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# Characteristics of Torrefied Woody Dust of *Terminalia superba*

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**ABSTRACT**

This research aimed to assess the effects of torrefaction on the energy value and chemical composition of wood dust of *Terminalia superba* (Afara). Physico-chemical analyses of the raw biomass were performed prior to torrefaction. Torrefaction was carried out in a laboratory-scale torrefier at 200 °C, 250 °C, and 300 °C. The energy content of *Terminalia superba* was initially 17.51 MJ/kg. Torrefaction increased the higher heating value (HHV), with the highest HHV of 27.70 MJ/kg observed at 300 °C, compared to 24.58 MJ/kg at 250 °C and 21.22 MJ/kg at 200 °C. At 300 °C, the fixed carbon content rose from 19.94% to 63.01%, while volatile matter decreased from 58.80% to 17.71%. The study also analyzed fiber composition, revealing a direct correlation between chemical structure and thermal stability, with hemicellulose < cellulose < lignin. Results were visualized in a Van Krevelen diagram, showing improved hydrophobicity and fuel characteristics due to moisture reduction. In conclusion, torrefaction significantly improved the energy value and fuel properties of *Terminalia superba.* Further research is recommended to optimize torrefaction conditions for other biomass species.

**Keywords:** Physico-chemical analyses, *Terminalia superba.* torrefaction, wood dust

## INTRODUCTION

In 2021, 71 percent of the global population had access to clean cooking fuels and technologies, an increase of 14 points since 2010. In spite of the progress, some 2.3 billion people still use polluting fuels and technologies for most of their cooking [1]. According to the Tracking SDG 7: The Energy Progress Report 2023, almost all of the 20 countries with the lowest rates of access to clean cooking, with the exception of Rwanda, have shown little or no progress in increasing their access rates between 2017 and 2021 (with an increase of less than 0.4 percentage points). Nineteen of these countries are in Africa, with Haiti the only non-African country among them.

Efforts to accelerate the achievement of universal access to clean cooking by 2030 are urgently needed [1].

In rural areas, unprocessed biomass was still the principal cooking fuel of 49 percent of people (1.5 billion) in 2021, more than any other type of fuel. Use of unprocessed biomass may be decreasing in both urban and rural areas, but primary reliance on charcoal persists and is increasing in some areas, particularly urban areas of Sub-Saharan Africa, where it was used by 30 percent of people (140 million) in 2021 [2]. Biomass is an abundant energy source that spans considerable portions of Earth's land surface and has a rapid life cycle. Consequently, pretreatment of biomass is essential to enhance its fuel characteristics before it can be utilized alongside or as a replacement for coal.

Torrefaction, a thermochemical process, is considered a straightforward and efficient technique for modifying biomass properties to closely resemble those of coal [3]. Significant attention has been directed towards torrefaction development in the past decade. This thermochemical conversion technology effectively enhances biomass properties and upgrades solid biomass-derived fuel [4]. Torrefaction yields several outcomes, including: heightened energy density, improved ignition properties, reduced moisture content, elevated carbon-to-oxygen (C/O) and carbon-to-hydrogen (C/H) ratios, enhanced grind-ability leading to reduced energy demand for grinding, hydrophobic biomass that displays decreased affinity for water, greater homogeneity in biomass as torrefaction undertakes devolatilization, depolymerization, and carbonization of the biomass, and suppression of microbial activity [5].

The aim of this research is to investigate the energy potential of *Terminalia superba* (Afara) wood dust for bio-coal production using torrefaction treatment method.

## MATERIALS AND METHODS

Wood dusts biomass samples were collected from Ajegunle sawmill in Akure, Nigeria. Preliminary analyses were conducted on the wood dusts sourced from African limba tree *Terminalia superba* (Afara). The samples were air-dried over a period of four days and then sifted using an analytical sieve set at a size of 450 µm, resulting in a particle size of 450 microns. These sifted samples were appropriately labeled. Initial assessments were subjected to torrefaction at various temperatures (200, 250, and 300 °C) using a torrefaction setup in a laboratory setting, aiming to optimize the temperature conditions. The energy content of the

biomass samples was determined using a bomb calorimeter. The elemental composition of the wood biomass, encompassing carbon, hydrogen, oxygen, nitrogen, and sulfur, was evaluated using the ultimate analysis method outlined by Ogunsuyi *et al* [6]. Their proportions were directly measured through instrument readings. The oxygen content in the biomass was calculated as the difference in composition, using the following formula:

$$[100 - (\%C+\%H+\%N+\%S)] = \%O \quad (1)$$

The moisture content (M.C), ash content, and volatile matters were analyzed according to the procedures outlined in ASTM standards: ASTM D-3173-03 for moisture content, ASTM D-3174 for ash content, and ASTM D-3175 for volatile matters. The fixed carbon (F.C) of the specimens was calculated using a method described by Lateef and Ogunsuyi [7], as specified in equation 2.

$$F.C = [100 - (\%M.C + \%A + \%V.M)] \quad (2)$$

The fiber constituents of the wood dust biomass sample, such as lignin, hemicellulose, alpha-cellulose, and extractives, were quantified. The lignin content in the biomass samples was determined following the guidelines of TAPPI T 222 om-88, as detailed by the Technical Association of the Pulp and Paper Industry (TAPPI), and further elaborated by Lateef and Ogunsuyi [7]. To measure lignin content, a 2.0 g portion of acetone extractive-free biomass was treated with a 15 mL solution of 72% $H_2SO_4$ for 2 hours at room temperature (29 °C) within a 250 mL beaker. This treatment aimed to break down and dissolve the carbohydrates present. The mixture was then diluted with 560 mL of distilled water to reduce the concentration of sulphuric acid and subsequently boiled for 4 hours. After boiling, the solution was allowed to settle, then filtered. The remaining residue was washed with 600 mL of hot water until it reached a neutral pH. The residue was air-dried for 12 hours and then oven-dried at a constant weight of 105 °C for 10 minutes. The dried insoluble residue, which corresponds to the acid-insoluble fraction and signifies the lignin content, was determined using Equation 3.

$$\text{Acid insoluble (\%)} = \frac{y}{w} \times 100 \quad (3)$$

Where, Y stands for the weight of the insoluble lignin (after oven-drying) in grams (g), and w represents the weight of the test sample that has been dried in the oven, also measured in grams (g).

The acetone-extractive-free biomass (weighing 5 g) was subjected to treatment in an acid solution with 160 mL sodium acetate at a temperature of 75 °C for a duration of 5 hours in a 500 mL beaker. Throughout the treatment, 4 mL of sodium chlorite was added to the mixture every hour. After the treatment period, the mixture was cooled. Then, the residue was washed using approximately 800 mL of distilled water and an additional 15 mL of acetone. Then, dried under the existing room temperature conditions of 29 °C. A portion of the residue was measured and subjected to drying at a temperature of 105 °C. This process aimed to determine the holocellulose content within the sample. The calculation for determining the holocellulose content was carried out using Equation 4.

$$\text{Holocellulose content} = \frac{W_2}{W_1} \times 100 \quad (4)$$

Where $w_1$ = weight of oven-dried extractive-free sample (g), and $w_2$ = weight of the holocellulose obtained (g).

To ascertain the α-cellulose content, 5 g of holocellulose was dissolved in a 17.5% weight concentration of NaOH solution, contained within a 250 mL beaker, and left to incubate at a temperature of 29 °C for a period of 30 minutes. After this incubation period, the remaining residue underwent filtration and was subsequently washed twice with approximately 200 mL of distilled water. Once again, filtration was carried out. Following these steps, an additional 15 mL of a solution containing 10% weight of acetic acid was introduced to the residue. Subsequently, the residue was filtered again and subjected to washing with a volume of 500 mL of hot water. The resulting residue was then dried within an oven at a temperature of 105 °C. The quantification of α-cellulose content was achieved through a gravimetric process using Equation 5.

$$\alpha \text{ cellulose content} = \frac{W_1 - W_2}{W_1} \times 100 \quad (5)$$

The torrefaction procedure at the laboratory scale, as outlined by Oyebode and Ogunsuyi [8] was modified and adapted. The torrefaction apparatus comprised of a gas cylinder flame source, an airtight container to hold the biomass sample, a digital thermometer to measure the temperature at which the biomass was torrefied with 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 was joined to a burner and connected to a gas cylinder through

a pipe. Under 45 minutes of residence time, torrefaction studies were conducted at three distinct process temperatures: 200 °C for light torrefaction, 250 °C for medium torrefaction, and 300 °C for severe torrefaction.

The solid yield, enhancement factor and energy yield of the samples were evaluated using Equations (6) to (8), respectively

$$\text{Solid yield (\%)} = \frac{\text{weight of the torrefied biomass collected (g)}}{\textit{weight of the biomass (g)}} \times 100 \quad (6)$$

$$\text{Enhancement factor of HHV} = \frac{\text{HHV of torrefied biomass}}{\textit{HHV of Raw Biomass}} \quad (7)$$

$$\text{Energy yield} = \text{Solid yield (\%)} \times \text{Enhancement factor of HHV} \quad (8)$$

Where HHV represents higher heating value.

**Analytical methods**

The calorific value determination for the samples was conducted utilizing the e2k calorimeter (model: Crl 001). A mass of 0.5 g from the sample was weighed and placed on a 2 cm firing cotton that was secured to the firing wire on the lid assembly. This assembly was then positioned within a metal crucible. Then, the lid assembly was sealed within the bomb (vessel) in an airtight manner. Oxygen was introduced into the bomb, reaching a pressure of 3000 MPa. The equipment was connected to a power source, and the operation was initiated by pressing the F1 key on the e2k Calorimeter's attached keyboard. The bomb calorimeter was ignited after housing the bomb, and the outcome of the combustion process was presented on the screen. Following combustion, the bomb was carefully extracted and degassed. The equipment was closed, and the oxygen cylinder was shut.

Upon the completion of the torrefaction process of the feedstock, the reactor was cooled to a temperature below 80 °C in the presence of an inert environment. This cooling step was implemented to prevent any potential self-ignition or combustion of the solid products. The solid products underwent post-treatment in order to improve their fuel qualities.

The primary method employed for the treatment of torrefied solid product is densification through pelletization and briquetting, with the objective of positioning it as a viable biofuel option with advantages in terms of transportation, storage, and handling. A pelleting mill, also known as a pelletizer, was employed for the purpose of manufacturing torrefied pellets. Due to the relatively low moisture content of torrefied products, a recommended method for

preconditioning involves the addition of hot water to samples in a 1:1 ratio prior to the pelleting process.

Both untreated and torrefied specimens were used to determine the equilibrium moisture content (hydrophobicity test). Consistent relative humidity levels were maintained by employing desiccant (Fisher S161-500 Silica Gel), purified water, and aqueous solutions saturated with KCl and $KNO_3$, as outlined in ASTM standard E104-02 (2012). Each pelleted sample initially weighed 5 g before being positioned in a controlled environment chamber set to specific relative humidity conditions. The weight of each sample in its container was logged every 24 hours. Equilibrium was considered achieved when the weight measurements showed a variance of less than 1 mg in three consecutive measurements.

The morphological visualization of the raw and torrefied biomass was executed through Scanning Electron Microscope (SEM) (Model - JSM-5600 manufactured in Japan). The samples were affixed to SEM stubs using double-sided carbon tape, and were placed into the SEM chamber, which operated at parameters including a 15 kV accelerating voltage, 5–100 μm working distances, and magnifications ranging from 5000 to 8000 times.

Thermal stability of the biomass was measured by thermo-gravimetric analyzer (TGA). The TGA was performed on a TGA8000 PerkinElmer from United States. Evaluation of relevant software, the air flow ratio of the thermal analyzer was under 50 $mL.min^{-1}$, and the temperature increased from the ambient temperature to 1000 °C at the heating rate of 10 $°C.min^{-1}$. Two replicates were tested for each sample.

## RESULTS AND DISCUSSION

### The high heating value of untorrefied (raw) samples

The results of the high heating values (HHV) for the untorrefied wood dust biomass samples are detailed in Table 1. The experiment shows that *Terminalia superba*, has energy values of 17.51 mJ/kg. .

Table 1: High Heating Value (HHV), effect of temperature, and enhancement factor of biomass

| S/N | Botanical Name | HHV Raw (mJ/Kg) | HHV at 200°C (mJ/Kg) | HHV at 250°C (mJ/Kg) | HHV at 300°C (mJ/Kg) | Enhancement Factor | Solid Yield (%) | Energy Yield (%) |
|---|---|---|---|---|---|---|---|---|
| 1 | *Terminalia superba* | 17.51 | 21.22 | 24.58 | 27.70 | 1.55 | 36.14 | 54.21 |

**The effect of torrefaction on the fibre composition of the biomass**

Table 2 details the effects of torrefaction on the fiber content of untorrefied and torrefied wood dust biomass from *Terminalia superba*. The cellulose content in the TS biomass sample decreased from 42.10% to 28.40% as the torrefaction temperature increased to 200 °C, 250 °C, and 300 °C. Initially, there was an increase in cellulose content under mild torrefaction conditions, but a decrease was observed under more severe conditions. This reduction in cellulose content can be attributed to depolymerization and devolatilization processes occurring within the 250-350 °C temperature range [9], which lead to the formation of anhydrous cellulose and levoglucosan [10].

Conversely, there was a significant increase in the lignin content of the biomass samples following torrefaction. The increase in lignin content, occurring as the temperature rose from 200 °C to 300 °C, suggests that lignin's depolymerization, devolatilization, and softening start around 290-500 °C. However, the study found that the optimal temperature of 300 °C did not notably affect the lignin content [11]. Specifically, the lignin concentration in TS rose from 22.50% to 65.30%.

Hemicellulose content significantly decomposed during torrefaction, likely due to its relatively low thermal stability, making it the first chemical component to break down. In the untorrefied TS samples, hemicellulose content decreased substantially from 26.40% to 3.50% at a high torrefaction temperature of 300 °C. The thermal degradation temperature range for hemicellulose is reported to be between 130 and 260 °C. A limited amount of extractive compounds was found in the thermally treated material.

Table 2: Variation of process temperature on fibre composition of *Terminalia superba*

| Sample | Cellulose | Lignin | Hemicellulose | Extractive | Holocellulose |
|---|---|---|---|---|---|
| Raw TS | 42.10 | 22.50 | 26.40 | 09.00 | 66.50 |
| 200 °C | 37.90 | 37.70 | 18.30 | 06.10 | 56.20 |
| 250 °C | 41.20 | 49.60 | 05.80 | 03.40 | 47.00 |
| 300 °C | 28.40 | 65.30 | 03.50 | 02.80 | 31.90 |

**The effect of torrefaction on proximate composition of the torrefied and untorrefied wood sample**

The results of the proximate analysis for both the untorrefied and torrefied samples of *Terminalia superba* wood dust biomass are shown in Table 3. The torrefied sample displayed a notable reduction in moisture content compared to the untorrefied sample. Initially, the moisture content of the unprocessed *Terminalia superba* wood dust was 21.16%. After torrefaction at 300 °C, this moisture content significantly decreased to 15.00%. Moisture content in biomass greatly influences its fuel quality, with lower moisture content resulting in a higher energy value, specifically a higher heating value (HHV).

The initial volatile matter content of the unprocessed *Terminalia superba* wood dust biomass was 59.80%, which significantly dropped to 17.71% after torrefaction at 300°C. This indicates that the torrefaction process was effective in enhancing the biomass's energy content. Additionally, the ash content of the untorrefied *Terminalia superba* wood dust was 2.81%. Upon torrefaction at 300°C, the ash content increased to 4.28%. The data also revealed that the untorrefied wood dust sample had a fixed carbon content of 19.94%, which significantly increased to 63.01% after torrefaction.

Table 3: The Effect of Variation in Process Temperature on Proximate Composition of *Terminalia superba*

| Sample | %Mc | %Ac | %Vm | %Fc |
|---|---|---|---|---|
| Raw TS | 21.16 | 2.81 | 59.80 | 19.94 |
| 200 °C | 19.08 | 5.86 | 40.52 | 34.54 |
| 250 °C | 17.95 | 5.07 | 20.63 | 56.30 |
| 300 °C | 15.00 | 4.28 | 17.71 | 63.01 |

Mc = Moisture content, Ac = Ash content, Vm = Volatile matter and Fc = Fixed carbon

**The effect of torrefaction on elemental composition of sample**

The data in Table 4 show the effects of varying torrefaction temperatures on the elemental composition of wood dust biomass samples from the *Terminalia superba* (TS) tree species, focusing on carbon, hydrogen, nitrogen, sulfur, and oxygen content. As the torrefaction temperature increases, the carbon content in the biomass also rises. Conversely, there is a noticeable decrease in the hydrogen and oxygen content of the sample with more severe torrefaction. The elemental composition of wood dust from *Terminalia superba* was reported to range between 80-50% carbon, 7-3% hydrogen, 60-40% oxygen, and 0.1-0.8% nitrogen, according to sources [12, 13]. The increase in carbon content can be linked to the reduction in hydrogen and oxygen, thereby enhancing the biomass's energy content. Combustion of biomass, an exothermic reaction with oxygen, produces carbon dioxide and water, confirming that biofuels' fuel qualities are largely determined by their carbon and hydrogen content. As the torrefaction temperature increases, the H/C (hydrogen to carbon) and O/C (oxygen to carbon) ratios decrease.

The Van Krevelen diagram in Figure 1 shows that the elemental composition of biomass can reliably indicate the quality of feedstock. As the torrefaction process intensifies, the biomass dehydrates, moving from the upper right to the lower left quadrant in the diagram, and begins to resemble coal.

Table 4: Effect of Torrefaction on Elemental Composition of Torrefied and Untorrefied Wood Dust

| Sample | % H | % S | % O | % C | % N | H/C | O/C |
|---|---|---|---|---|---|---|---|
| RTS | 6.53 | 0.08 | 43.06 | 49.98 | 0.35 | 0.15 | 0.86 |
| 200 °C | 5.51 | 0.04 | 35.63 | 58.43 | 0.39 | 0.09 | 0.61 |
| 250 °C | 4.55 | 0.03 | 30.28 | 64.68 | 0.46 | 0.07 | 0.47 |
| 300 °C | 3.51 | 0.02 | 23. 34 | 72.50 | 0.63 | 0.05 | 0.32 |

%O[a]: Oxygen is evaluated by difference; Raw *Terminalia superba* (RTS).

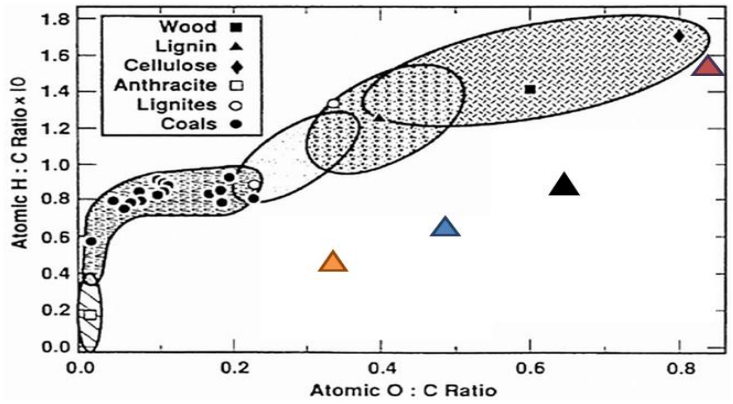


Figure 1: Van Krevelen diagram for tested biomass, coals, and several known species

Raw *Terminalia superba* (RTS).

Torrefied *Terminalia superba* 200 °C

Torrefied *Terminalia superba* 250 °C

Torrefied *Terminalia superba* 300 °C

**Surface morphology of the biomass**

The findings illustrated in Figures 2, 3 and 4 indicate that the torrefaction process significantly alters the surface morphology of the samples. The sample that was not torrefied showed a tightly packed and consolidated fiber structure, whereas the torrefied sample revealed a more disordered, porous, and less fibrous configuration. Additionally, it has been found that torrefaction increases the energy content of biomass, a change linked to processes such as dehydration, devolatilization, and depolymerization. Therefore, the analysis of surface morphology supports the conclusion that torrefaction enhances the grindability, porosity, and energy content of biomass.

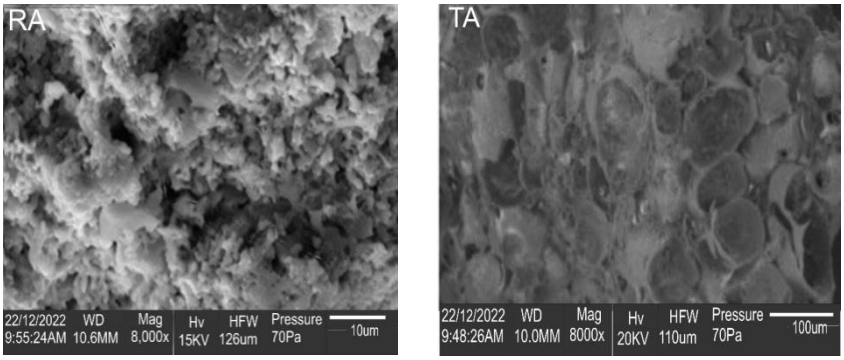


Figure 2: The surface morphology of the untorrefied (Raw) and torrefied wood dust biomass of *Terminalia superba* at 8000x magnification.

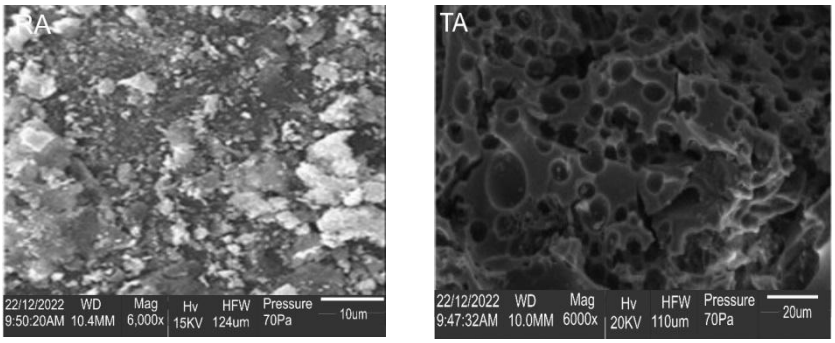


Figure 3: The surface morphology of the untorrefied (Raw) and torrefied wood dust biomass of *Terminalia superba* at 6000x magnification.

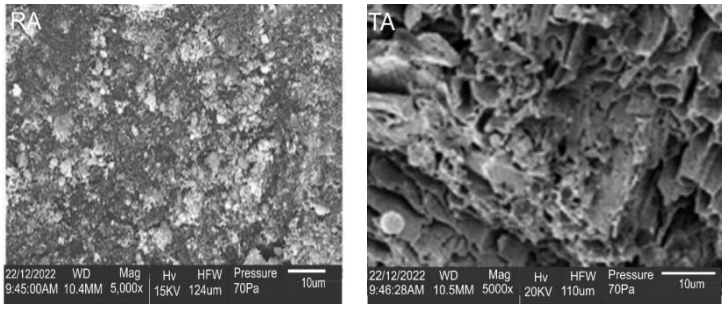


Figure 4: The surface morphology of the untorrefied (Raw) and torrefied wood dust biomass of *Terminalia superba* at 5000x magnification.

**Hydrophobicity testing**

The hydrophobic nature of biomass can be evaluated by examining its equilibrium moisture content (EMC), as shown in previous studies [14]. EMC represents the moisture level at which biomass neither gains nor loses moisture. The data in Table  reveals a decrease in EMC after the torrefaction process. Specifically, the moisture content of *Terminalia superba dust* decreased from 7.00% to 3.50%. This observation suggests that the extent of torrefaction positively impacts the hydrophobic characteristics of biomass.

Table 5: Effect of torrefaction on hydrophobicity of torrefied and untorrefied wood dust of *Terminalia superba*

| Samples | % Equilibrium Moisture Content (EMC) |
|---|---|
| RMI | 7.00 |
| TMI | 3.50 |

## CONCLUSION

The torrefaction temperature has a significant impact on the fuel characteristics of wood dust of *Terminalia superba*. As the torrefaction temperature increases, the moisture and volatile contents of the biomass decrease, while the fixed carbon content increases, indicating a higher energy potential. Specifically, torrefaction at 300 °C reduces the hydrogen and oxygen content in the *Terminalia superba* as evidenced by the declining H/C and O/C ratios in the torrefied samples. This suggests that torrefied *Terminalia superba* may burn more efficiently. The study also found that the torrefaction process temperature enhances the lignin concentration in the biomass, which improves pellet formation and binding. Torrefaction at 300 °C destroys the hydroxyl and methoxyl groups, leading to a reduction in oxygen and hydrogen levels in the *Terminalia superba*, further enhancing its fuel characteristics. SEM showed changes in the sample structure. The findings indicate that the optimal torrefaction temperature for *Terminalia superba* wood dust is 300 °C, as it improves the fuel properties and reduces the moisture, volatile, and oxygen content, while increasing the fixed carbon content and lignin concentration.

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