*Article*

# Investigation of the Effects of Torrefaction Temperature and Residence Time on the Fuel Quality of Corncobs in a Fixed-Bed Reactor

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**Abstract:** Biomass from agriculture is a promising alternative fuel due to its carbon-neutral feature. However, raw biomass does not have properties required for its direct utilization for energy generation. Torrefaction is considered as a pretreatment method to improve the properties of biomass for energy applications. This study was aimed at investigating the effects of torrefaction temperature and residence time on some physical and chemical properties of torrefied corncobs. Therefore, a fixed-bed torrefaction reactor was developed and used in the torrefaction of corncobs. The torrefaction process parameters investigated were the torrefaction temperature (200, 240, and 280 °C) and the residence time (30, 60, and 90 min). The effects of these parameters on the mass loss, grindability, chemical composition, and calorific value of biomass were investigated. It was shown that the mass loss increased with increasing torrefaction temperature and residence time. The grinding throughput of the biomass was improved by increasing both the torrefaction temperature and the residence time. Torrefaction at higher temperatures and longer residence times had greater effects on the reduction in particle size of the milled corncobs. The calorific value was highest at a torrefaction temperature of 280 °C and a residence time of 90 min. The energy yield for all treatments ranged between 92.8 and 99.2%. The results obtained in this study could be useful in the operation and design of torrefaction reactors. They also provided insight into parameters to be investigated for optimization of the torrefaction reactor.

**Keywords:** corncob; torrefaction; temperature; residence time; bioenergy

## 1. Introduction

Biomass is a preferred fuel for domestic and industrial use due to its carbon-neutral feature and its regional availability. It is also readily available and in large quantities. Studies on global and regional estimates of biomass energy have noted that biomass has a large technical potential for generation of energy and power [1–5]. However, biomass sourced from agricultural residues has poor fuel properties that hinder its use in the generation of energy in its raw form. Properties that are inherent in raw biomass include a high moisture content, high hygroscopicity, low bulk density, low energy density, high ash content and chemical compositions characterized by elevated amounts of alkaline and acid-forming components [6,7]. The oxygen content in raw biomass is high, which results in the production of a large volume of flue gases during combustion [8]. Biomass can also

be difficult to comminute due to its fibrous nature, and comminution is often related to a high quantity of fines and losses.

Corncobs are available in large quantities and are viable for the production of energy locally and regionally [9]. Asonja et al. [10] noted that corncobs are environmentally friendly and cost-effective when used as biofuel, and are recommended for heat energy production in agriculture. There are different biomass conversion technologies that have been explored to extract energy from corncobs. Biomass conversion technologies have been classified into thermochemical processes, biochemical processes, and physical or mechanical processes. There have been studies seeking to improve the efficiency of corncob utilization for bioenergy through thermochemical processes such as combustion [11,12], gasification [13,14], and carbonization [15]. There has been some work to investigate biochemical processes such as anaerobic digestion [16] and ethanol fermentation [17]. For physical and mechanical processes, studies have been carried out on comminution [18] and densification [19,20].

Torrefaction is a thermochemical process for upgrading biomass for energy applications such as direct combustion and cocombustion with coal [21]. It allows biomass to be stored so that there is little or no microbial degradation. Apart from enhancing the energy density, torrefaction also improves the grindability of fibrous biomass materials and the particle size distribution [22]. Torrefaction involves heating biomass in an oxygen-starved environment within temperatures ranging from 200 to 300 °C with a heating rate less than 50 °C/min at ambient pressure [23]. The process involves several complex reactions through which biomass is converted into a mixture of solid, liquid, and gaseous products. Torrefaction has been referred to as mild or partial pyrolysis of biomass. The main product from torrefaction is the solid char. Nhuchhen et al. [24] noted that dry torrefaction comprises four steps that include drying, postdrying, torrefaction, and cooling. During drying, the free or surface moisture is removed. During postdrying, the bound moisture and some hydrocarbons are removed. Torrefaction or an isothermal heating process results in depolymerization, partial devolatilization, and partial carbonization reactions. A cooling process brings the reactor back to ambient temperature.

There are a number of benefits derived from the torrefaction of biomass. Torrefaction produces moisture-free hydrophobic solid products with a decreased oxygen-to-carbon ratio [25]. Torrefaction is also known to increase the bulk density of biomass, which simplifies storage and transportation [22]. Furthermore, smokeless and cleaner combustion is achieved when torrefied biomass is used, and with a better performance than raw biomass in gasifiers [26,27].

Tumuluru et al. [28] noted that during torrefaction, the major decomposition reactions affect the hemicellulose, whilst lignin and cellulose are less affected. It has been noted that torrefied biomass retains most of its energy and simultaneously loses its hygroscopic properties. Temperatures higher than 300 °C were also not recommended because they may lead to excessive devolatilization of biomass due to the pyrolysis process being initialized. Torrefaction improves the ultimate and proximate composition by increasing the carbon content and calorific value and decreasing the moisture and oxygen content. In addition, the biochemical composition is improved by decomposing the hemicelluloses and softening the lignin, which results in better binding during pelletization.

Factors affecting the torrefaction process include temperature, residence time, oxygen concentration, particle size, and reactor type [24]. Strandberg et al. [29] investigated the effects of temperature and residence time on the continuous torrefaction of spruce wood and found that the increased torrefaction severity resulted in decreased milling energy consumption, angle of repose, mass and energy yield, content of volatile matter, hydrogen, cellulose, and hemicellulose. Barta-Rajnai et al. [30] also investigated the effect of temperature on the thermal behavior of different parts of Norway spruce and found that the effect of temperature was greater than the effect of residence time up to 275 °C, whilst at 300 °C, the composition of torrefied samples was significantly influenced by the residence time.

Ramos-Carmona et al. [31] studied the effect of temperature on properties of *Pinus patula* and observed that there was no significant difference in chemical composition and thermal behavior between raw biomass and biomass torrefied at 200 and 250 °C. However, material torrefied at 300 °C showed important changes in chemical composition and thermal behavior. Wang et al. [32] observed that, compared to particle size, temperature was the more important variable. However, as the particle size increased, more of the torrefied product remained in the reactor, and energy yields decreased.

In investigations carried out by Chen et al. [33], it was discovered that after torrefaction, the grinding performance and hydrophobicity of cotton stalk were improved and the number of functional groups containing oxygen was decreased. Rodrigues and Rousset [34] investigated the energy properties of torrefied Eucalyptus grandis wood at 220, 250, and 280 °C and found that a temperature of 250 °C generated the best energy density occasioned by an increase in the heating value and a slight decrease in the bulk density. Wang et al. [35] observed that particles from ground torrefied samples of stem wood, stump, and bark of Norway spruce were much smaller than those of untreated samples.

Chin et al. [36] utilized response surface methodology in the optimization of torrefaction conditions and observed an increased degradation of material due to the combined effects of temperature and treatment time. It was also found that each type of biomass had its own unique set of operating conditions to achieve the same product quality. In a study by Batidzirai et al. [37], it was realized that for full commercialization, torrefaction reactors need to be optimized to obtain the desired performance. An optimum performance will result in a consistent, homogenous, fully hydrophobic, and stable product that is capable of using different feedstocks.

Lu and Chen [38] investigated the product yield and characteristics resulting from the torrefaction of corncobs at temperatures of 250 °C and 300 °C for 1 h using nitrogen, carbon dioxide, and a gas mixture of air and carbon dioxide as carrier gases. It was found that a higher torrefaction temperature resulted in a higher ignition temperature of the treated biomass except for when the mixture of air and carbon dioxide was used as carrier gas. Klaas et al. [39] observed that the grinding energy of corncobs could be reduced by 69% when torrefaction was applied from 240 to 260 °C.

Corn is one of the major staple foods in developing countries, accounting for most of the calorie intake of the rural population [1]. Corncobs, therefore, are available in large quantities, which makes them viable as solid fuel. Although corncobs are currently used as cooking fuels, they are used inefficiently. The efficiency of using corncobs as a solid fuel can be improved via pretreating by using methods such as torrefaction. Torrefaction has been utilized as a pretreatment step for the production of bio-oils using pyrolysis. Klaas et al. [39] carried out torrefaction temperatures between 240 and 300 °C in a batch reactor with heated nitrogen as the carrier gas and subsequent pyrolysis at temperatures of 400 to 550 °C. Torrefaction was found to be capable of reducing the water content and viscosity of the bio-oil. Similarly, Zheng et al. [40] carried out torrefaction in an auger reactor at 250 to 300 °C with residence times of 10 to 60 min and fast pyrolysis at 470 °C. The study found that the heating value and the pH of the bio-oil improved with the application of torrefaction. A similar study by Zheng et al. [41] showed that severe torrefaction at 270 to 300 °C can lead to a sharp decrease in the coke yield and a reduction in the aromatic yield.

Other studies investigating the properties of torrefied corncob have also been carried out. Zheng et al. [42] compared wet and dry torrefaction of corncobs in a high-pressure tube-type reactor and found that hemicellulose was effectively removed from the corncobs by torrefaction. However, dry torrefaction resulted in a severe degradation of cellulose, as well as the cross-linking and charring of the corncobs. In a study by Tian et al. [43], it was shown that torrefaction in a tubular furnace at a heating rate of 15 °C min$^{-1}$ increased the calorific value of corncobs, reduced the oxygen content, reduced the mass yield, and increased the energy yield. It was found that the optimum pretreatment temperature of corncobs was 240 °C. In an investigation by Medic et al. [44] using a thermogravimetric analyzer and temperatures of 250 and 280 °C, corncob shells torrefied at 280 °C had the highest

energy density, reaching 21.5 MJ/kg, whilst the highest yield of 85% was obtained at 250 °C. Akhtar et al. [45] performed torrefaction of corncobs using a tube furnace reactor using nitrogen as the carrier gas. The optimum temperature, residence time, and gross calorific value obtained for the torrefied corncobs were 290 °C, 20 min, and 22.8 MJ/kg, respectively. Garba et al. [46] utilized a furnace-heated tubular reactor with nitrogen gas to investigate the effect of torrefaction temperature on different biomass feedstock, including corncobs. The study showed that at low torrefaction temperatures of 200 and 250 °C, the weight loss was pronounced compared to torrefaction carried out at 300 °C. Kanwal et al. [47] carried out torrefaction of corncobs at temperatures between 200 and 300 °C and residence times between 15 and 60 min in a lab-scale tube furnace using nitrogen as the carrier gas. It was found that as the torrefaction temperature and residence time increased, significant improvements in the physical and chemical characteristics of the torrefied corncobs were observed. At the highest torrefaction temperature and residence time, the physical and chemical characteristics of the torrefied corncobs were equivalent to those of Thar coal.

Several studies have reported the investigation of torrefaction parameters on corncobs and other biomass sources in an inert environment created by passing nitrogen or other inert gases such as carbon dioxide [38,48] through torrefaction reactors. Fewer studies have attempted the investigation of torrefaction in other environments such as oxidative atmospheres [49] and a vacuum environment [50]. In this study, a fixed-bed reactor that did not require the use of carrier gases was designed and used to investigate the effects of torrefaction process variables on the properties of torrefied corncobs. The variables considered included the torrefaction temperature and the residence time in the reactor. This study examined the weight loss, grindability, calorific value, and proximate analyses of the corncobs torrefied under different torrefaction temperatures and residence times.

## 2. Materials and Methods

### 2.1. Acquisition of Biomass

The corncobs used in this study were obtained from corn harvested in November. The cobs were collected from corn-processing sites in Osun state, Nigeria, after the harvested corn was hand-shelled. About 165 kg of corncobs were collected from the farms and transported to the laboratory in jute bags. The collected corncobs were then air-dried under laboratory conditions for three months to a moisture content of <8% before being stored in airtight bags. For the study, 93 kg of the collected corncobs were used for the torrefaction and milling experiments.

### 2.2. Design of a Reactor for Torrefaction of Biomass

A fixed-bed reactor was designed for torrefaction. The reactor was a batch-type and was electrically heated in order to control the heating rate and regulate the temperature effectively. The reactor is shown in Figure 1.

#### 2.2.1. Geometric Specifications of the Reactor

The mass of torrefied biomass to be produced in a batch was assumed to be 7.5 kg. According to Tumuluru et al. [28], the loss in mass of biomass material during the torrefaction process is 30%. Therefore, the initial mass of biomass material to be loaded into the reactor was estimated to be 11 kg. By taking the bulk density of the corncobs as 50.32 kg m$^{-3}$ [51], the volume of the reactor was estimated to be 0.22 m$^3$. Taking the ratio of the height to the diameter as 2.5, an inner diameter of 304 mm and a height of 760 mm were chosen for the torrefaction reactor.
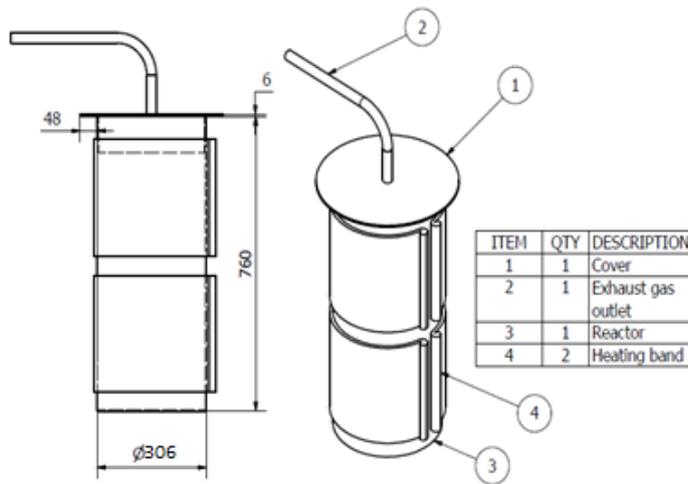
**Figure 1.** The torrefaction reactor.

2.2.2. Heating-Energy Requirement

The stages in the torrefaction process included an increase in temperature from the ambient temperature to the drying temperature (between 90 and 105 °C), drying of the raw biomass at the drying temperature, heating the biomass up to the torrefaction temperature, and maintaining a constant torrefaction temperature for a required retention time. Thereafter, cooling to the ambient temperature was allowed to take place in an inert environment.

The total energy supplied for the torrefaction process, $Q_{tor}$, was estimated using:

$$Q_{tor} = Q_1 + Q_2 + Q_3 + Q_4 \tag{1}$$

where $Q_1$ is the heat required to raise biomass from ambient to drying temperature; $Q_2$ is the heat required to evaporate unbound moisture in the biomass at the drying temperature; $Q_3$ is the heat required to raise the temperature from the drying temperature to the torrefaction temperature; and $Q_4$ is the heat required for torrefaction at the torrefaction temperature.

The first phase of the reaction required the biomass to be heated to a temperature at which the unbound moisture was evaporated. The heat required for this stage, $Q_1$, was obtained as [23]:

$$Q_1 = (m_d c_{p,b} + m_w c_{p,w})\Delta T_1 \tag{2}$$

where $m_d$ represents the mass of the dry content of the biomass material; $m_w$ is the mass of moisture present in the biomass material; $\Delta T_1$ is the temperature change from the ambient condition to the drying temperature; $c_{p,b}$ is the specific heat capacity of the biomass material; and $c_{p,w}$ is the specific heat capacity of water (see Table 1). The second stage involved the evaporation of unbound moisture. The energy, $Q_2$, to evaporate the moisture in the biomass on basis of the required evaporation heat of water $h_{ev}$, was estimated as [23]:

$$Q_2 = m_w h_{ev} \tag{3}$$

**Table 1.** Parameters used in the estimation of heat energy requirement.

| Parameter | Definition | Value |
|---|---|---|
| $m_w$ | Mass of moisture per batch | 1.65 kg |
| $m_d$ | Dry mass per batch | 9.35 kg |
| $m_{tor}$ | Dry-matter loss during torrefaction | 1.65 kg |
| $h_{ev}$ | Heat of vaporization of water | 2.27 MJkg$^{-1}$ |
| $c_{p,b}$ | Specific heat capacity of the biomass | 1.40 kJkg$^{-1}$K$^{-1}$ |
| $c_{p,w}$ | Specific heat capacity of water | 4.18 kJkg$^{-1}$K$^{-1}$ |
| $h_{tor}$ | Heat of reaction for torrefaction | 1.00 MJkg$^{-1}$ |
| $\Delta T_1$ | Temperature difference between ambient and drying temperature | 73 °C |
| $\Delta T_3$ | Temperature difference between drying and torrefaction temperature | 200 °C |

The third stage involved increasing the temperature of the biomass to the torrefaction temperature. The heat required to raise the temperature of the dried biomass to the temperature of torrefaction was calculated as [23]:

$$Q_3 = m_d c_{p,b} \Delta T_3 \tag{4}$$

The fourth stage involved the devolatilization, or decomposition, of biomass at the torrefaction temperature. The heat, $Q_4$, required for this stage was obtained from [23]:

$$Q_4 = m_{tor} h_{tor} \tag{5}$$

where $m_{tor}$ is the mass of biomass material lost to the torrefaction process and $h_{tor}$ is the heat of reaction for torrefaction. Tumuluru et al. [28] stated that the mass yield is approximately 70% of the initial mass of biomass used. The parameters used in the estimation of the heating energy requirement are presented in Table 1. The torrefaction temperature considered was 300 °C, which is the highest torrefaction temperature. An ambient temperature of 27 °C was utilized, being the average for Lagos, Nigeria. Using the estimation of the heat requirement at the different stages, $Q_1$, $Q_2$, $Q_3$, $Q_4$, and $Q_{tor}$ were estimated to be 1.46 MJ, 3.75 MJ, 2.61 MJ, 1.65 MJ, and 9.47 MJ, respectively, for the torrefaction of one batch of 11 kg air-dried corncobs in the reactor shown in Figure 1.

2.2.3. Power Requirement for Heating

The heating rate for the torrefaction of biomass is less than 50 °C min$^{-1}$ [23]. A heating rate of 30 °C min$^{-1}$ was chosen for the reactor. The heating power requirement, $\dot{Q}$, to achieve the heating rate was estimated as:

$$\dot{Q} = (m_d c_{p,b} + m_w c_{p,w}) \frac{dT}{dt} \tag{6}$$

where $dT/dt$ is the heating rate. The power required for heating, $\dot{Q}$, was 10 kW.

*2.3. Operating Principle of the Torrefaction Reactor, Execution of the Trials, and Sampling*
2.3.1. Experimental Design

The factors investigated during torrefaction were the residence time and the temperature and their influences on mass during torrefaction and on the material properties. Three levels each of the residence time (30, 60, and 90 min) and the temperature (200, 240, and 280 °C) were used. All possible combinations of parameters, comprising precisely nine runs (Exp. 1–9), were used in the study. As a result of the large batch, randomly collected samples were considered to be representative averages of each experimental run. The results from every experimental run were compared with the untreated corncobs taken as a reference (Exp. 10).

### 2.3.2. Operation of the Torrefaction Reactor

To carry out torrefaction with corncobs, the cobs were placed in the torrefaction reactor until it was filled to about 90% of its volume. The cover of the torrefaction reactor was fastened, and the reactor was made airtight with the aid of gaskets. The electrical heater was turned on, and the required temperature for torrefaction was set on the temperature controller. The temperature change was monitored with a digital thermometer. The setup was heated to the required torrefaction temperature and maintained at that temperature for the desired residence time, after which the heater was turned off. The setup was then allowed to cool to room temperature. Thereafter, the torrefied biomass was removed from the reactor.

### 2.3.3. Mass Loss during Torrefaction

Mass loss is due to moisture loss and the thermal decomposition of biomass [52]. The mass loss during torrefaction was determined for each batch from every experimental run. The losses were calculated on the basis of the mass of the batch before the torrefaction treatment using [53]:

$$\text{Mass loss} = \frac{(\text{Mass of raw biomass} - \text{Mass of torrefied biomass})}{\text{Mass of raw biomass}} \times 100\% \qquad (7)$$

### 2.3.4. Effect of Torrefaction on Grindability (Grinding Time and Particle Size Distribution)

To determine the effect of torrefaction on the grindability, the cobs were torrefied using the levels of variables specified. For every experimental run, a 5 kg sample of the torrefied product was milled using a laboratory scale hammer mill with a screen mesh size of 6 mm. The corresponding grinding times for each treatment were determined and utilized in estimating the grinding throughput using:

$$\text{Grinding throughput} = \frac{\text{Mass of material grinded}}{\text{grinding time}} \; (\text{kg/h}) \qquad (8)$$

The particle sizes of the milled corncobs were characterized using a vibrating sieve (sieve sizes 9.5, 4.75, 1.18, 0.60, 0.30, and 0.15 mm). The results of the particle size analyses of the torrefied corncobs were compared to raw corncobs (referred to as the reference).

### 2.3.5. Effect of Torrefaction on the Proximate Analyses of Corncobs

Proximate analyses of the corncobs were conducted before torrefaction and after torrefaction at different temperatures and residence times. The samples for the proximate analyses were taken from the milled and well-mixed material. The proximate analyses characterized the ground cob samples in terms of the volatile matter, fixed carbon, and ash content. This gave an idea of the bulk components that composed the biomass. The procedures used in carrying out the proximate analyses were conducted according to the ASTM standard D 1762-84 [54].

#### Effect of Torrefaction on Moisture Content

Depending on the torrefaction time and temperature, the biomass lost moisture during the treatment in the reactor. For determination of the moisture content at the end of torrefaction, the oven-dry method was used; 1 g of the ground sample was weighed into a crucible and oven-dried at 105 °C for 2 h. Thereafter, the change in weight was determined after every hour until the change in mass was negligible. The percentage moisture content was estimated as:

$$\text{MC} = \frac{(\text{Initial mass of sample}) - (\text{Mass of dried sample})}{(\text{Initial mass of sample})} \times 100\% \qquad (9)$$

Determination of Volatile Matter

The volatile matter was determined using a muffle furnace heated to 950 °C. The sample obtained from the previous determination of moisture content was utilized in the determination of the volatile matter. The sample, held in a crucible, was put in a furnace with the lid in place at a temperature of 950 °C for 6 min and then cooled to room temperature. The percentage of volatile matter was determined as:

$$\text{VM} = \frac{(\text{Initial mass of dried sample}) - (\text{Final mass of sample})}{\text{Initial mass of dried sample}} \times 100\% \qquad (10)$$

Determination of Ash Content

The muffle furnace was utilized to determine the ash content of the biomass samples. The sample utilized from the determination of the volatile matter was used to determine the ash content, *AC*. The sample was heated without a lid in a furnace at 750 °C for 6 h, then the sample was cooled and weighed. The percentage of ash content was estimated as:

$$\text{AC} = \frac{\text{Mass of ash left}}{\text{Initial mass of dried sample}} \times 100\% \qquad (11)$$

Determination of Fixed Carbon

The fixed carbon, *FC*, was computed by subtracting the sum of the percentage content of the moisture, volatile matter, and ash content from 100. The estimation was calculated as:

$$\text{FC} = 100\% - (\text{MC} + \text{VM} + \text{AC}) \qquad (12)$$

2.3.6. Effect of Torrefaction on the Calorific Value of Corncobs

The effects of the levels of the torrefaction variables on the calorific value of corncobs were determined using an oxygen bomb calorimeter (Model 6100, Parr Instrument Co., IL, USA). The higher heating value (HHV) was measured in the calorimeter according to the ASTM standard D2382-88 [55]. A weighed sample of approximately 0.1 g was combusted inside the calibrated adiabatic bomb calorimeter, which was pressurized with pure oxygen (99.99%) to 2.0 MPa. The result was taken from the display unit of the bomb calorimeter.

The energy densification was determined as:

$$\text{Energy densification} = \frac{\text{HHV of torrefied biomass} - \text{HHV of raw biomass}}{\text{HHV of raw biomass}} \times 100\% \qquad (13)$$

Energy yield was used to measure the torrefaction efficiency [52], and was estimated as:

$$\text{Energy yield} = \text{Mass yield} \times \frac{\text{HHV of torrefied biomass}}{\text{HHV of raw biomass}} \qquad (14)$$

The mass yield was obtained from Equation (7) as:

$$\text{Mass yield} = 100\% - \text{Mass loss} \qquad (15)$$

## 3. Results and Discussion

### 3.1. Mass Loss during Torrefaction

Mass loss was experienced during torrefaction due to the removal of moisture content and volatile matter caused by the process. The mass loss observed during the torrefaction of the corncobs varied from 15 to 40% depending on the temperature, residence time, and their interactions (Figure 2 and Table 2). It was observed that as the torrefaction temperature increased, the mass loss of the biomass also increased. Furthermore, the mass loss of the biomass increased with an increase in the residence time for all temperatures. Consequently, the highest mass loss of 40% was measured for a torrefaction temperature of 280 °C and a residence time of 90 min.
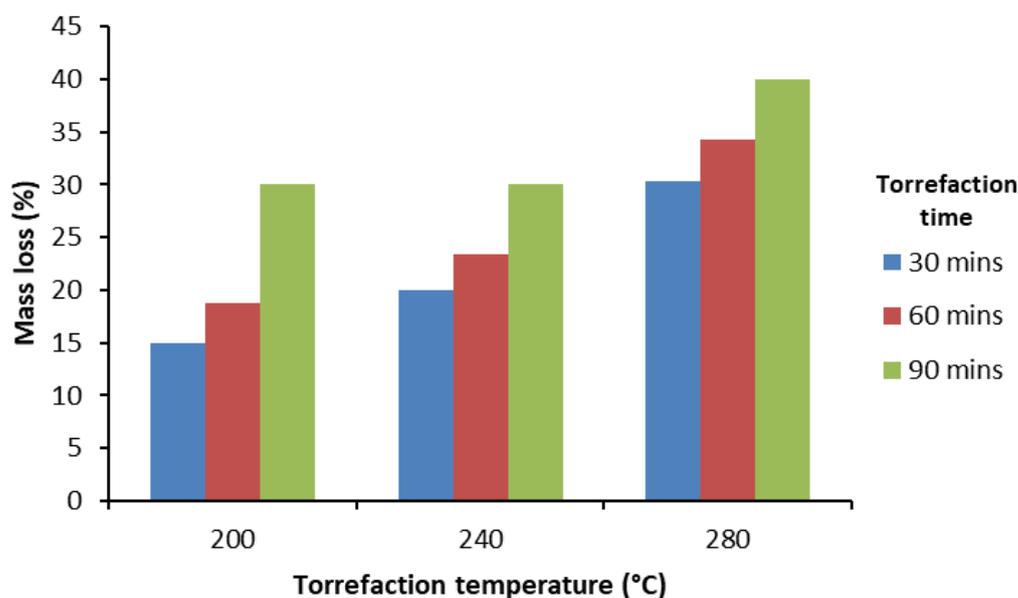
**Figure 2.** Mass loss during torrefaction of corncobs.

**Table 2.** Results of grinding throughput, proximate analyses, and energy yield of raw and torrefied corncobs.

| Exp. Run | Temp. (°C) | Time (min) | Mass Loss (%) | Grinding Throughput (kg h$^{-1}$) | Moisture Content (%) | Volatile Matter (%) | Ash Content (%) | Fixed Carbon (%) | Calorific Value (MJkg$^{-1}$) | Energy Densification (%) | Energy Yield (%) |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | 200 | 30 | 15.0 | 8.6 | 3.00 | 62.5 | 2.99 | 31.51 | 23.22 | 9.2 | 92.8 |
| 2 | 200 | 60 | 18.8 | 10.0 | 2.95 | 59.5 | 2.80 | 34.75 | 25.11 | 18.0 | 95.9 |
| 3 | 200 | 90 | 30.2 | 12.0 | 2.75 | 58.5 | 2.60 | 36.15 | 29.26 | 37.6 | 96.0 |
| 4 | 240 | 30 | 20.0 | 12.0 | 2.70 | 57.0 | 2.60 | 37.70 | 24.12 | 13.4 | 90.7 |
| 5 | 240 | 60 | 23.3 | 10.7 | 2.55 | 56.5 | 2.45 | 38.50 | 26.20 | 23.2 | 94.4 |
| 6 | 240 | 90 | 30.1 | 15.0 | 2.56 | 58.0 | 2.30 | 37.14 | 29.76 | 39.9 | 97.8 |
| 7 | 280 | 30 | 30.3 | 16.7 | 2.45 | 58.9 | 3.50 | 35.15 | 30.24 | 42.2 | 99.0 |
| 8 | 280 | 60 | 34.3 | 25.0 | 2.10 | 61.0 | 2.75 | 34.15 | 31.02 | 45.8 | 97.2 |
| 9 | 280 | 90 | 40.0 | 30.0 | 1.90 | 64.5 | 2.45 | 31.15 | 35.20 | 65.5 | 99.2 |
| 10 | - | - | - | 3.3 | 7.76 | 79.6 | 2.54 | 10.10 | 21.27 | - | - |

Granados et al. [56] performed torrefaction of different biomass feedstocks at a temperature of 250 °C with a heating rate of 10 °C min$^{-1}$ and residence time of 30 min. The researchers obtained mass losses of 38% for palm oil fiber, 42% for banana rachis, 18% for sugarcane bagasse, 22% for rice husk, 25% for sawdust, and 30% for coffee waste. The mass losses obtained in our study reasonably agreed with those of Granados et al. [56], but we also observed that the mass losses were dependent on the type of biomass. Demirbas [57] stated that mass loss is due to the degradation of hemicellulose, but is dependent on the chemical nature and relationship with lignin. Di and Lanzetta [58] also agreed that the mass loss during torrefaction depends on the type of biomass and chemical composition.

Anukam et al. [59] stated that the loss in weight of the biomass during torrefaction significantly depends on temperature as a consequence of the breakdown of the cellulose and hemicellulose content of the biomass. Mamvura et al. [60] also observed an increase in weight loss with an increase in temperature for marula seed and blue gum wood.

In the torrefied products obtained in the study under the different experimental conditions, it was observed that at 200 °C and 30 min, being the least invasive experimental run, a golden brown color of the corncobs was obtained. However, at the highest temperature of 280 °C and the highest residence time, the cobs had a black color. We also observed that the core of the cobs was least affected at the lowest residence times. The results of this study were similar to those obtained by Mamvura et al. [60], who observed that the torrefaction products turned darker with an increase in the temperature. The change in color has been attributed to increase in the breakdown of hemicellulose, which leaves a higher content

of carbon black. The milled products showing the changes in appearance of the torrefied corncobs with different treatment conditions are presented in Figure 3.
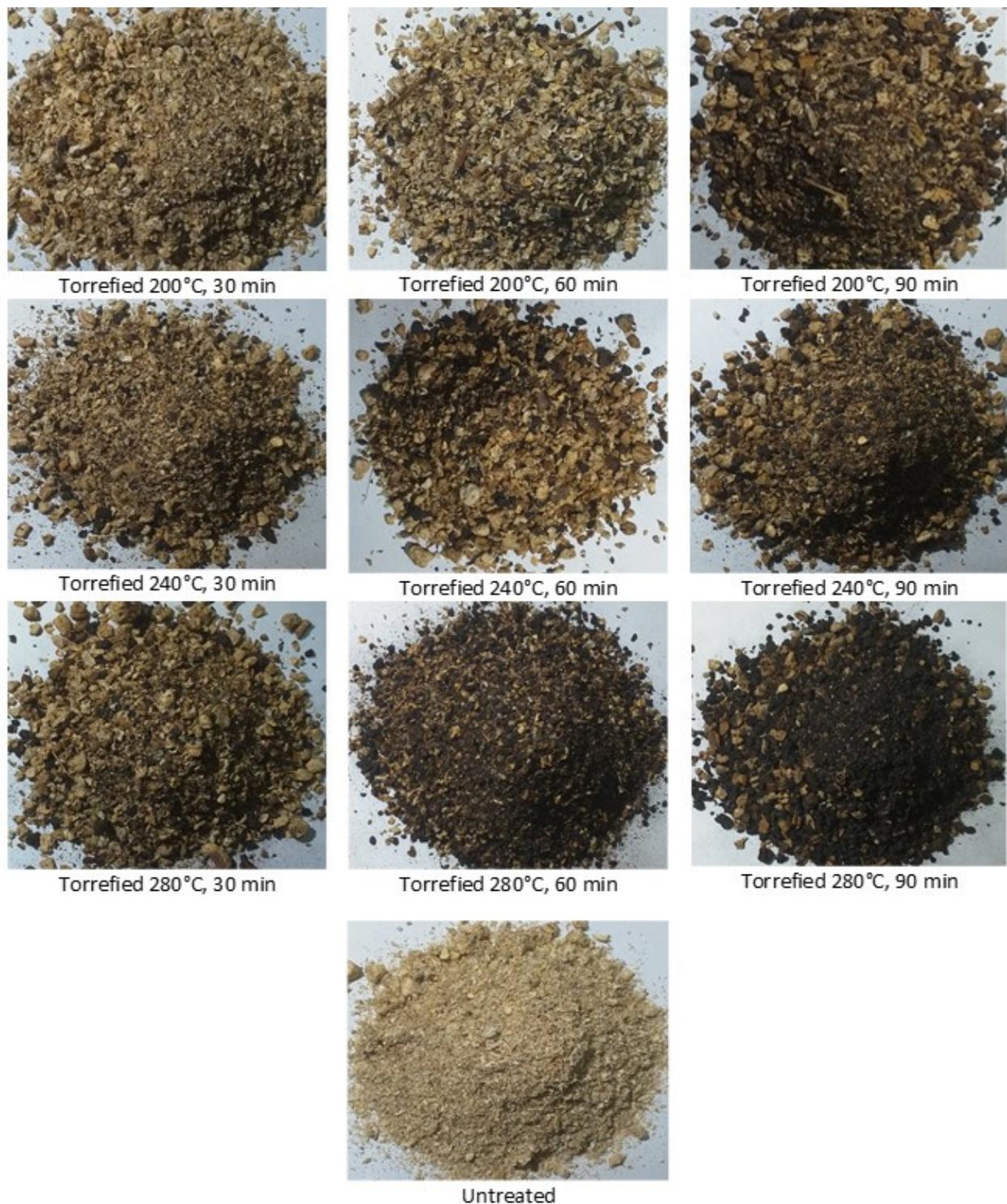


**Figure 3.** Milled products from untreated corncobs and torrefied corncobs produced under different temperatures and residence times.

*3.2. Effects of Torrefaction on Grindability*

The effects of torrefaction on the grinding throughput of torrefied corncobs compared to untorrefied corncobs (reference) are shown in Figure 4. We observed that the time taken

to mill raw corncobs was the highest, resulting in a low grinding throughput. The highest grinding throughput was observed for corncobs torrefied at the highest temperature of 280 °C and for 90 min. The time taken during milling was observed to be reduced with the torrefaction temperature. This implied that as the temperature increased, the fibrous nature of the material was degraded, and consequently, the cobs became more brittle. With the exception of the observation of milled corncobs torrefied at 240 °C, we also observed that the torrefaction time influenced the time taken in milling. An increase in the residence time at any torrefaction temperature decreased the milling time, which implied that the grindability of the cobs was increased. When comparing Figures 2 and 4, we observed that the mass loss had a positive correlation with the grinding throughput. According to Ohliger et al. [61], grindability depends on the degree of torrefaction, for which the mass loss is a good indicator.
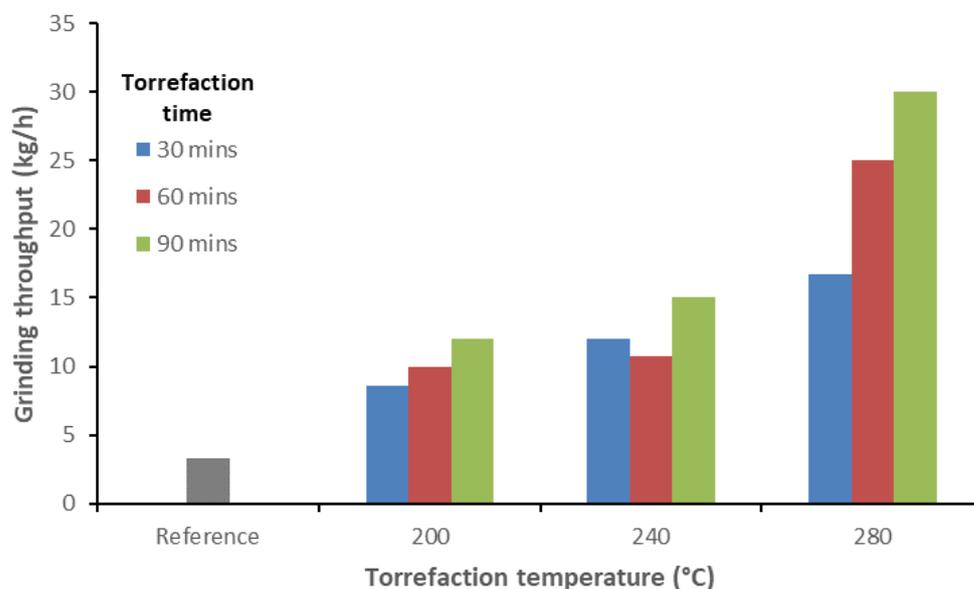


**Figure 4.** Grinding throughput of raw and torrefied corncobs.

The particle size analyses of milled corncobs torrefied at different temperatures and time compared with untorrefied corncobs are shown in Figure 5. Figure 5a shows the results for torrefaction carried out at 200 °C, whilst Figure 5b,c show the results for 240 and 280 °C, respectively. As shown in the figures, the X10 percentile values for the milled torrefied cobs had a range of 0.3 to 0.45 mm, which was less than the 0.8 mm obtained for the untorrefied cobs. Similarly, the median values of the particle size distribution, X50, indicated that the untorrefied corncobs had higher fractions of coarse particles than the torrefied cobs. We observed in the results that the highest torrefaction temperature of 280 °C along with a torrefaction time between 60 and 90 min resulted in the lowest values for X10, X50, and X90 compared to other treatments. This implied that torrefaction at higher temperatures coupled with long residence times had the greatest effect on the reduction in particle size. Repellin et al. [62] found that the particle size distribution decreased with the anhydrous weight loss due to increases in torrefaction. Brue et al. [63] found that corn stover was more brittle when torrefied and resulted in higher fraction of fine grounds than the untreated material.
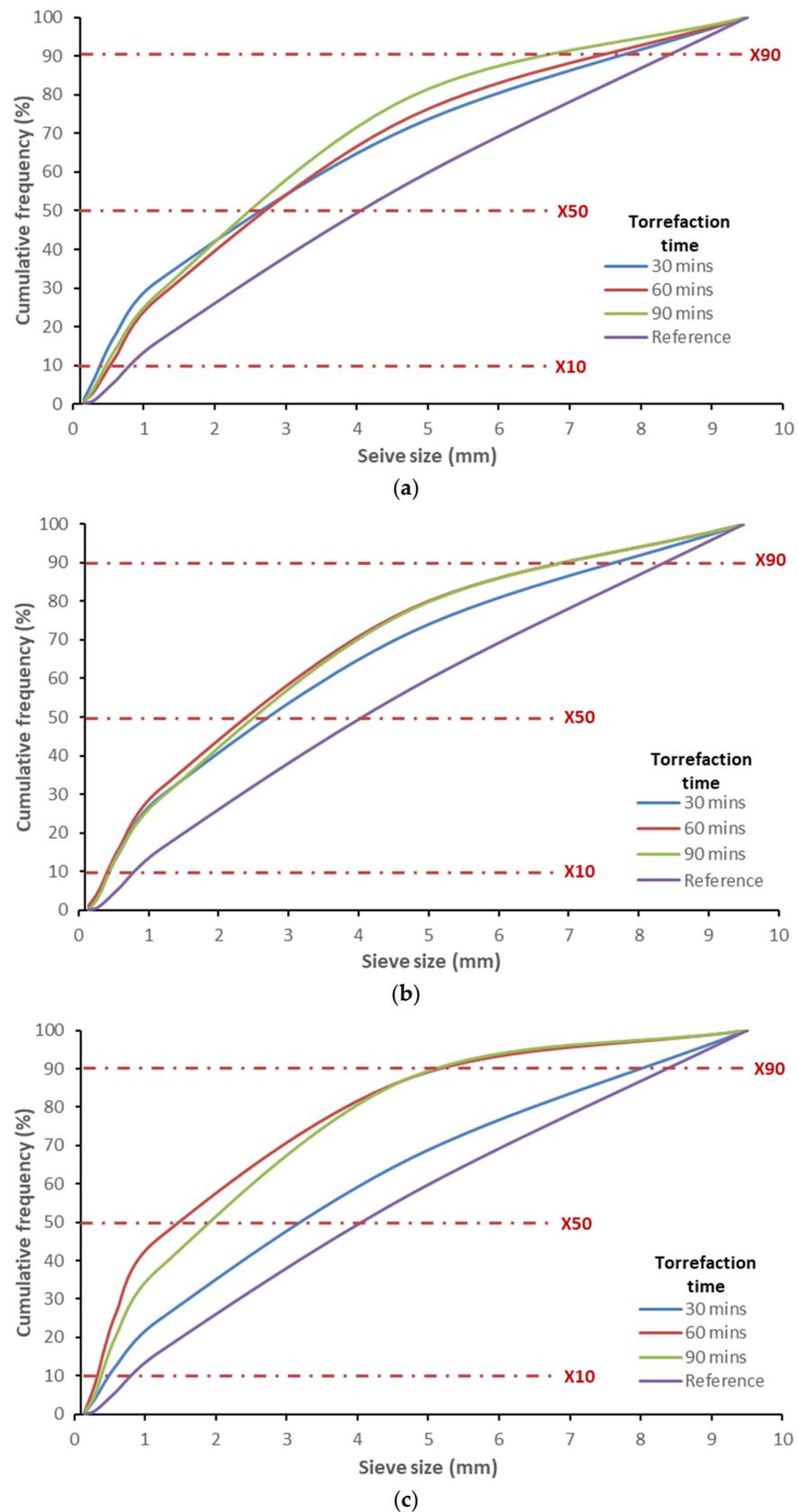
**Figure 5.** Particle size analysis of raw biomass compared with torrefied corncobs at: (**a**) 200 °C; (**b**) 240 °C; (**c**) 280 °C.

### 3.3. Proximate Analyses of Torrefied Corncobs

The proximate analyses gave a broad measurement of the constituents of the biomass material. The results of the proximate analyses of raw and torrefied corncobs are presented in Table 2. We observed that the percentage reduction in moisture content ranged from 61.3% for the mildest treatment (200 °C and 30 min) to 75.5% at the highest torrefaction temperature (280 °C) and torrefaction time (90 min). Tumuluru [64] also observed that the moisture content of pine grounds decreased from 4.2% with an increase in the torrefaction temperature and time. The percentage decrease in moisture content ranged from about 29.04% for a torrefaction temperature and time of 160 °C and 15 min, respectively, to about 72.61% at a torrefaction temperature and time of 270 °C and 120 min, respectively. For corn stover and switch grass, Tumuluru [65] observed a moisture loss of 56% and 73%, respectively, at 180 °C and 120 min, which increased to between 78.8 and 88.18% for both feedstocks. Tumuluru et al. [66] observed up to an 82.68% loss in moisture for a torrefaction temperature of 250 °C.

In Table 2, it can be observed that the volatile matter was reduced and the fixed carbon was increased. The ash content, however, was almost constant. In the table, it can be observed that the highest values of fixed carbon were obtained for torrefaction at 240 °C, and specifically when the corncobs were retained in the reactor for a residence time of 60 min. Tumuluru et al. [66] noted that torrefaction resulted in significant changes in the proximate composition of the biomass, making it more suitable for fuel applications. Bridgeman et al. [67] also noted that there was an increase in carbon content with an increase in the torrefaction temperature. However, in our study, this assertion was true only up to 240 °C. In general, the fixed carbon increased from 10.10% for the raw corncobs to between 31.15% and 38.50% for the torrefied corncobs. This implied that torrefaction increased the carbon content; however, optimum processing conditions must be utilized to maximize the carbon content for different biomass feedstocks. This will, however, be dependent on the feedstock being processed.

### 3.4. Calorific Value of Torrefied Corncobs

The calorific values (HHV) of the torrefied and untreated corncobs are presented in Table 2. It can be seen that the HHV of the torrefied corncobs increased with an increase in the residence time, and also with an increase in the torrefaction temperature. The HHV for the torrefied corncobs compared to the untreated corncobs increased by up to 65% at the highest settings of the torrefaction temperature and residence time. A similar observation was made by Pahla et al. [68], who noted that the HHV of torrefied corncobs and pinewood had a direct relationship with the torrefaction temperature, with the HHV increasing by up to 55.2% for corncobs. Tumuluru [64] obtained an increase in the value of the calorific value of lodgepole pine grind of about 21.9%.

Furthermore, we observed that the energy yield for all treatments had values above 90% but less than 100% (Table 2). It is characteristic of torrefaction processes to have energy yields less than 100% because the impact of weight loss has a greater impact than the increase in HHV [69]. Limousy et al. [69] obtained an energy yield in the range of 88 to 99% for coffee residues. Bangkha et al. [70] also obtained an energy yield between 58.7 and 99.4% for spent coffee grounds.

## 4. Conclusions

Torrefaction is important in the improvement of biomass for energy applications. It is therefore key to investigate the effects of the process parameters on the properties of torrefied products. In this study, a fixed-bed torrefaction reactor was developed and used in the torrefaction of corncobs. It was shown that torrefaction could be achieved using this type of reactor without necessarily utilizing nitrogen or other gases to create an inert environment within the reactor. The torrefaction process parameters investigated were the torrefaction temperature (200, 240, and 280 °C) and the residence time (30, 60, and 90 min). It was observed that both the torrefaction temperature and residence time influenced the

torrefaction of the corncobs. The mass loss during torrefaction was found to increase with an increasing temperature and also an increasing residence time. The grindability was also improved by increasing both the torrefaction temperature and the residence time. The fixed carbon increased from 10.10% to between 31.15% and 38.50%. However, in the proximate analyses, the fixed carbon was found to be maximized when the corncobs were torrefied at 240 °C and for 60 min. Torrefaction also increased the calorific value of the corncobs, with the highest value obtained in torrefaction conditions of 280 °C and 90 min. Overall, the best torrefaction results regarding product quality and yield were achieved at this high temperature and residence time. However, the high rate of energy densification and energy yield should be compared with the energy required for the torrefaction process in a subsequent study after optimization of the entire system. The results obtained in this study could be useful in the operation and design of torrefaction reactors, as well as in the further optimization of the torrefaction process.

**Author Contributions:** Conceptualization, J.I.O., S.O.J., R.P., A.A.O., M.O.A. and O.L.F.; methodology, J.I.O., S.O.J., R.P., A.A.O., M.O.A. and O.L.F.; investigation, J.I.O. and A.A.O.; writing—original draft preparation, J.I.O. and A.A.O.; writing—review and editing, J.I.O., S.O.J. and R.P.; supervision, S.O.J. and R.P.; project administration, J.I.O. and S.O.J.; funding acquisition, R.P. All authors have read and agreed to the published version of the manuscript.

**Conflicts of Interest:** The authors declare no conflict of interest.

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