a low level of extractives. Its cellular structure comprises cellulose, hemicellulose, and lignin as fundamental building blocks [6]. Cellulose, the predominant component in woody biomass, typically accounts for 65–75 wt% of the material. It exhibits greater resistance to depolymerization compared to hemicellulose, decomposing within the temperature range of 250°C to 350°C. Hemicellulose constitutes approximately 25–35 wt% of wood and degrades between temperatures of 130°C and 260°C [7]. Lignin, situated in the cell wall among hemicellulose, cellulose, and components of pectin, acts as a binding agent in biomass. It demonstrates higher resistance to thermal degradation than cellulose or hemicelluloses, decomposing within the temperature range of 290°C and 500°C. Lignin typically comprises 30–45 wt% of biomass content [8]. Additionally, the proportions of these chemical constituents in biomass vary depending on the type of biomass [9]. Different types of biomass have been considered as energy source, ranging from oilseeds, woods, agricultural residues, biomass-processing wastes and even pure chemicals [10].

The waste generated from *Lophira alata* (LA) is subjected to torrefaction to produce bio-coal or solid fuel. Although there are numerous potential applications, products, and markets associated with this significant wood species (*Lophira alata*), research efforts on the bio-refining concept are still in the early stages in Nigeria. A bio-refinery represents a zero-waste process where waste from one process is utilized as raw material for another process, thereby enhancing efficiency and/or income [11]. One of the most prevalent types of biomass that is currently utilized extensively for the manufacture of high-value chemicals and commodities as well as renewable bioenergy is woody biomass [12].

Biomass can be transformed into gas, liquid or solid fuel through a variety of chemical processes such as gasification, anaerobic digestion, pyrolysis, fermentation, combustion and trans-esterification for the purpose of obtaining heat, power, synthesis gas and biofuels [13]. However, due to its disadvantages as solid fuel, large-scale use of woody biomass has been limited. Past studies suggest that untreated woody biomass exhibits characteristics such as elevated moisture content, diminished heating value, hygroscopic properties (absorption of moisture), challenging grindability (resistance to pulverization), low energy density (excessive bulkiness, rendering it uneconomical for long-distance transportation), non-uniformity (significant variability in combustion properties, as well as in sizes, shapes, and types), and reduced combustion efficiency (resulting in smoking during combustion) [14]. One of the ways to

address this issues to a reasonable extent is torrefaction. Some agro-forestry residues biomasses such as wood dust are used to generate energy in form of heat and power [15]. Wood dust popularly known as sawdust in Nigeria, is mill waste in form of powder that is recovered from wood when being cut by a saw. It is available in wood processing industries such as saw mill. Wood dust biomass residue is commonly utilized as household fuel in numerous Nigerian homes due to its elevated energy content and heating values. However, wood dust is not optimally efficient as fuel in its natural state but can be significantly improved through thermo-conversion processes like torrefaction [16]. Traditional bio-coal is generated via torrefaction. Bio-coal derived from wood dust biomass has the potential to serve as a reliable alternative to fossil fuels, given its renewability and sustainability [17].

Torrefaction is a procedure for transforming biomass into a substance resembling coal, typically carried out at moderate temperatures ranging from 200–300°C, with low or no oxygen present and under atmospheric pressure conditions [18]. This conversion process, known as torrefaction, reduces moisture content and increases the calorific value of biomass, thereby enhancing its energy potential. During torrefaction, the heating of biomass causes slight degradation of cellulose, with the majority of hemicellulose decomposing through deoxygenation, dehydration, and dehydrogenation reactions [19]. Following torrefaction, the result is a uniform solid product with higher energy content and lower moisture content compared to untreated biomass. Another implication of torrefaction is that it reduces oxygen-carbon (O/C) and hydrogen-carbon (H/C) ratio whereas the content of high energy C-C bond is increased and makes it effective for combustion and gasification [20]. Moreover, the treatment influenced the grindability and hydrophobicity properties of biomass i.e. it makes biomass easy to grind and also improved its water-hating property with reduced O/C and H/C ratio [3]. During torrefaction, hemicellulose decomposition is initiated and leads to biomass's change in color due to loss of moisture, CO_2, and large amounts of acetic acid and phenols [21]. An increase in fixed carbon through torrefaction makes it easy for biomass to be able to burn for a long period of time and also, less smoke is detected due to the fact that volatile compounds are separated by torrefaction [22].

Lophira alata, commonly known as Azobe/Ekki or the Red Ironwood tree, is a pioneer species abundant in wet evergreen forests, thriving in environments such as evergreen and moist deciduous forests, freshwater swamp forests, and near riverbanks [23]. This species, belonging to the Ochnaceae family, is distributed across various African countries including Cameroon, the Republic of the Congo, the Democratic Republic of the Congo, Ivory Coast, Equatorial Guinea, Gabon, Ghana, Liberia, Nigeria, Sierra Leone, Sudan, and Uganda [24]. Typically found in subtropical or tropical moist lowland forests, its habitat faces threats due to habitat loss.

Lophira alata is renowned for its highly durable wood, utilized for various purposes such as railroad ties, groynes, and bridge planking. The tree typically features a straight trunk, occasionally swollen at the base, and usually lacks buttress roots, with branches absent for approximately 30 meters from the ground [25].

MATERIALS AND METHODS

Materials

The Plant sample Lophira alata Banks ex Gaertn. F. (1805) was harvested legally from Kingtilo Sawmill according to the relevant sawmill conservation guidelines ondo state. A voucher specimen was taken to the Herbarium Centre of Federal University of Technology Akure (FUTA), Ondo State Nigeria where it was authenticated by a botanist, Dr. Oseyemi O. J with the voucher specimen number as FUTA0109 (Supplementary Material, Fig. S1). All reagents used were sourced from Chemistry Department Federal University of Technology, Akure and were of analytical grade. All the equipment used were obtained from chemistry laboratory and neighbouring universities.

Sample Preparation and Analysis

The samples were oven dried at a temperature of 50^{O}C for 4hr to a point of achieving a constant weight, and screened with an analytical sieve size 1.7 mm mesh to obtain particle size of 1.7mm. The screened samples were properly labelled as raw iron wood dust (RIWD).

Preliminary Analysis of the Raw Wood Dust Biomass

Prior to the torrefaction process, preliminary analysis were conducted on the untorrefied (raw) sample to ascertain the initial heating value of the wood dust using bomb calorimeter, proximate analysis to evaluate the fixed carbon of the raw sample, fibre analysis to understand chemical component such as hemicellulose, cellulose, lignin, extractives and elemental composition to gain insight into H, C, O, S, N contents of the biomass.

Information on the Structural composition, surface morphology and thermal stability of the raw biomass were obtained by using Fourier-transform infrared spectrometry, scanning electron microscope and thermo-gravimetry analysis.

Proximate Analysis of the Raw Wood Dust Biomass

Moisture content (M.C), ash content (A.C), and volatile matters (V.C) were assessed following the guidelines of ASTM D-3173-03, ASTM D-3174, and ASTM D-3175, respectively. The determination of fixed carbon (F.C) in the samples was achieved using the method of calculation outlined by [26].

Determination of Percentage Moisture

The initial moisture content of the samples was determined by oven drying method (modified) as described by [27].

A clean porcelain dish was dried in oven and weighed (w_1). 1.034 g of the biomass sample was put into the porcelain dish and weighed (w_2). The dish and the biomass samples were placed in the oven at 105 ℃ for 2h [28]. The dish and content were removed from the oven, and cooled in a dessicator and then re-weighed. The heating and weighing were repeated at intervals of 30 min until constant weights (w_3) of the samples were attained.

Percentage moisture of the biomass was estimated from the expression in Eq. 1.

$$\%MC = \frac{w_2 - w_3}{w_2 - w_1} x\ 100\%$$

1

Determination of Percentage Ash of the Biomass

The measurement of ash content in the samples was conducted following the standardized procedure outlined in ASTM D-3174 [29]. A clean crucible was placed inside an oven for 20 min at 105 ℃, cooled at 32 ℃ in a dessicator and weighed (w_1). 2 g of the moisture- free sample was weighed and placed into the crucible. The crucible containing the sample was put inside a muffle furnace for a duration of 4 h, maintaining a temperature of 550°C. The percentage of ash in the sample was determined using Eq. 2:

$$\%\ A = \left(\frac{d}{a}\right)\ 100\ \%$$

2

Where; a = weight of original sample and d = weight of ash

Determination of Percentage Volatile Matter of the Biomass

The volatile matter was determined using ASTM standard method (ASTM D-3175) as described by [30] without any modification. A crucible was cleaned and placed in an oven for about 20 min and the dried weight of the empty crucible was recorded after cooling. Two (2) grams of the moisture-free sample was weighed into the clean and dried empty crucible and covered to avoid contact with air during devolatilization. The covered crucible was then placed in a muffle furnace (SXL) maintained at a temperature of 950 ^{o}C. The crucible was heated for 7 min. After 7 min heating time, the crucible was taken out and cooled in desiccator and weighed immediately to evaluate the weight loss due to devolatilization. The weight loss was calculated using Eq. (3).

$$\%\ Volatile\ matter\ \left(\frac{b-c}{a}\right) 100\ \%$$

3

Where a = weight of the original sample, b = weight of the oven dried sample at 105 ^{O}C and c = weight of the dried sample at 950 ^{O}C.

Determination of the percentage fixed carbon content of the biomass

The percentage of fixed carbon of the biomass (untorrefied and torrefied) was estimated by difference as given in Eq. 4 [31].

$$\% F.C. = 100 - (\% M.C. + \% V.M. + \% A.C.)$$

4

Where: F.C. = Fixed Carbon, M.C. = Moisture Content, V.M. = Volatile Matter and A.C. = Ash Content

Fibre Composition Analysis of the Raw Wood Dust

Iron (*Lophira alata*) wood dust was used as a biomass sample which representatives of highest production of local biomass. The iron wood dust was cut sieved tiny pieces (range 1–2 mm in length). They have undergone the drying process by following the LAP-Preparation of the sample for compositional analysis [32]. The biomass was placed under direct sun for three months before the size reduction process applied. By using laboratory blender with a single speed of 230 V by WARING/USA brand, the biomass was blend to a smaller size and passed the test sieve pan with size 35 meshes (500 microns).

The amount three components of lignocellulosic (cellulose, hemicellulose and lignin) in Iron wood dust was determined by the method proposed by [33]. For the experimental process, 4 types of the chemical will be used; (1) Acetone, (2) Sodium hydroxide (NaOH − 0.5 mol/L), (3) Sulphuric acid (98%) and (4) Barium chloride solution.

Determination of the Extractive Content of the Biomass

Acetone (60 mL) was added to 1 g iron wood dust (A). The temperature 90°C controlled by using a hot plate for 2 hours [33]. After 2 hours, the sample dried in an oven at 105–110°C until constant weight was obtained (B). By using Eq. (5), the amount of extractives was identified.

A-B = Amount of Extractives (g) (5)

Determination of the Hemicellulose Content of the Biomass

150 mL of Sodium Hydroxide (NaOH) solution (0.5 mol/L) was added to 1 g of iron wood dust with extractives free (B). The temperature 80°C controlled by using a hot plate for 3.5 hours [34]. After that, the sample was washed with deionised 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). By using Eq. (6), the amount of hemicellulose was identified.

B-C = Amount of Hemicellulose (g) (6)

Determination of the Lignin Content (acid insoluble lignin) of the Biomass

30 mL of 98% Sulphuric Acid added to 1 g iron wood dust with extractives free (B). The sample left at ambient temperature for 24 hours before boiled at temperature 100°C [35] controlled by using a hot plate for 1 hour. 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 in an oven at 105–110°C until constant weight was obtained (D). The final weight of residue is recorded as lignin content seen in Eq. (7)

D = Amount of Lignin (g) (7)

Determination of the Cellulose Content of 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 [36], the content of cellulose (E) will be identified.

(A-B) + (B-C) + D + E = 1g (8)

Elemental Composition Analysis of Raw Wood dust

The analysis of the fundamental makeup of wood biomass, encompassing carbon (C), hydrogen (H), oxygen (O), nitrogen (N), and sulfur (S), was carried out through ultimate analysis, following the procedure detailed in the methodology described by [37]. The device was employed to measure and document the elemental composition percentages of carbon (C), hydrogen (H), nitrogen (N), and sulfur (S). The oxygen proportion within the biomass was approximated by deducing the combined percentages of C, H, N, and S from a total of 100%. The percentage elemental composition was calculated using Eq. (9)

$$[100 - (\%\,C + \%\,H + \%\,N + \%\,S)]$$

9

Determination of the High Heating Value of the Raw (Untrorrefied Sample)

The energy value also known as heating value was evaluated as described by [37]. 0.5g of the sample was used for the analysis using bomb calorimeter (e2K combustion calorimeter). Determination was carried out in accordance with ASTM D5865-04.

Torrefaction of the Wood Dust Biomass

The torrefaction procedure adopted was as described by [21], using a Laboratory–scale torrefier with a slight modification. The improverised torrefaction Machine **(Fig. 1a)** comprised a gas cylinder flame source, an air-tight inner container to hold the biomass sample, a digital thermometer to measure the temperature at which the biomass was torrefied, and a digital temperature sensor. To conduct the experiment with a restricted amount of oxygen, the laboratory sample was put inside the cylindrical steel container and sealed. In order to heat the biomass indirectly, the sealed container was put inside the torrefier, which is joined to a burner and connected to a gas cylinder through a pipe **(Fig. 1b)**. Under residence time of 45 min, the torrefaction experiment were conducted at three distinct process temperatures: 200 ^{o}C for light torrefaction, 250 ^{o}C for medium torrefaction, and 300 ^{o}C for severe torrefaction. The following analysis were conducted on the torrefied samples to ascertain; the calorific value of the torrefied wood dust using bomb calorimeter as earlier described, proximate analysis to evaluate fixed carbon content, fibre analysis to evaluate lignin content after torrefaction and elemental composition to gain insight into H, C, O, S, N contents of the biomass.

Information on the Structural composition, surface morphology and thermal stability of the torrefied biomass were obtained by using Fourier-transform infrared spectrometry, scanning electron microscope and thermo-gravimetry analysis.

Acid hydrolysis of the Raw (untorrified) and Torrefied samples

Three hundred (300) grams of the raw (untorrefied) biomass sample were soaked in 4% sulphuric acid solution and subjected to heating at 102 ^{o}C for 30 min using the modified procedure described by [38]. The pretreated biomass was washed thoroughly with tap water, subsequently, 0.1 M sodium hydroxide was added to neutralise the acid, and the sample was stirred thoroughly with tap water and then with distilled water until neutrality. The supernatant was drained from the residual biomass using a sieve and tested with litmus paper to ascertain neutrality. The residual biomass was air-dried for 6 d to ensure thorough dryness. The dried biomass was weighed, packed and labelled as hydrolysed-untorrefied sample.

The procedure was repeated for the torrefied sample using the same quantity of the biomass and under the same experimental conditions. The dried sample obtained was labelled as hydrolysed–torrefied sample.

Similarly, the calorific value of the sample was determined as well as the compositional analysis such as proximate, fibre and ultimate were performed on the sample. Information on the Structural composition, surface morphology and thermal stability of the sample were obtained by using Fourier-transform infrared spectrometry, scanning electron microscope and thermo-gravimetry analysis.

Removal of Lignin

The lignin content of the sample was evaluated by using 15% alkali solutions of NaOH in distilled water which was be placed in a beaker and 20 g of wood dust will be added and stirred for about 1hr using shaker [39]. The solution was removed from the shaker and filtered using the vacuum filter to obtain liquor filtrate. The filtrate is acidified by adding 4% sulphuric acid drop by drop until it reaches PH 2. After acidifying process, the solution was be centrifuged at 3500 rpm for 10 mins to obtain the lignin precipitate. After centrifugation, two layers was obtained (liquid and precipitate) and the liquid layer was discarded. The precipitate was oven dried overnight at 50 ^{O}C to recover the dried precipitate of the lignin. The dried lignin will be labelled and stored as binder for biomass densification and the lignin obtained will be subjected to statistical analysis using appropriate software.

Fourier-transform infrared spectrometric analysis (FTIR)

Sample (1g) was weighed and mixed with 100g of the binder (KBr). The mixture was ground to fine powder making sure a homogenous mixture is achieved. The “O”-ring was placed on one piece of the die, the homogenous mixture was then added on the “O”-ring and the die was assembled on the hand press. Pressure was applied using the hand press for about 1 minute to compressed the sample onto the “O”-ring.

A plunger was used to remove the “O”-ring from the die and the compressed sample was placed in the pellet holder and then placed on to the sample holder of the FTIR equipment scanning [40].

Scanning Electron Microscopy (SEM) analysis of raw and treated samples

The morphological characteristics of raw and treated biomass samples used for Bio-coal production were examined by electron microscope (SEM, JSM-6390 LV, 6733 B-IUUSSN). The samples were mounted on the SEM stubs with adhesive carbon and coated in 20nm Carbon with a QUORUM Q150RES mini sputter coater and then analysed with a Phenom PRO-X SEM equipped with an Oxford XMax 50 Silicon Drift Energy Dispersive X-ray detector at 15KV under high vacuum [41].

Densification of wood dust biomass

The sample was prepared using lignin extracted from the wood dust biomass. The extracted lignin was mixed with warm water in the ratio of 1:2 that is one milliliter (1ml) of warm water to two milliliters (2ml) of lignin [42]. The prepared sample was loaded into a mechanical hydraulic press for compaction of the wood dust biomass. During this process in the hydraulic press, water was removed and after two minutes (2mins), the Briquette sample was removed from the mechanical hydraulic press and dried for three (3) days. The small round cylindrical shape was measured to be 6mm in diameter using vernier caliper. The shapes are uniform in size, making them easy to handle and burn.

Thermogravimetric Analysis (TGA)

The thermal study of the raw and treated samples were carried out using thermogravimetric analyzer. The samples were dried to remove moisture. The moisture-free samples (20g) was placed in a sample pan or crucible. Thermogravimetric analyzer was set on heating rate of 20 ℃ per minute and conducted on temperature range from 27 ℃ – 884 ℃ [43]. Nitrogen gas was used to minimize oxidation of the sample. The instrument recorded moisture and the weight loss of the sample as a function of temperature.

The weight loss was expressed as a percentage of the initial weight.

% weight loss = $\frac{WL}{Wi} X\ 100$

Where Wi = initial weight, $Wf =$ final weight and $WL =$ weight loss

Hence, $WL =\ Wi -\ Wf$

RESULTS AND DISCUSSION

Proximate Analysis of the raw (Untorrefied), Torrefied, Hydrolysed and Hydrolysed-torrefied Wood Dust Biomass Samples

Percentage Moisture Content, Volatile Matter, Ash content and Fixed Carbon of the Biomass

Proximate analysis was performed on the raw iron wood dust (RIWD), torrefied iron wood dust (TIWD), Hydrolyzed raw iron wood dust (HRIWD) and Hydrolyzed- torrefied iron wood dust(HTIWD) samples in order to assess potential of the biomass as energy feedstock. Therefore, parameters such as moisture content (MC), volatile matter (VM), ash content (A) and fixed carbon (FC) were evaluated for each set of the biomass samples.

Table 1 shows the percentage moisture content of the wood dust samples. The raw (RIWD) sample has a moisture content of 15.28%, Torrefied (TIWD) wood dust shows the least moisture content of 2.82%, at a process temperature of 300℃, (HIWD) sample recorded 15.16% while the Hyrdrolysed-torrefied (HTIWD) sample has 3.89% at 283℃ process temperature.. Comparing the moisture contents of the biomass with the energy values depicted in Table 1 shows inverse proportionality between moisture content and energy value of the biomass. Such comparison provides valuable information on the combustion efficiency and storage stability of the wood dust biomass. The raw wood dust with the highest moisture content of 15.28% has a corresponding calorific value of 17.32MJ/Kg which is the lowest. Conversely, the hydrolysed sample of the biomass torrefied at 283℃ has a reduced moisture content of 3. 89% and a corresponding high heating value of 22.72 MJ/Kg. Generally, the resultsshow that moisture content of the biomass has direct impact on its high heating value and subsequently on

the ignition temperature of the biomass. As stated in the findings of, by [44], low moisture content and high energy value of biomass reduce the likelihood of slag formation during combustion. Hence, coupled with the positive impact of hydrolysis and torrefaction on the energy value of the biomass, *Lophira alata* wood dust exhibits viable potential as feedstock for briquettes and pellet fuels production.

Volatile matter (VM) is a critical component of biomass energy. It plays a significant role in the combustion process and energy production. The lower the volatile matters in a wood biomass, the higher its combustion efficiency.

In Table 1, the volatile matter for RIWD, TIWD 200 ℃, TIWD 250 ℃, TIWD 300 ℃, HIWD, HTIWD 200 ℃, HTIWD 250 ℃ and HTIWD 283 ℃ were 1.57%, 2.45%, 7.38%, 18.69%, 0.44%, 1.93%, 3.44% respectively as depicted in Table 1. The result shows that there is more solid content that is reactive and could easily ignite in TIWD @ 300 ℃ when heated than those of RIWD, TIWD 200 ℃, TIWD 250 ℃, HIWD, HTIWD 200 ℃, HTIWD 250 ℃ and HTIWD 283 ℃. The lowest volatile matters of the wood dust was observed in HIWD (0.44%) while the highest value was found to be 18.69% in TIWD @ 300 ℃. Generally, it observed that the volatile matters of all the hydrolyzed samples are relatively lower than those of the raw and torrefied samples. This simply implies higher energy density, lower emission (less pollution), higher thermal efficiency, improved ignition and combustion stability of the wood biomass upon acid hydrolysis. Hence, volatile matter has significant influence on the combustion efficiency of fuel [45].

Table 1 shows the percentage ash content of the wood dust samples. The raw (RIWD) sample has an ash content of 1.06%, Torrefied (TIWD) wood dust has the lowest ash content of 2.29% at a process temperature of 200 ℃, Hydrolysed (HIWD) sample recorded 0.60% while the Hyrdrolysed-torrefied (HTIWD) sample has 0.01% at 283 ℃which is the least of all sample hydrolysed. The result shows a clear impact of hydrolysis on the ash content of the wood biomass, as all the samples subjected to hydrolysis process reveal appreciate reduction in their ash contents compared with the raw and torrefied samples Comparing the ash contents of the biomass with the energy values depicted in Table 1 shows inverse proportionality between ash content and energy value of the biomass. Such comparison provides valuable information on the thermal efficiency of the wood dust biomass. The raw wood dust with the lowest ash content of 1.06% has a corresponding calorific value of 17.32 MJ/Kg which is the lowest. Conversely, the hydrolysed sample of the biomass torrefied at 283℃ has a more reduced ash content of 0. 01% and a relatively higher heating value of 22.72 MJ/Kg. The ash content of the TIWD 283 biomass (3.70%) is comparable to that of other agro-residues, such as tamarind husk, coffee husk, cotton shells, tannin waste, and almond shell, which range from 4.2% to 4.8% [46]. The results of this analysis clearly show the effectiveness of hydrolysis treatment on the ash content, which remarkably reduced from 1.06% in the raw biomass to 0.60% in the hydrolyzed. Elevated ash levels pose challenges in furnaces due to increased risks of ash deposition, slag formation, and furnace surface corrosion. Ash content serves as an indicator of biomass slagging behavior, with lower ash content generally preferred for biomass conversion processes.

Fixed carbon is the amount of materials burns in solid state which can be attributed to the amount of carbon that remain after volatile matters has been released from combustion process. Table 1 shows the percentage fixed carbon of the wood dust samples. The raw (RIWD) sample has fixed carbon of 82.09%, Torrefied (TIWD) wood dust has the lowest fixed carbon of 74.79% at a process temperature of 300 ℃, and Hydrolyzed (HIWD) sample recorded 83.80% while the Hyrdrolysed-torrefied (HTIWD) sample has the highest value as 91.66% at 283 ℃process temperature. Correlating the fixed carbon values and energy values of the biomass, a direct proportionality between the two parameters was observed, that is, the more the fixed carbon, the higher the energy value. The raw wood dust with the lowest fixed carbon of 82.09% has a corresponding calorific value of 17.32 MJ/Kg which is the lowest. Conversely, the hydrolyzed sample of the biomass torrefied at 283℃ has a fixed carbon of 91.66% with a correspondingly high heating value of 22.72 MJ/Kg. Percentage fixed carbon of 66.0% reported for Alstonia cogenesis sample torrefied at the optimum process temperature of 300℃ by [3] was comparatively lower than 74.79% recorded for *Lophira alata* at the same process temperature of 300℃. This clearly shows that wood dust biomass of *Lophira alata* contain more carbon. The percentage fixed carbon of the biomass was remarkably enhanced upon hydrolysis of its torrefied sample. Thus making the biomass a more efficient and valuable energy source.

Table 1
Proximate Composition of the raw, torrecfied, hydrolyzed and hydrolyzed torrified wood dust of**Lophira alata**

Sample	**Moisture (%)**	**Volatile matter (%)**	**Ash (%)**	**Fixed carbon (%)**
RIWD	13.28	1.57	1.06	80.09
TIWD 200^{O}C	6.60	2.45	2.29	88.66
TIWD 250^{O}C	5.77	7.38	2.89	83.96
TIWD 300^{O}C	6.82	20.69	5.70	82.79
HIWD	15.16	0.44	0.60	83.80
HTIWD 200^{O}C	7.04	1.93	0.41	90.62
HTIWD 250^{O}C	4.81	4.79	0.10	90.30
HTIWD 283 ^{O}C	3.89	3.44	1.01	91.66

Where RIWD means Raw Iron Wood Dust

TIWD 200 ^{O}C, 250 ^{O}C, 300 ^{O}C means Torrefied Iron Wood Dust at 200 ^{O}C, 250 ^{O}C, 300 ^{O}C

HIWD means Hydrolysed Iron Wood Dust

HTIWD 200 ^{O}C, 250 ^{O}C, 283 ^{O}C means Hydrolysed Torrefied Iron Wood Dust at 200 ^{O}C, 250 ^{O}C, 283 ^{O}C

The Fibre Composition of the Untorrefied (raw) and Torrefied Samples

Percentage Hemicellulose, Lignin, Cellulose and Extractive

Table 2 shows the fibre composition of wood dust biomass samples derived from *Lophira alata* tree. Preliminary fibre analysis was performed to confirm the chemical structure and the mass percentage of hemicellulose, lignin, cellulose and extractive of wood dust biomass sample. It was seen that, the chemical composition such as hemicellulose, lignin, cellulose, and extractive content of raw and torrefied at 300 ℃, are in the range of 10–20%, 15–25%, 55–70% and 7–11% respectively. This result is in accordance with the assumed range of chemical components of the biomass reported by [3]. The lignin content of a biomass can be attributed to the pelletability of the biomass. Lignin in a biomass serve as a binding agent, it acts as a cementing material and fill the space between the hemicellulose and cellulose. However, it can be concluded that raw wood dust biomass sample with the lowest lignin have poor pelletability while the hydrolysed-torrefied biomass would give great response to pelletalization due to its higher percentage of binding agent (lignin).

Table 2
Fibre Composition of Untorrefied, Torrefied and Hydrolysed Wood Dust Sample

Sample	Hemicellulose%	Lignin%	Cellulose%	Extractive%
RIWD	11.71	15.43	65.58	7.28
RTIWD 300 ^{O}C	7.60	29.73	60.67	2.07
HTIWD 283 ^{O}C	3.58	35.33	59.10	1.99

Where RIWD means Raw Iron Wood Dust

RTIWD 300 ^{O}C means Raw Torrefied Iron Wood Dust at 300 ^{O}C

HTIWD 283 ^{O}C means Hydrolysed-torrefied Iron Wood Dust at 283 ^{O}C

The Effect of Hydrolysis on the Fibre Composition of the Biomass

Table 2 depicts the effect of hydrolysis on the fibre composition of the biomass samples sourced from Red Iron tree (*Lophira alata*) tree. The Table 2 depicts the fibre composition of raw, torrefied and hydrolysed samples at process temperature of 300°C. The Lignin content was discovered to increase to 29.73% after torrefaction which was initially 15.43% in its raw form and increase to 35.33% after hydrolysis. It was also observed in cellulose content of the biomass sample, 65.58% was detected in the biomass before treatment which was later decreased to 60.67% upon torrefaction and 59.10% upon hydrolysis. Due to the increase in the lignin content, this result showed that hydrolyzing biomass prior to

torrefaction increases the lignin content which enhances binding property of biomass during densification [47]. However, hemicelluloses and extractives which were 11.71%, 7.28% respectively in their raw forms decreased to 7.60%, 2.07% after torrefaction and 3.58% and 1.99% respectively after hydrolysis. This implies that the fuel properties of the biomass can be enhanced through the process of torrefaction [48].

Torrefaction of the Raw (untorrefied), Torrefied and Hydrolysed Biomass

Colours of the Torrefied and Untorrefied Biomass

Colour differences among raw (nontorrefied) and various torrefaction temperature levels are shown in Fig. 1.1. The torrefed samples were visually different in colour from the untreated biomass. With the increasing torrefaction temperature approach from 200°C to 300°C, biomass changed to darken sequentially, and it can even be observed with the naked eye that those samples were exposed to visible light treatment (treat), medium heat treatment and strong heat treatment [49].. When mildly torrefied, the samples were dark brown, while in light torrefaction, they were graded from light brown to brown. A dark grey product with much loss of moisture and volatile compounds was obtained when torrefaction was at 300°C. These colour changes correspond to degradation levels and reveal the extensive chemical modification of cellulose, hemicellulose, and lignins [50]. The variation in colour can be explained by the reaction units of functional groups, such as hydroxyl, carbonyl, methoxyl, and phenolic compounds, that absorb visible light, causing those parts of biomass to darken. Under extreme torrefaction, as many as 78% of C-H and C-O bonds are broken, resulting in the decomposition of oxygenated and hydrophilic groups and promoting hydrophobicity [51].. Further, phenol and aldehyde formation at low torrefaction could impart a brown appearance to the moderately treated samples.

Figure 1 shows the colors of the untorrefied (raw) wood dust biomass sample with some of the torrefied samples under different thermal treatment conditions. The torrefied biomasses produced upon torrefaction pre-treatment method were noticeably different in colour from the untorrefied (raw) samples. There were obvious changes in the colour of the samples as process temperature increased from 200 ºC to 300 ºC. Therefore, samples that were exposed to light, mild and severe thermal treatments can be easily distinguished. The samples subjected to light thermal treatment changed from light brown to brown while the mild torrefied samples yielded a dark brown colour. The samples subjected to severe thermal treatment, that is, those samples torrefied at process temperature of 300 ºC yielded a black product. From this result, it was observed that highest amount of moisture and volatile matters were lost under severe torrefaction. The frequent change in colour as temperature increases indicated the degree of conversion of the samples. This indicates that there was a great transformation in the chemical nature of cellulose, hemicellulose and lignin as a result of thermal degradation [49]. The alteration in biomass color can be ascribed to the generation of hydroxyls, carbonyls, methoxyls, and phenolic compounds during thermal treatment. These chemophoric compounds have the capability to absorb incident visible light, resulting in the dark coloration observed in biomass [50]. Consequently, understanding the behavior of biomass under severe torrefaction conditions becomes important, as

these compounds are formed during such conditions. Under severe torrefaction, approximately 78% of molecular C-H and C-O bonds are destroyed, leading to the breakdown of oxygenated and hydrophilic compounds and thereby enhancing the hydrophobic properties of the biomass through the formation of black hydrophobic compounds [51]. Additionally, the emergence of phenols and aldehydes during mild torrefaction conditions might contribute to the coloration of mildly torrefied biomass.

This result is in agreement with the findings reported on the upgrading of fuel properties of rice husk [52] and Melina and Teak wood [50, 53]. Also this findings is consistently reported on the viability of Astonia congenisis by [3].

High Heating Value of the Raw (Untorrefied), Hydrolysed and Torrefied Samples

Effect of Torrefaction on the High Heating Values (Energy Contents) of the Wood Dust Samples

Table 3 shows the results of high heating values (HHV) of raw (untorrefied) wood dust samples investigated. This preliminary investigation was conducted to examine the most viable biomass as feedstock for bio-coal production. Hydrolysed-torrefied wood dust at 300 ℃ showed the highest energy value 22.721 MJ/Kg which is comparatively higher than the energy values of other samples.

The high energy value recorded in Hydrolysed-torrefied Wood Dust biomass can be attributed to the relatively low moisture content (3.89%) recorded in the biomass compared to its other counterparts that were relatively higher in moisture content. Furthermore, the fixed carbon content of 91.66% also confirmed the high energy content of the biomass, which will greatly enhance its fuel property as solid fuel. The heating value or energy value of a biomass depends greatly on the moisture content and chemical composition of the biomass [54]. This finding validates the effectiveness of the torrefaction process in augmenting the energy content of the biomass. However, the heightened energy content of the torrefied wood dust biomass may be attributed to the reduced moisture content of the biomass following torrefaction [53].

Table 3
The High Heating Value (Energy Value) of the Raw (Untorrefied), Torrefied, Hydrolysed and Hydrolysed-torrefied Wood Dust of *Lophira alata*

Sample	Energy (MJ/Kg)
RIWD	17.315
TIWD 200	18.740
TIWD 250	19.776
TIWD 300	19.921
HIWD	16.390
HTIWD 200	18.497
HTIWD 250	19.508
HTIWD 283	22.721

The Effect of High Heating Value of Hydrolysed and Hydrolysed-torrefied wood dust biomass

The result (Table 3) clearly shows the effect of hydrolysis process on the wood dust biomass. Generally, the energy value of the hydrolysed-torrefied wood dust biomass (*Lophira alata*) sample was comparatively higher than its hydrolysed-untorrefied counterpart. From Table 4, this shows the energy value of 16.390 MJ/kg recorded for the hydrolysed-untorrefied sample of *Lophira alata* remarkably increased to 22.721 MJ/kg upon torrefaction of the hydrolysed sample at 283 ºC process temperature. This observation confirmed the efficiency of the hydrolysis pretreatment before torrefying the biomass. The goal of the pretreatment process is to break down the lignin structure and disrupt the crystalline structure of cellulose, so that the acids can easily access and hydrolyze the cellulose [55].

Table 4
The Effect Variation in Temperature on the Energy Value of Hydrolysed-torrefied *Lophira alata*

Sample	Energy MJ/Kg
HIWD	16.390
HTIWD 200	18.497
HTIWD 250	19.508
HTIWD 283	22.721

The Effect of Hydrolysis of Process Temperature on HHV and Energy Yield of the Torrefied and Hydrolysed-torrefied Sample

The influence of process temperature on the heating value and energy yield of torrefied and hydrolyzed-torrefied samples is summarized in Table 4. With the increase in process temperature, HHV increased proportionally, leading to an energy yield increase. The increment of energy value on hydrolysis could be due to the high moisture content loss at high reaction temperature. Once the biomass was torrefied, carbonization further decreased the moisture level of the biomass to increase the HHV of the previously mentioned raw and dry feedstock, and that means better energy density [56]. Torrefaction at high temperatures also increased the carbon content of the biomass by reducing its moisture. Lower moisture results in higher carbon (carbon content) and leads to a higher HHV [57]. Accordingly, at higher torrefaction temperatures, the moisture content was reduced and carbon content and HHV increased [58]. The energy yield, which is the percentage of the enthalpy after torrefaction from initial biomass enthalpy (i.e., HHV) [59], increased to 57.55%, while the HHV value improved by 41.40% at a hydrolyzed-torrefied process temperature of 283°C. This behavior corroborates other literature reports [53] that recorded energy yields between 50 and 79% for biochar produced under serious torrefaction severity conditions. The enhanced energy yield and energy density can be ascribed to the solid weight loss during hydrolysis and torrefaction. This weight loss (decreased porosity) will further increase the concentration of energy-dense species in the biomass [60]. The mass yield for the thermal treatment of lignocellulosic biomass is usually low, as a significant proportion of the resulting products are water, acetic acid, and sugars (which release during processing), and condensable gases such as C1-C3 hydrocarbons, benzene, etc., leading to weight loss by about 60–80% [59, 61]. Water as the major condensable product is generated mainly by dehydration and evaporation reactions during pyrolysis, which account for much of the biomass weight loss [62]. Solid yield is also an important parameter to determine the efficiency of torrefaction, which denotes how much material and energy is conserved after torrefaction [63]. As shown in Table 4, increasing the processing temperature of raw biomass resulted in a decrease in solid yield (49.00% at 300°C). Furthermore, for hydrolyzed-torrefied biomass,

the solid yield decreases to around 41.40% during severe torrefaction conditions (300°C). This decrease is primarily caused by devolatilization, dehydration, depolymerization, and evaporation reactions in the thermal treatment. Both HHV and solid yield were used to estimate energy yields. The energy density is usually represented by the energy enhancement factor. From Table 5, it can be seen that raw torrefied biomass had an energy enhancement factor of 1.15, whereas hydrolyzed-torrefied biomass has a higher energy enrichment factor of up to 1.39. A negative correlation between solid yield, energy yield, and energy density and initial biomass moisture content has been reported in previous studies [64]. Moreover, energy output largely depends on the original density and form of biomass [65]. The energy generation and the relevant parameters were estimated by Equations (1), (2), and (3) [63].

Ultimate Analysis of Hydrolysed-torrefied, Hydrolysed, Torrefied and Untorrefied Samples

Ultimate Analysis of Untorrefied and Torrefied Sample

Table 5 shows the ultimate composition of raw (Untorrefied), torrefied, hydrolyzed and hydrolysed-torrefied samples*Lophira alata* wood dust biomass. The elemental composition of the torrefied wood dust at 300 ℃ shows that the higher the HHV, the higher the carbon content of the biomass. Torrefied wood dust biomass at 300 ℃ sample with the highest amount of HHV (Table 4) has highest content of carbon amounted to 75.36% as well as lowest percentage of oxygen (4.53%) and hydrogen content (0.32%). However, the hydrogen and oxygen content of a biomass can be attributed to the amount of water and lower molecular weight components present in a biomass, whereas, high amount of water content has an advert effect on the combustion property of a biomass. This elemental composition results are consistent with the results of [3, 66]. The little variation in elemental composition of the raw samples may be due to different species of wood dusts biomass samples used for the study. Notwithstanding, the carbon, hydrogen, nitrogen, oxygen and sulfur content present in the samples were found at level range from 40–50%, 6–7%, 0.1–0.5%, 40–50% and 0.01–0.10% respectively [67]. Raw wood dust biomass sample has the amount of HHV of about 55.83% has lowest amount of carbon content as well as highest percentage of hydrogen (2.45%) and oxygen content (21.58%). This implies that high carbon content with little oxygen and hydrogen content leads to improvement in quality of a biomass as fuel.

The result of sulphur content in a raw wood dust biomass is very negligible (less than 0.01%). Furthermore, an increase in carbon content of a biomass lead to an increase in calorific value (HHV) which is as a result of lower moisture content, a decrease in the amount of water (moisture content) in a biomass also result in lower O/C and H/C ratio of the biomass, as reported by [62, 68]. A lower H/C and O/C ratio of a biomass results in smokeless, less energy loss and lower water vapor formation during combustion process. However, it can be seen in Table 4 that biomass with the highest calorific value (HHV) and carbon content have lowest H/C and O/C ratio and vice versa.

The Effect of Hydrolysis on Elemental Composition of Sample

Table 5 presents the findings of the ultimate analysis conducted on wood dust biomass subjected to hydrolysis and hydrolysis followed by torrefaction, sourced from raw wood dust of the *Lophira alata* tree. The carbon content of the hydrolyzed wood dust biomass sample, initially at 56.07%, notably increased to 81.31% after torrefaction at a temperature of 300 ºC. Conversely, the hydrogen content of the hydrolyzed-torrefied sample decreased to 0.31% post-torrefaction, down from 6.09% in its hydrolyzed state. A similar trend was observed in the oxygen content, with the biomass sample's oxygen content decreasing from 32.78% in its hydrolyzed state to 8.20% after torrefaction at 300 ºC. As depicted in the table, the sulfur content of the torrefied-hydrolyzed sample increased to 1.92% compared to 0.00% in its hydrolyzed state. However, the nitrogen content, initially at 0.137% in its hydrolyzed form, significantly rose to 0.412% following torrefaction at 300 ºC. This trend suggests that the torrefaction process enhances the biomass's carbon content while reducing the levels of oxygen and hydrogen. The implication of this is that torrefaction, as a treatment method, effectively boosts the higher heating value of the biomass by augmenting its carbon content. Additionally, the heightened carbon content validates the biomass's high energy value, thereby greatly improving its fuel properties [69].

Table 5
Ultimate result of raw, torrefied, hydrolyzed and hydrolyzed-torrefied wood dust biomass

Sample	%C	%S	%H	%N	%O H/C O/C
RIWD	55.83	0.00	2.45	0.149	21.58 0.043 0.386
TIWD 300^{O}C	75.36	1.36	0.32	0.380	4.53 0.004 0.060
HIWD	56.07	0.00	6.09	0.412	32.78 0.109 0.585
HTIWD 283^{O}C	81.31	1.92	0.31	0.137	8.20 0.003 0.100

Where RIWD means Raw Iron Wood Dust, TIWD 300 ^{O}C means Torrefied Iron Wood Dust at 300 ^{O}C, HIWD means Hydrolysed Iron Wood Dust, HTIWD 283 ^{O}C means Hydrolysed-torrefied Iron Wood Dust 283 ^{O}C.

Surface morphology observation

The SEM images of untorrefied (raw), hydrolysed, torrefied and hydrolysed-torrefied samples of *Lophira alata* at different temperature values are presented in Figs. 2. There are two SEM images with different magnifications (X1000 and X2000) for all the samples.

Morphological characteristics of Raw (untreated), Torrefied, Hydrolysed and Hydrolysed-torrefied Wood Dust Samples

Results of scanning electron microscopy (SEM) analysis showed that the raw ironwood (Lophira alata) had a well-compact shape and rigid structure with a large grain size compared to an enhanced surface area. The microparticles had a rough surface texture and were dispersed in a wide range, resulting in visible pore openings. In contrast, the torrefied samples shown in Figures 2(a–e) exhibited a more disordered morphology with a porous structure and less fibrousness. These findings show that the torrefaction would lose the structural characteristics and density of biomass cell wall components. At the torrefaction temperature of 200°C, there were no remarkable morphological changes in the surfaces of raw LA biomass. However, when torrefaction temperature was raised to 250°C, micropores decreased and compactness decreased as well, and hollow circular ducts appeared. This data is in good agreement with the discovery reported by [70, 71]: hemicellulose and cellulose start to decompose as the temperature of torrefaction gets near 250°C. At 300°C a relatively high reduction in microparticle architecture along with many hollow circular ducts was visible, confirming the promotion of wax degradation. Based on [72], increasing torrefaction temperature exacerbates the thermopretreatment effect, and that will cause more damage and provide a high potential for smaller biomass particles. The LA biomass underwent large chemical and structural changes after acid pretreatment with H_2SO_4. The SEM images of acid-hydrolyzed LA showed a relatively more porous surface morphology than that of the dense-packed structure observed from raw biomass. Raw LA showed a dense and stiff matrix; however, pretreated materials presented partial breakdown of the structure of cellulose, probably through some pores with different sizes. Furthermore, the lignin–hemicellulose network in the acid-pretreated LA was dramatically destroyed and presented uneven and loose surfaces. Generally, the lignin and parts of hemicellulose can be removed through breaking the cell wall structure by H_2SO_4 pretreatment for biomass to improve digestibility according to the acid concentration. At 300°C, the acid-hydrolyzed LA became more and more loosely packed as microparticles dwindled. Furthermore, the enhancement of heating value due to torrefaction is attributed to dehydration, devolatilization, and depolymerization reactions, and it has been widely reported. Thus, analysis of surface morphology is solid proof that torrefaction pretreatment improves biomass grindability, porosity, and energy content.

Fourier-Transform Infrared Spectrometric Analysis (FTIR)

The FTIR was implemented in the study of chemical structure variation of raw (torrefied and hydrolyzed) Lophira alata wood dust at different torrefaction temperature values of 200, 250, and 300°C. Figures (3–7) show the FTIR spectra of the specimens. The main functional groups were assigned according to the reflectance bands. The band labelling of the spectra is given as in biomass torrefaction studies [73]. The FTIR spectra of the original, treated, and torrefied biomass with peaks corresponding to important functional groups did not change. The absorption peak (O–H stretch peak) [3680 – 3200 cm^{-1}] is observed in all the samples. [74] reported that the hydroxyl group in all biomass feedstocks (both raw and hydrolyzed) can be attributed to the presence of approximately 1% moisture, and they further concluded that at temperatures above 300°C, the hydroxyl group (-OH) on biomass is destroyed, resulting in a loss of the ability to form hydrogen bonds with water. Thus, torrefaction enables a reduction in moisture content of the biomass feedstock and hydrophobes the torrefied biomass. The

bands at 3300 – 2850 cm^{-1} are due to aliphatic (C–H stretching) in raw and hydrolyzed samples of developed film. The bands of around 1705 and 1473 cm^{-1}, observed for the raw and acid-hydrolyzed LA, are attributed to carbonyl groups (conjugated C = O stretching) [53, 74]. The higher intensity C–O-bond peak of torrefied samples for raw and acid LA might be a result of the degradation of carbohydrate and, thus, the relative augmentation in carbonyl or carboxyl groups of the lignin contained by biomass [75]. The absorption band at 1435 cm^{-1} of acid-hydrolyzed LA, even though weak in intensity (Table 1), could be assigned to aromatic skeletal vibration and indicates an increase in the proportion of aromatic fraction during thermal modification. The observed bands at 1430–1485 cm^{-1} (assigned) and 1030–1120 cm^{-1} (observed due to C–O stretching of cellulose and hemicelluloses) [74]. Bands from 1000 cm^{-1} to 750 cm^{-1} in the raw sample indicate aromatic deformation of C–H only, and bands from 1419 cm^{-1} to 1435 cm^{-1} show C–H deformation (methyl and methylene).

Thermo-Gravimetric Analysis (TGA)

Using a thermo-gravimetric analyzer, the biomass sample of untorrefied, torrefied and hydrolysed-torrefied wood dust biomass was thermally degraded at a rate of 20°C per minute. The temperature range at which the analysis was conducted was 27 ^{o}C to 884 ^{o}C. The mass loss during the pyrolysis of *Lophira alata* wood dust, both untorrefied, torrefied and hydrolysed are illustrated in Figs. 8 and 10. Three distinct stages of thermal degradation, dehydration, devolatilization, and char formation were identified in the untorrefied sample. The weight percentage (wt%) of the raw, *Lophira alata* declined to 97% at a temperature of 104 ^{o}C, whereas the sample that had been thermally pre-treated (torrefied) decreased to 99%. It appears that the torrefied sample's fractional mass change at this stage is less than the untorrefied sample's. Furthermore, the evaporation of the sample's water content is responsible for the dehydration that was noticed at this point [75]. Because the sample had been pretreated beforehand, a relatively smaller amount of moisture evaporated in the torrefied sample. Devolatilization took place between 200 ^{o}C and 450 ^{o}C. It was at this temperature that more volatile materials were lost from the torrefied *Lophira alata*, which led to a weight reduction of 17% at 400 ºC, and hydrolysed-torrefied *Lophira alata* (HTWID), which also saw a weight reduction of 18% at 470 ^{o}C. In contrast, the torrefied sample released less volatile materials at the same temperature [75]. This resulted from the torrefied biomass's previous devolatilization during torrefaction. The weight of the torrefied *Lophira alata* (raw) decreased to 8% at 500 ºC, but the weight of the hydrolysed sample decreased to 25%. The formation of char, which was the carbonization stage, happened at a temperature over 450 ºC. The torrefied sample was more thermally stable than the untorrefied counterpart, according to this study. The thermal degradation of Raw torrefied and Hydrolysed-torrefied can be inferred from the TGA curves shown in 113 Figs. 8 and 10. This indicates that the process starts with drying or dehydration and continues through virtually chemical freezing, mild pyrolysis, severe pyrolysis, and carbonization. This result is in line with studies on the heat stability of maize stalks published by [76] and [77].

CONCLUSION

The torrefaction temperature was observed to greatly affect the fuel characteristics of Lophira alata wood. The increase in higher heating value (HHV) was found based on process temperature. On the other hand, solid yield decreased with rising torrefaction temperatures because of continuous weight loss during the thermal conversion of biomass. The decreased contents of moisture and volatile matter proved the efficiency of the proposed hydrolysis–torrefaction treatment. These modifications resulted in the increased HHV, making the biomass a solid fuel. The increase of the fixed carbon content in hydrolyzed-torrefied biomass also shows that the hydrolysis pretreatment significantly improved the energy hydrolysis value of the biomass. In addition, processing the hydrolyzed-torrefied energy product at 283°C led to a significant decrease in hydrogen and oxygen contents, as indicated by the lower H/C and O/C atomic ratios. These showed that the quality of the fuel and energy densification potential of Lophira alata solidless has been enriched. The results also indicate that the increase of process temperature resulted in the elevation of relative lignin content of hydrolyzed-torrefied biomass, thereby improving its binding properties and potential for pelletization. It stems from the thermal stability series of components in biomass fiber: hemicellulose < cellulose < lignin. Furthermore, biomass became brittle and fragile at the optimum process temperature of 283°C, leading to its superior grindability caused by depolymerization. The low EMC acquired at this temperature is an indication that the pelletized hydrolyzed-torrefied Lophira alata biomass can be stored for a prolonged time without excessive moisture reabsorption due to superior hydrophobicity. This characteristic is also beneficial for the handling and transportation of the biocoal produced. TGA analysis indicated that hydrolyzed-torrefied samples had increased thermal stability compared with torrefied-only and raw biomass according to the lower rates of mass loss in ignition. FTIR spectra also showed the degradation of hydroxyl and methoxyl groups during both the hydrolysis and 283°C torrefaction, which resulted in decreased oxygen and hydrogen levels that subsequently led to better fuel quality. In summary, hydrolysis-torrefaction using a process temperature of 283°C was found to be the optimal condition for Lophira alata wood dust biomass. At this temperature, the energy value and chemical components of the biomass were greatly improved, indicating that it is a feasible feedstock for the production of bio-coal. In summary, the torrefaction process is an appropriate thermochemical conversion technology for the sustainable utilization of wood dust waste with the potential to obtain value-added energy products.

Declarations

Ethical approval

This study did not include human subjects or human data or material, and no live vertebrate animals were used. As such, ethical approval and consent were not required..

Consent to participate

Not applicable.

Consent to publish (Ethical)

Not applicable.

Author Contribution

I, Agamu Francisca Obiajulu, on behalf of all co-authors, hereby give our full consent for the publication of our manuscript titled: Impact of hydrolysis on thermal stability and binding capacity of torrefied-densified iron (*lophira alata*) wood dust Manuscript.We confirm that:1. All authors have read and approved the final version of the manuscript.2. The manuscript is an original work and has not been published elsewhere, nor is it under consideration by any other journal.3. All authors agree to transfer or grant the necessary rights to Journal of Discovery Chemistry by Springer Nature for the purposes of publication, distribution, and archiving, in accordance with the journal's policies.4. All authors agree to comply with Journal of Discovery Chemistry by Springer Nature's ethical and publication guidelines.5. The authors confirm that there is no conflict of interest, except as stated in the manuscript.1. The authors give consent for any editorial changes required for clarity and quality without affecting the scientific content.Corresponding Author Information:1.Agamu Francisca Obiajulu,Department of Chemistry,FUTA,Nigeriaobiajuluagams77@gmail.com+2348138817717Co-Authors:**Authors:**2. Ogunsuyi Helen Olayinka, Department of Chemistry, Federal University of Technology, Akure, Ondo State Nigeria. Email: hoogunsuyi@futa.edu.ng3. Jabar Jamiu Mosebolatan, Department of Chemistry, Federal University of Technology, Akure, Ondo State Nigeria. Email: jmjabar@futa.edu.ng4. Raheemat Oyiza Muhammed, Department of Botany, Bayero University Kanu, Kanu State Nigeria. Email: raheemahoyiza96@gmail.comAgamu Francisca Obiajulu

Data Availability

All data generated or analysed during this study are included in this published article and its supplementay information files.

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Figures

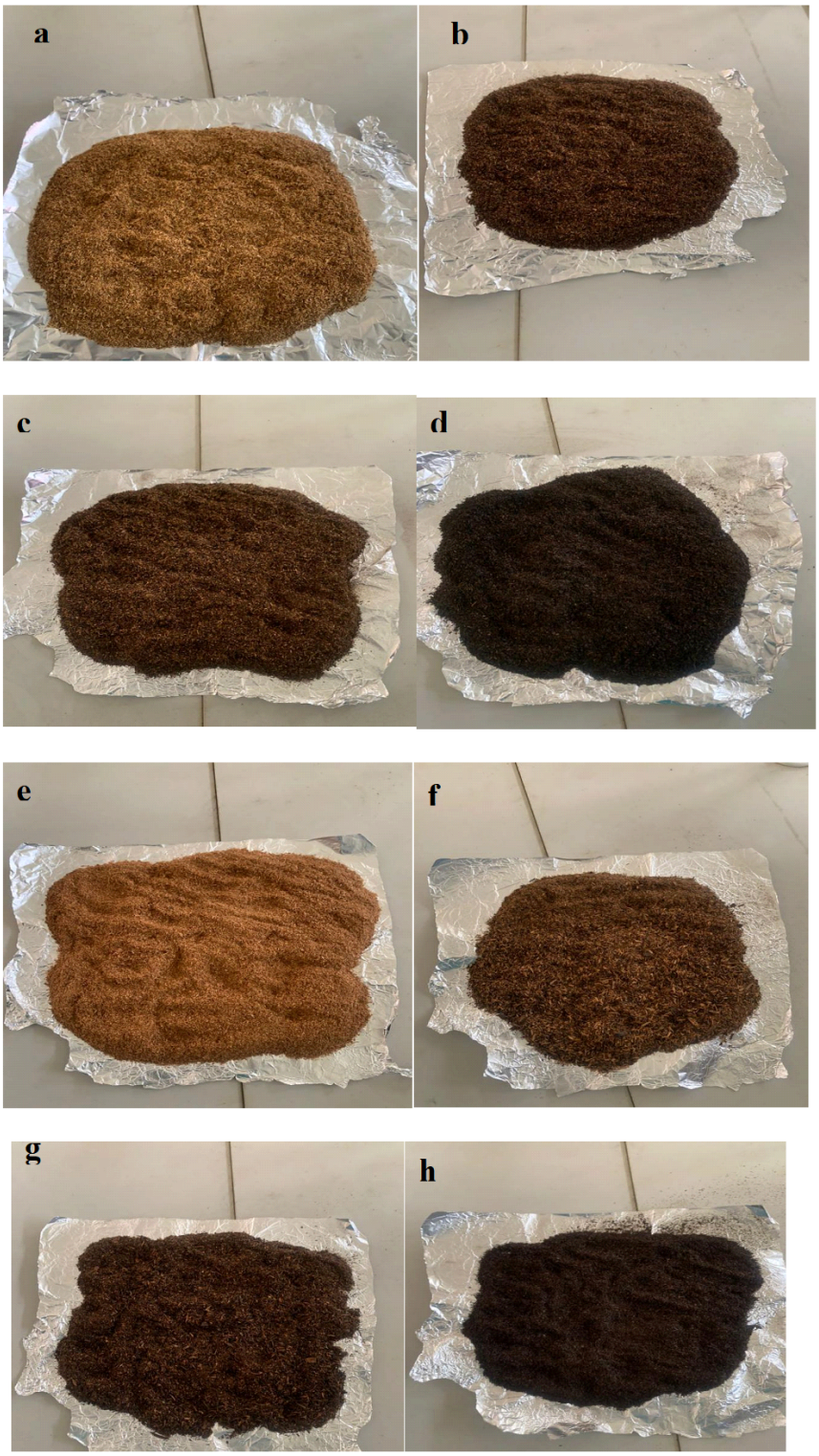

Figure 1

Pictures of: (**a**) Raw Iron Wood Dust, (**b**) Torrefied Iron Wood Dust at 200 °C, (**c**) Torrefied Iron Wood Dust at 250 °C (**d)**Torrefied Iron Wood Dust at 300 °C (e) Hydrolysed Iron Wood Dust(f) HydrolysedTorrefied Iron Wood Dust at 200 °C (g) Hydrolysed Torrefied Iron Wood Dust at 250 °C (h) Hydrolysed Torrefied Iron Wood Dust at 283 °C.

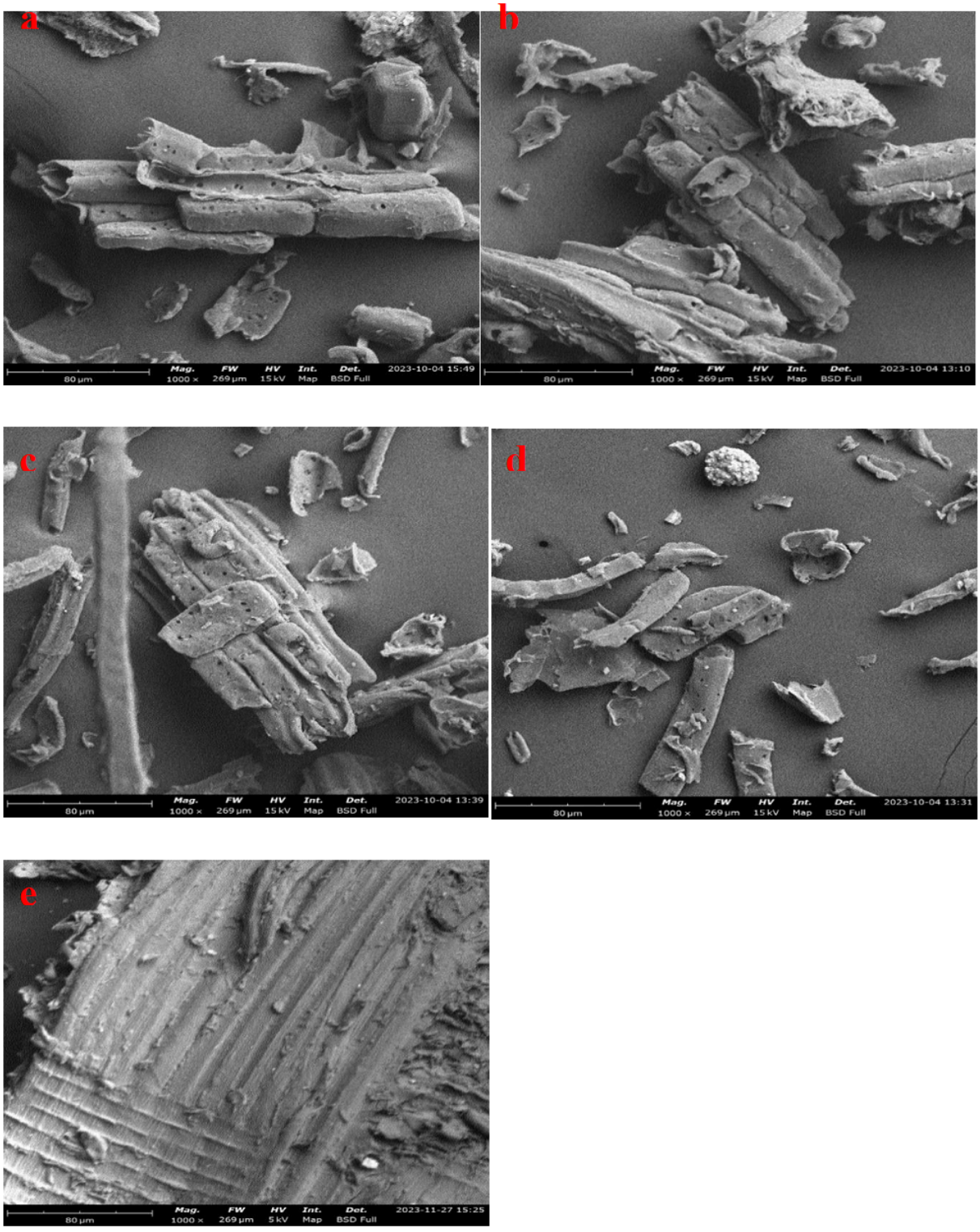

Figure 2

Surface morphologies of (**a**) Untorrefied Wood Dust Biomass of *Lophira alata*, (**b**) Torrefied Wood Dust Biomass at 200°C, (c) Torrefied Wood Dust Biomass at 250°C, (d) Torrefied Wood Dust Biomass at 300°C, (e) Hydrolysed-Torrefied Wood Dust Biomass at 283 °C

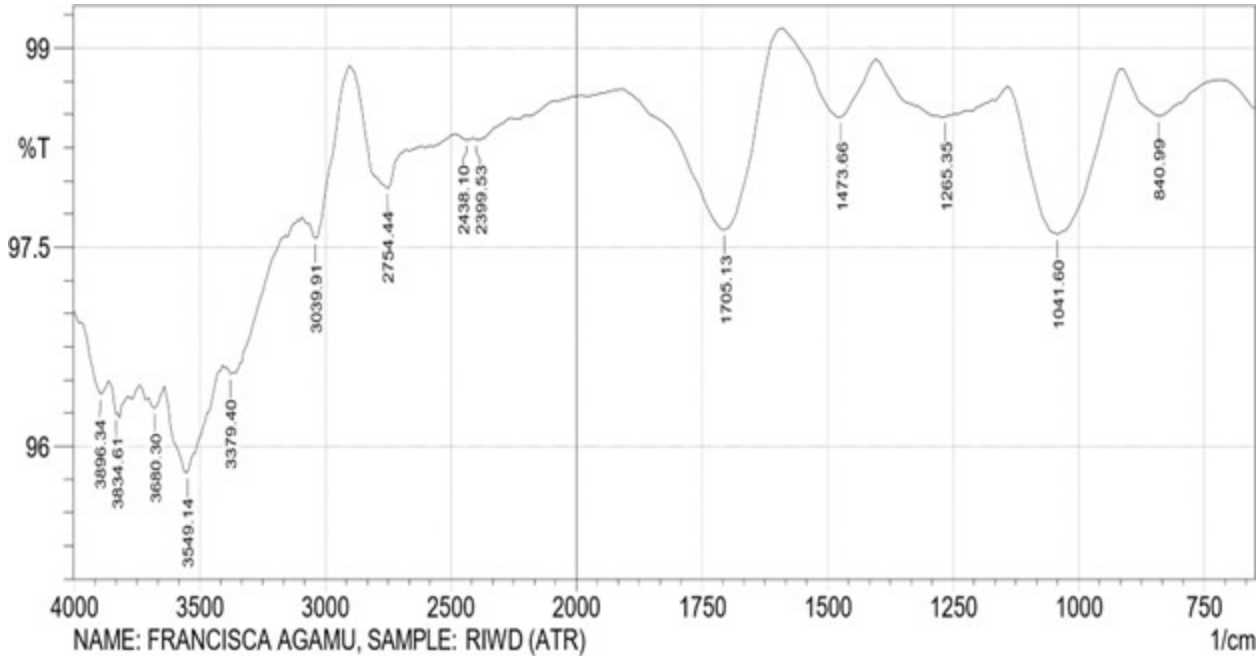

Figure 3

The FTIR Spectra of untorrefied (raw) wood dust sample sourced from *Lophira alata* (LA)

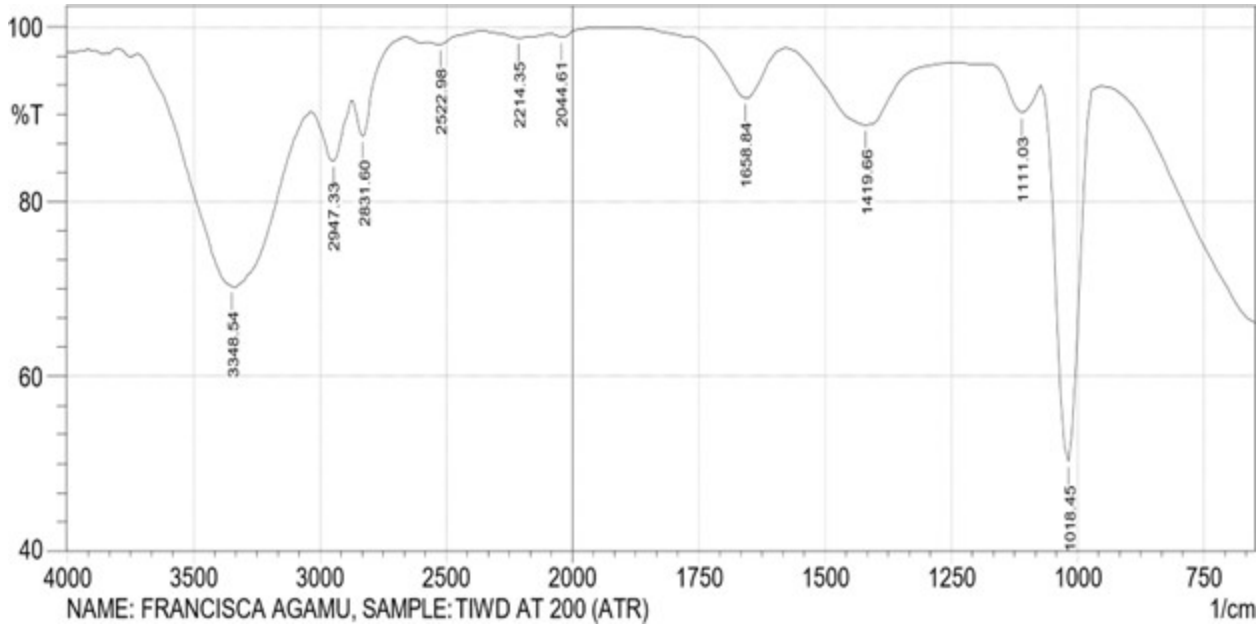

Figure 4

The FTIR Spectra of torrefied at 200 ℃ wood dust sample sourced from *Lophira alata* (LA)

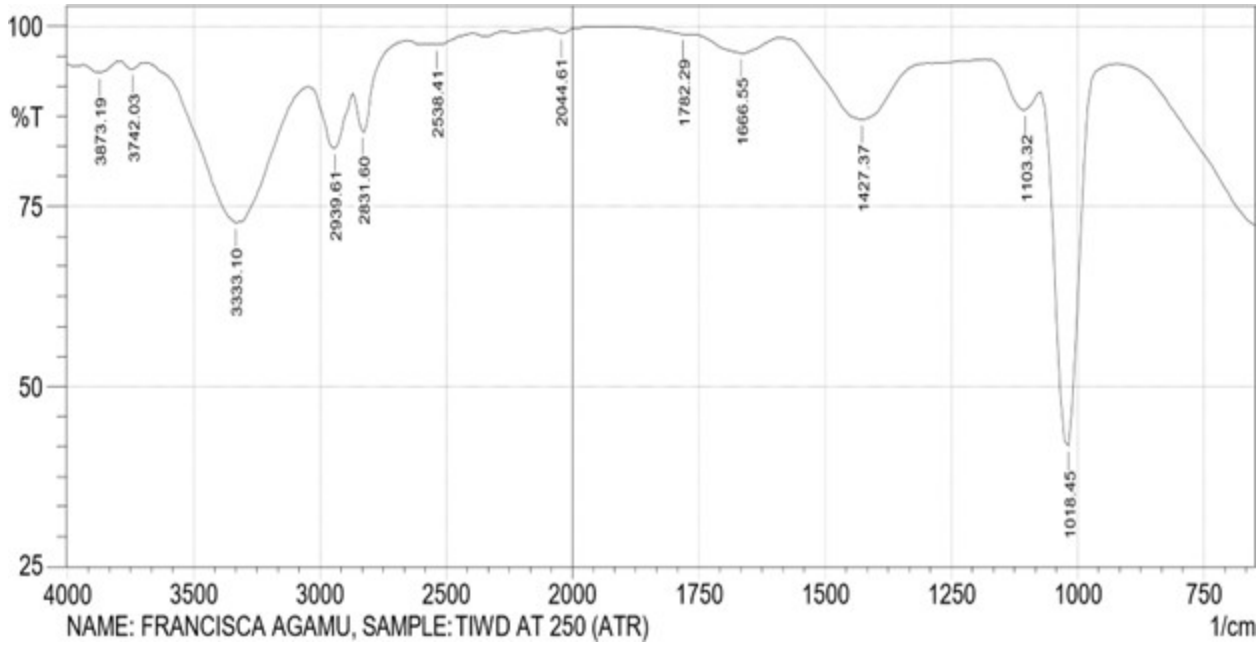

Figure 5

The FTIR Spectra of torrefied at 250 ℃ wood dust sample sourced from *Lophira alata* (LA)

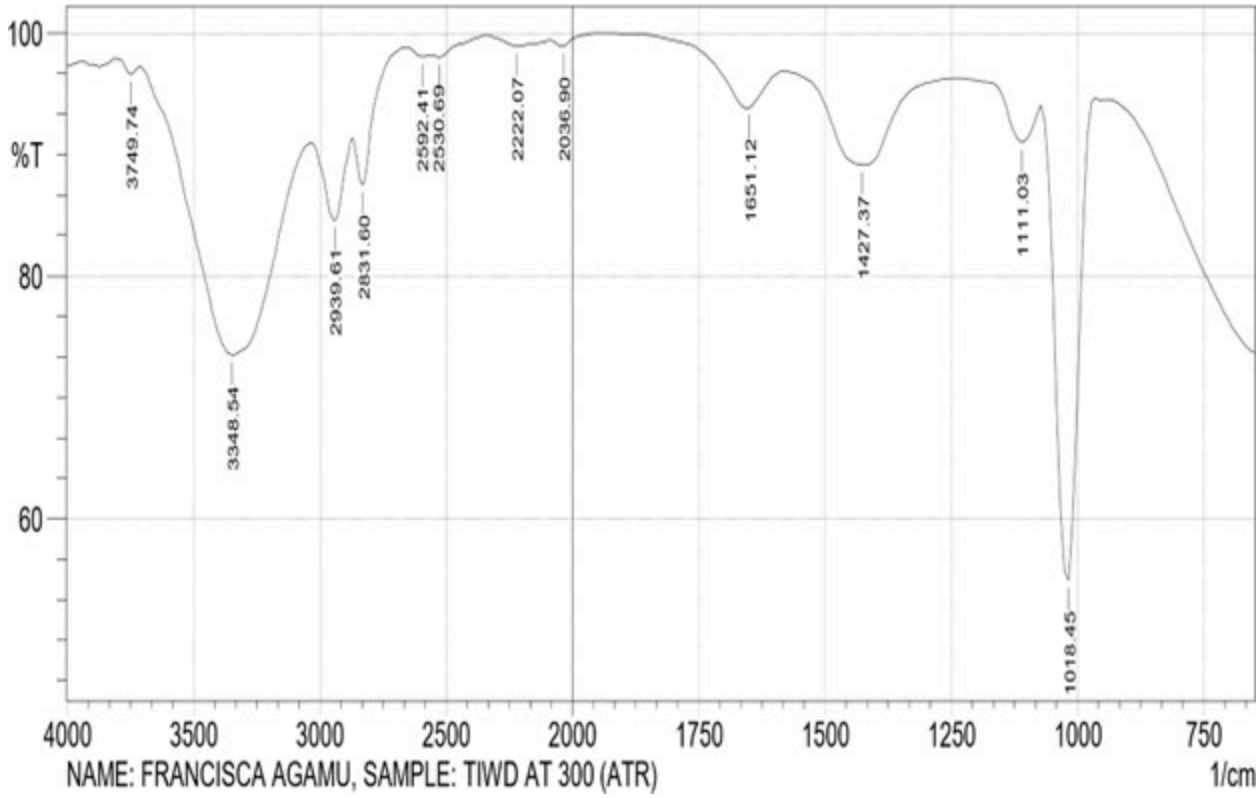

Figure 6

The FTIR Spectra of torrefied at 300 ℃ wood dust sample sourced from *Lophira alata* (LA)

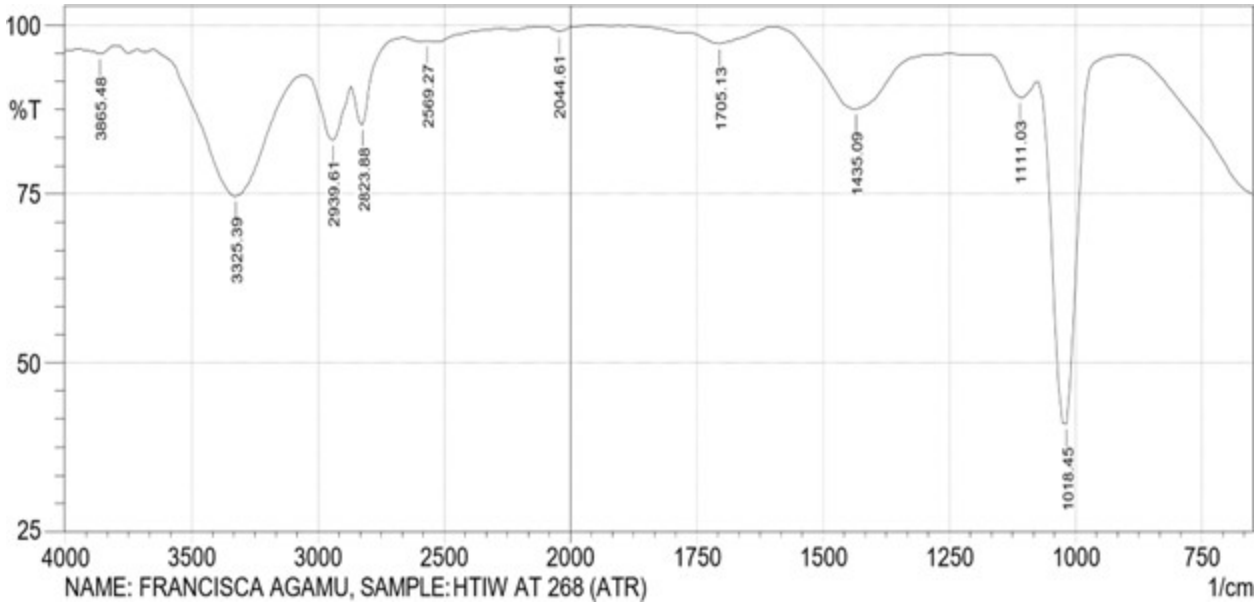

Figure 7

The FTIR Spectra of hydrolysed-torrefied wood dust sample sourced from *Lophira alata* (LA)

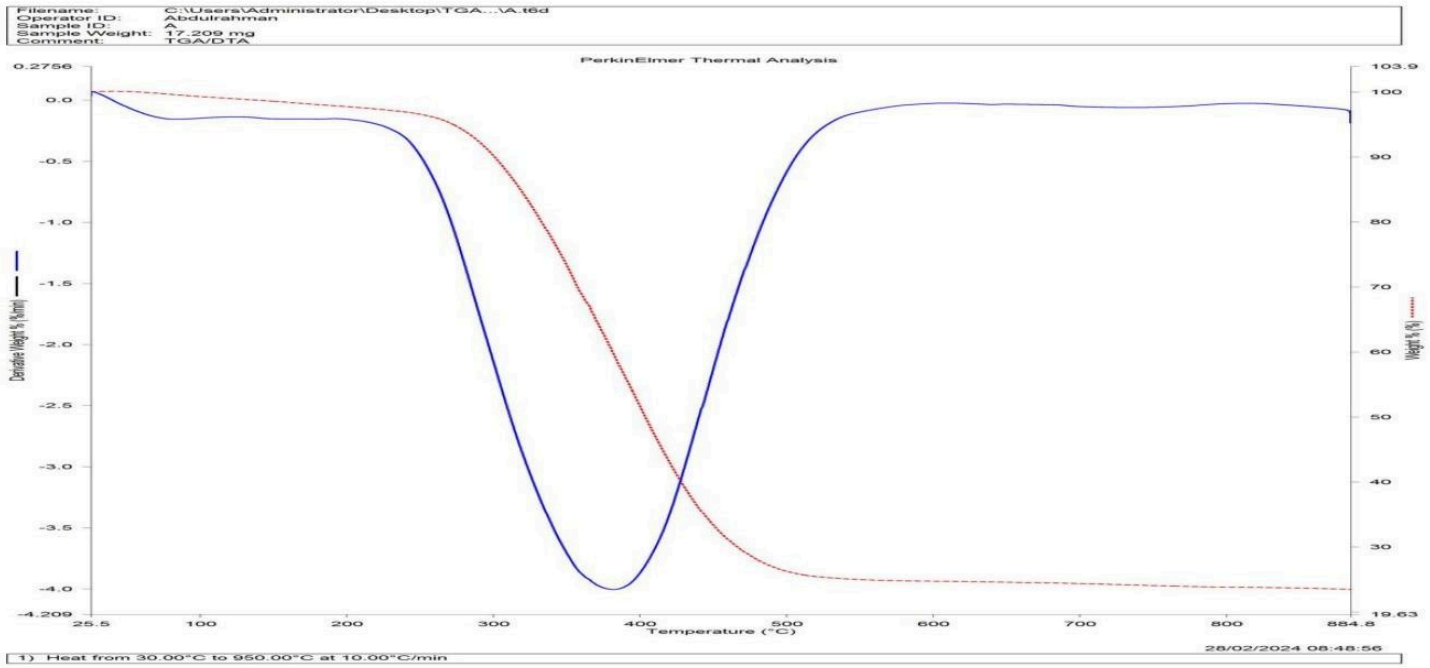

Figure 8

The Thermogram of Untorrefied Wood Dust Sample of Lophira alata tree

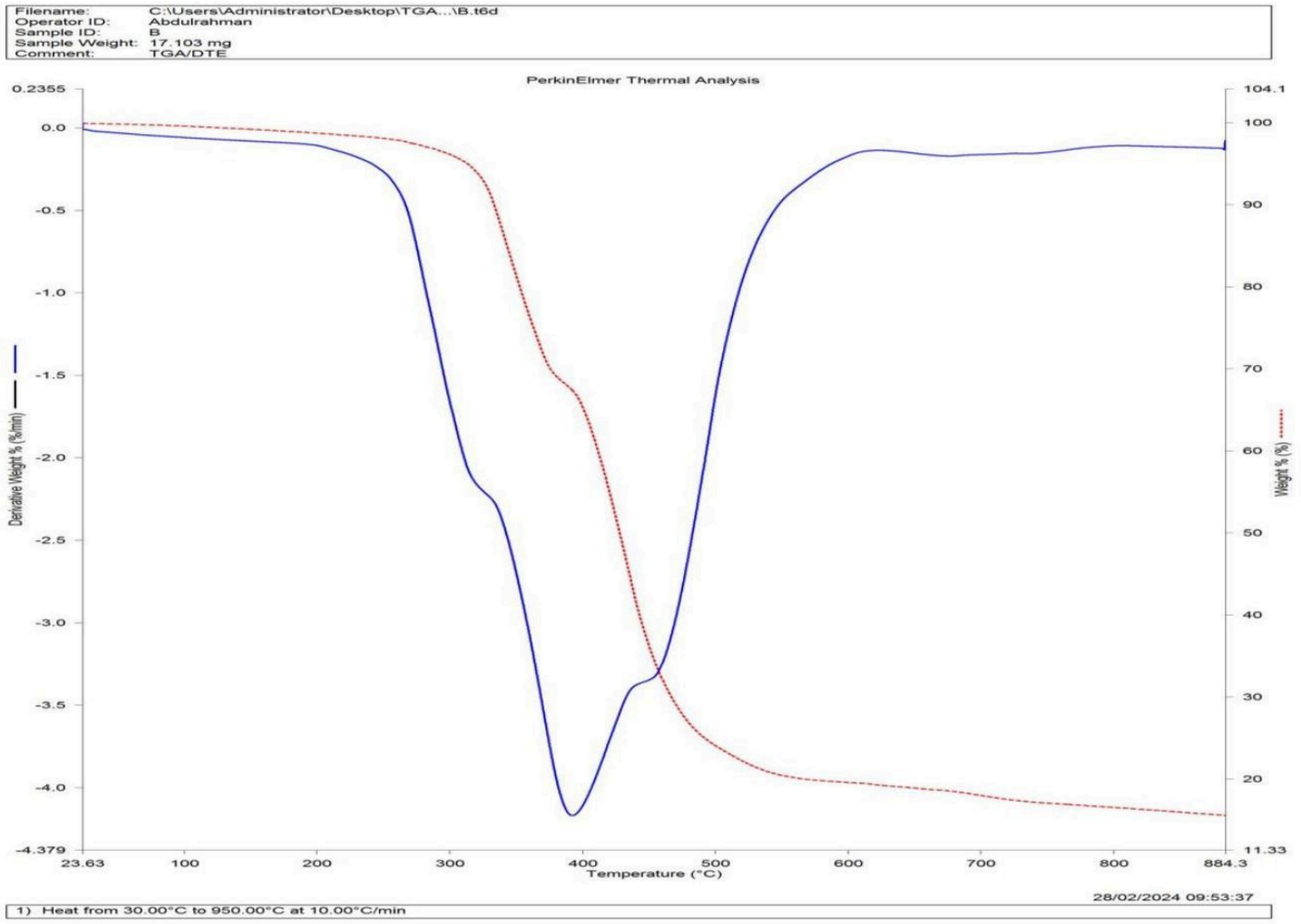

Figure 9

The Thermogram of Torrefied Wood Dust Sample at 300 ℃ of Lophira alata tree

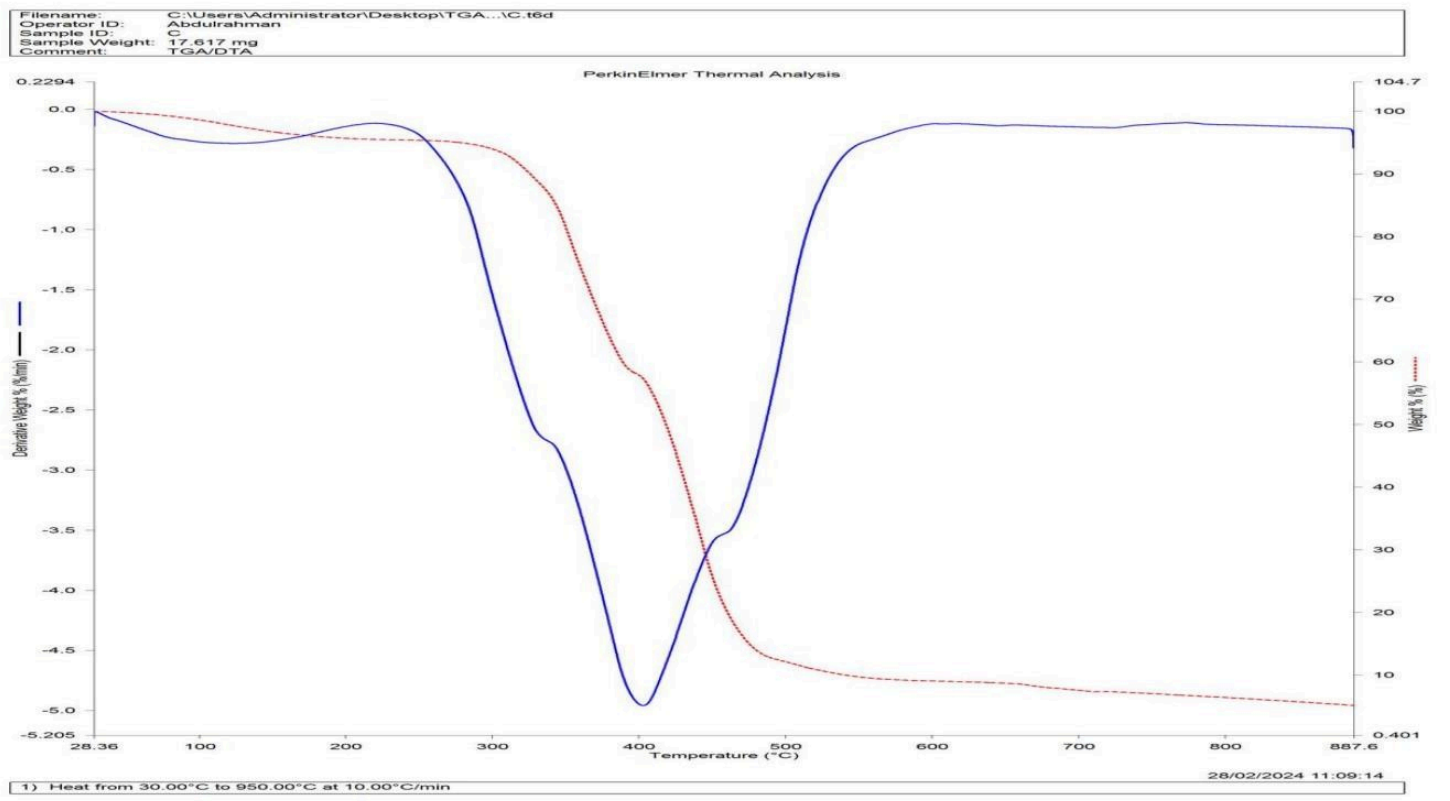

Figure 10

The Thermogram of Hydrolysed-torrefied Wood Dust Sample at 300 ℃ of Lophira alata tree

Supplementary Files

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