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Analysis of Mechanical Durability, Hydrophobicity, Pyrolysis and Combustion Properties of Solid Biofuel Pellets Made from Mildly Torrefied Biomass

Kanageswari Singara veloo, Anthony Lau * and Shahab Sokhansanj

Biomass and Bioenergy Research Group, Department of Chemical and Biological Engineering, University of British Columbia, Vancouver, BC V6T 1Z3, Canada; kanages3@student.ubc.ca (K.S.v.); shahab.sokhansanj@ubc.ca (S.S.)

* Correspondence: anthony.lau@ubc.ca

Abstract

The production of solid biofuels from torrefied biomass holds significant potential for renewable energy applications. Durable pellet formation from severely torrefied biomass is hindered by the loss of natural binding properties, yet studies on mild torrefaction that preserves sufficient binding capacity for pellet production without external binders or changes to die conditions remain scarce. This paper investigated the production of fuel pellets from torrefied biomass without using external binders or adjusting pelletization parameters. Experiments were conducted using a mild torrefaction temperature (230 °C and 250 °C) and shorter residence time (10, 15, and 30 min). The torrefied materials were then subjected to pelletization using a single-pellet press; and the influence of torrefaction on the mechanical durability, hydrophobicity, and fuel characteristics of the pellets was examined. Results indicated that the mass loss ranging from 10 to 20% among the mild torrefaction treatments was less than the typical extent of mass loss due to severe torrefaction. Pellets made from torrefied biomass (torrefied pellets) had improvement in the hydrophobicity (moisture resistance) when compared to pellets made from untreated biomass (untreated pellets). Improved hydrophobicity is important for storage and transportation of pellets that are exposed to humid environmental conditions, as it reduces the risk of pellet degradation and spoilage. Thermogravimetric analysis of the pyrolysis and combustion behaviour of torrefied pellets indicated the improvement of fuel characteristics in terms of a much higher comprehensive pyrolysis index and greater thermal stability compared to untreated pellets, as evidenced by the prolonged burnout time and reduced combustion characteristics index. Residence time had a more significant impact on pellet durability than temperature, but the durability of the torrefied pellets was lower than that of the untreated pellets. Further research is required to explore the feasibility of producing binder-free durable pellets under mild torrefaction conditions. Overall, the study demonstrated that mild torrefaction could enhance the fuel quality and moisture resistance of biomass pellets, offering promising advantages for energy applications, despite some trade-offs in mechanical durability.

Keywords: pelletization; mild torrefaction; pellet quality; fuel characteristics; kinetic parameters

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1. Introduction

Excessive reliance on fossil fuels to meet the growing energy demand driven by urbanization and industrialization has led to significant challenges, including climate change, greenhouse gas emissions, rising global temperatures, and various environmental

issues [1,2]. As a result, there is an urgent need to adopt alternative, sustainable energy sources to reduce fossil fuel dependency and meet decarbonization mandates. Lignocellulosic biomass residues hold promise as a renewable energy source that can address both energy needs and environmental concerns caused by fossil fuel overuse [1].

Raw biomass residues present several challenges in handling, transport, storage, and utilization, due to their high moisture and ash content, low bulk density, irregular shapes, and low heating value [3,4]. Additionally, biomass harvesting sites such as forests and agricultural lands are often located far from industrial and residential areas, necessitating significant logistical efforts for transportation and storage [4,5]. To overcome these challenges, biomass waste can undergo pretreatment processes such as torrefaction and pelletization, which result in a high-energy-density, brittle solid biofuel known as torrefied pellets [6–8]. Torrefaction involves heating biomass to 200–300 °C in an oxygen-free environment [9,10]. This process enhances the heating value, hydrophobicity, and grindability of biomass while lowering its oxygen-to-carbon (O/C) ratio, making it suitable for combustion and gasification applications [7]. Moreover, pyrolysis as a key thermochemical conversion technology decomposes biomass into biochar, bio-oil, and combustible gas, catering to various energy demands. Using torrefied biomass in pyrolysis offers notable advantages over raw biomass, including higher conversion efficiency and improved product quality [11,12]. It yields bio-oils with higher carbon content, lower moisture, and improved aromatic and calorific properties compared to raw biomass [11–13]. In combustion applications, torrefied biomass has been successfully used in pulverized coal boilers without compromising efficiency or stability [14,15]. Co-firing with coal in existing plants has also been achieved using modified burners and mills, with minimal operational issues [8,16]. To date, torrefied biofuel has been demonstrated to be a cleaner biofuel for biomass pyrolysis, combustion, and gasification [17,18].

Pelletization is a physical pretreatment process that increases bulk density and reduces storage and transportation costs [5,8,19,20]. Conventional wood pellets have limitations for co-firing, due to their fibrous and hydrophilic nature, as well as their relatively low energy density compared to coal. This is partly due to their higher oxygen content, which is 1.7–1.9 times that of coal [6]. Combining torrefaction and pelletization offers a drop-in replacement for coal, reducing costs associated with handling, shipping, and storage, and is compatible with existing pellet industry infrastructure.

Despite these advantages, producing torrefied pellets poses challenges, such as achieving high durability and density and maintaining acceptable production capacities [21]. Torrefied materials often require higher pressure for pellet extrusion from the pelletizer compared to untreated biomass, resulting in increased energy consumption and costs [22]. The use of binders, such as sawdust, lignin, or starch, is a common strategy to improve pellet durability. However, many binders are hydrophilic, which can compromise the hydrophobic characteristics of torrefied pellets and reduce their moisture resistance, leading to weathering issues [9,23]. Severely torrefied biomass (at 270–300 °C) can also degrade the natural binding properties of lignin, weakening inter-particle bonding and complicating pelletization [4,21,24,25]. Garcia et al. [26] conducted a comprehensive study on the pelletization of raw pine sawdust and its torrefied counterpart, produced after 1 h of torrefaction at 280 °C. They found that the mechanical durability of untreated pellets was 77%, which dropped significantly to 15% for the highly torrefied samples. A similar observation was reported by Shang et al. [27], who stated that the pellet tensile strength was reduced by 90% after severe torrefaction. Moreover, the torrefaction process leads to mass (dry matter) loss that may have adverse effects on the economics of the process.

There is a growing interest in mildly torrefied biomass (230–260 °C) that can retain sufficient binding properties for pelletization but without the need for external binders or

increased die temperature and pressure. Manouchehrinejad et al. [28] conducted a detailed techno-economic assessment of integrated torrefaction and pelletization systems, whereby wood residues were torrefied at 250, 270, and 290 °C for 30 min. The results showed that while higher torrefaction temperatures slightly increased the energy content of the pellets, they significantly reduced mass yield, from 87% at 250 °C to 75% at 270 °C, and further, to 53% at 290 °C. Consequently, the minimum selling price (MSP) rose from USD183/tonne at 250 °C to USD207/tonne at 270 °C (13% increase), and further to USD277/tonne at 290 °C (51% increase). These findings underscore the fact that a mild torrefaction temperature of 250 °C offers a more favorable trade-off between energy enhancement, product yield, and economic viability, making it a practical choice for pellet production.

Torrefaction below 230 °C is not desirable, due to the resulting low heating values, which undermine the benefits of the process. As reported by Molinari et al. [29], torrefying loblolly pine at 220 °C for 30 min could attain a higher heating value (HHV) of 19.5 MJ/kg db (dry basis), while torrefaction at 200 °C required 90 min to attain 21 MJ/kg. These findings highlight the necessity of somewhat higher torrefaction temperatures to achieve significant improvement in the heating value, thereby justifying the conduction of torrefaction at temperatures above 230 °C.

This study explores the production of pellets from mildly torrefied woody biomass without external binders. It follows from our earlier research, which examined the structural and compositional changes in torrefied loblolly pine residues and their suitability as a pelletization feedstock [30]. The focus of this paper is on the analysis of mechanical durability, hydrophobicity, and pyrolysis and combustion characteristics of the fuel pellets made from mildly torrefied woody biomass (loblolly pine) residue. Since there is limited research on the pyrolysis kinetics of torrefied biomass, this paper also aims to fill the knowledge gap by providing insights into the pyrolysis and combustion behavior of the torrefied pellets. The research results can provide important information for designing pyrolyzers and combustors that utilize torrefied biomass pellets as the fuel.

2. Materials and Methods

2.1. Materials

Ground samples of Loblolly pine (*Pinus taeda*) residues were received from the industry partner. The as-received moisture content of the samples averaged 9.0 wt%. Before undergoing torrefaction, the raw biomass was dried in a convection oven at 105 °C for 24 h, and the dried material was sealed in a glass jar until use.

2.2. Torrefaction Experiment

Torrefaction was conducted using a custom-built thin-layer thermogravimetric analyzer (macro TGA), as described in Kanageswari et al. [30], to produce torrefied woody biomass. Approximately 15 g of biomass feedstock was utilized for all the treatments in the experiment. A continuous nitrogen gas flow rate of 50 mL/min was maintained inside the macro TGA unit to ensure an inert atmosphere. The samples underwent torrefaction at various combinations of mild temperature (230 °C and 250 °C) and shorter residence time (10, 15, and 30 min). The torrefied biomass was compared with untreated biomass, as well as biomass torrefied at (270 °C, 30 min), which represents one of the severe torrefaction conditions. For each torrefaction treatment, mass loss is calculated as the ratio of the sample's change in mass after torrefaction to the sample's mass before torrefaction, and expressed in [%]. To quantify the torrefaction of biomass, a severity factor (SF) was employed to

combine the influences of temperature and time into a unified variable [8,9,31]. The SF used in this research is defined by Equation (1).

$$SF = Log\left[t.exp\left(\frac{T_H - T_R}{14.75}\right)\right] \quad (1)$$

where t is the residence time of the torrefaction in [min], T_H is the torrefaction reaction temperature in [°C], and T_R the reference temperature, most often 100 °C. Table 1 shows the SF values, indicating an increase in SF with temperature and time.

Table 1. Severity factor of torrefaction of loblolly pine residues.

Temperature (°C)	Time (min)	Severity Factor (*SF*)
230	10	4.8
230	15	5.0
230	30	5.3
250	10	5.4
250	15	5.6
250	30	5.9
270	30	6.5

2.3. Pelletization

For pelletization, single pellets were made using a single-pellet manual press, as shown in Figure 1a. Given the small scale of the torrefaction process, a single-pellet press was more suitable for pelletization. In lab-scale fuel pellet research that involves data analysis and model building, a single-pellet mechanical press is often used to make biomass pellets (<1 g) at a slow production rate [32]. This approach allows for precise control over pelletization parameters and is ideal for detailed experimental studies.

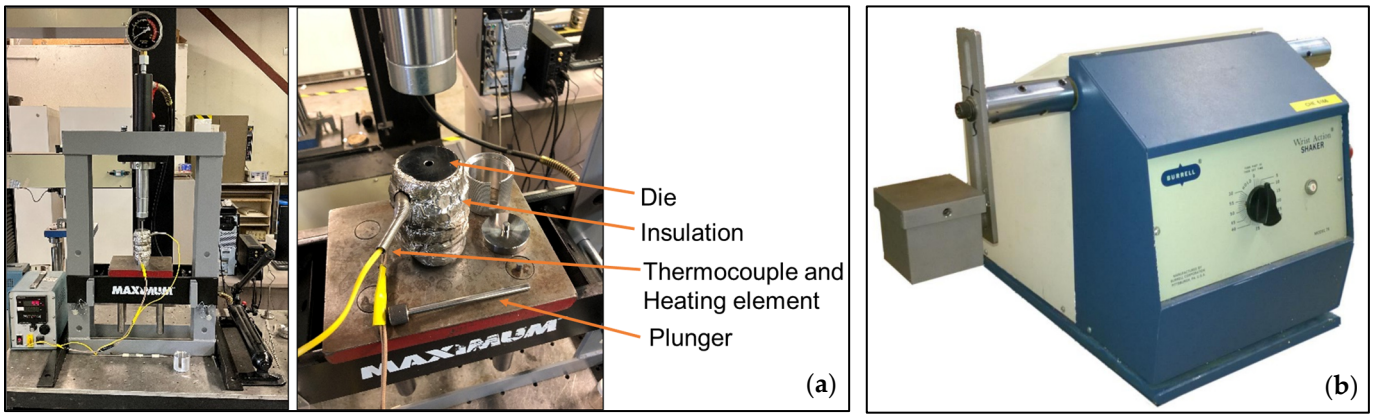


Figure 1. (**a**) Single-pellet manual press and pellet die set (**left**) and (**b**) single-pellet durability tester attached to the arm of a Burrel Model 75 Shaker (**right**).

In the biomass pelletization process, water acts as a natural binder and lubricant [33]. The moisture content of the torrefied biomass samples was adjusted to 12% wb (wet basis). Approximately 0.6 g of biomass was used to make one pellet. The die was heated to 100 °C using a heating tape and a temperature controller to simulate commercial pelleting conditions. Moreover, a 10 kN force was applied for 5 min duration. Ten pellets were made from both untreated and treated samples in the experiment.

2.4. Properties of Pellets

2.4.1. Pellet Density and Durability

The unit density of the pellet (PD) was determined as "mass per unit volume". Volume was calculated using the diameter and length of the pellet, which were measured with a digital caliper. The pellet durability (DB) was measured using a single-pellet durability tester developed in the BBRG Laboratory of Chemical & Biological Engineering at UBC [34]. The tester consisted of a cubic container with inner dimensions 60 × 60 × 60 mm. The upper housing section includes a removable lid for sample loading and unloading. A single biomass pellet and a metal pellet with dimensions of 6.3 mm in diameter and 12 mm in length were placed in the container. The metal box was attached to the arm of the laboratory shaker (Burrell Scientific Wrist Action—Model 75 Shaker, Burrell Scientific, Zelienople, PA, USA), as shown in Figure 1b. The shaker was operated at 416 rpm for 10 min. Subsequently, all contents were sieved through a screen with a size of 3.15 mm to separate the broken particles generated during the shaking process.

The single-pellet durability was calculated using Equation (2).

$$DB = \frac{m_a}{m_b} \times 100\% \quad (2)$$

where m_a is the mass of pellets retained on the sieve, m_b is the original mass of the pellets before shaking, and DB is the durability of the single pellet in percent.

2.4.2. Moisture Uptake

Hydrophobicity or moisture resistance was a critical parameter to consider during pelletization, as it affected ocean transportation and long-term storage. Firstly, moisture-free pellets were obtained by placing the pellets in an oven for 12 h at 105 °C. The dried pellets were then placed in a humidity chamber (Memmert HCP50, FRG, Germany), where the temperature and relative humidity were maintained at 40 °C and 90%, respectively. During the first 4 h, changes in pellet mass were recorded every 30 min. Later, the sample was weighed every 8 h and then left for 48 h to attain a constant moisture content. The final moisture content will be the equilibrium moisture content (EMC). The kinetics of moisture sorption are described by the following equation:

$$\frac{M - M_e}{M_i - M_e} = e^{-kt} \quad (3)$$

where M, M_e, and M_i are moisture content at time (t), EMC, and initial moisture content on dry basis, respectively; the coefficient k is the moisture absorption constant (1/min), and t is the exposure time (min).

2.5. Thermogravimetric Analysis (Pyrolysis)

The pyrolysis of untreated and torrefied loblolly pine pellets was conducted in a thermogravimetric analyzer (TA Instruments, TGA Model Q550) under inert conditions with nitrogen. Samples (about 10 mg in mass) were heated, and the biomass decomposition behavior was tracked by the following procedure: (1) a heating rate of 20 °C/min from ambient temperature to 105 °C, (2) isothermal for 10 min, (3) a heating rate of 10 °C/min from 105 °C to 800 °C for non-isothermal, and (4) isothermal for 10 min [35]. The comprehensive pyrolysis index (CPI) was calculated to quantitatively assess the pyrolysis reactivity of biomass [36]. The CPI is a synthetic parameter combining various aspects that include the decomposition rate and thermal characteristics of the material. It encompasses the

overall efficiency and effectiveness of the pyrolysis process [37,38], and it is calculated by the following equation:

$$CPI = \frac{D_{max}}{T_{max}\left(T_f - T_i\right)} \tag{4}$$

In Equation (4), D_{max} is the maximum mass loss rate (%/min) obtained from the peak value of the derivative thermogravimetric (DTG) curve. T_{max} is the temperature that corresponds to D_{max}. T_i is the initial devolatilization temperature, which corresponds to a weight loss within 5% of the final weight loss; T_f is the final pyrolysis temperature.

Pyrolysis Kinetic Analysis

Pyrolysis kinetic analysis was conducted to investigate the pyrolysis characteristics of untreated and torrefied loblolly pine pellets, following the methods of Yu et al. [35] and Khairy et al. [39]. The decomposition rate is described by Equation (5).

$$\frac{d\alpha}{dt} = k(T)f(\alpha) \tag{5}$$

where α is the mass decomposition defined by Equation (6) and constant k (T) is the temperature-dependent reaction rate constant defined by the Arrhenius Equation (7).

$$\alpha = \frac{m_i - m_T}{m_i - m_f} \tag{6}$$

$$k(T) = A\exp\left(-\frac{E}{RT}\right) \tag{7}$$

where m_i, m_T, and m_f are the initial mass, sample mass at temperature T and final mass in mg, respectively. A is the pre-exponential factor, E (kJ/mol) is the activation energy, and R (0.008314 kJ/mol·K) is the universal ideal gas constant. β is the constant heating rate.

$$d(t) = \beta\, d(T) \tag{8}$$

Substituting Equations (6)–(8) into Equation (5) gives

$$\int_0^1 \frac{d\alpha}{f(\alpha)} = \frac{A}{\beta}\int_0^T \exp\left(-\frac{E}{RT}\right)dT = g(\alpha) \tag{9}$$

To analyze the kinetic data obtained from thermogravimetric curves, the graphical method developed by Coats and Redfern [40] was applied, using Equation (10).

$$In\left[\frac{g(\alpha)}{T^2}\right] = In\left[\frac{AR}{\beta T\left(1 - \frac{2RT}{E}\right)}\right] - \frac{E}{RT} \tag{10}$$

Since $2RT/E << 1$, Equation (10) can be rewritten as

$$In\left[\frac{g(\alpha)}{T^2}\right] = In\left[\frac{AR}{\beta T}\right] - \frac{E}{RT} \tag{11}$$

In terms of reaction kinetics, $f\ (\alpha)$ is described as

$$f(\alpha) = (1 - \alpha)^n \tag{12}$$

where n is the reaction's order. This nth-order reaction is the most prevalent type for biomass, indicating that the reaction rate is directly related to the remaining reactants.

According to Wang [41], the reaction order model (n = 1) represents the chemical reaction accurately, so

$$g(\alpha) = -\ln(1-\alpha) \tag{13}$$

With Equations (9) and (11) can be rewritten as

$$In\left[\frac{-\ln(1-\alpha)}{T^2}\right] = In\left[\frac{AR}{\beta T}\right] - \frac{E}{RT} \tag{14}$$

A plot of $\ln[-\ln((1-\alpha)/(T^2))]$ versus $(1/T)$ gives a straight line with slope of $(-E/R)$ and intercept of $\ln(AR/\beta E)$. From the slope of the line, the activation energy (E) can be estimated, while the pre-exponential factor (A) can be determined from the intercept.

2.6. Thermogravimetric Analysis (Combustion)

Combustion tests were carried out using the thermogravimetric analyzer in oxidative conditions using air and employing a procedure similar to that used for pyrolysis tests. The intersection technique, known for its simplicity, was used to determine ignition temperature (T_i), burnout temperature (T_f) and maximum burning rate (R_{max}), by analyzing TGA and DTG combustion profiles [42]. The burnout temperature (T_f) was the temperature at which fuel conversion achieved 99%. Using Equations (15) and (16), the combustion characteristic parameters were calculated as average burning rate (R_{mean}) and combustion characteristic index (S_n), which are important indicators of combustion performance [43].

$$R_{mean} = \beta[\frac{\alpha_i - \alpha_f}{T_f - T_i}] \tag{15}$$

$$S_n = \frac{R_{max}R_{mean}}{T_i^2 T_f} \tag{16}$$

where α_i is the percentage of the remaining samples corresponding to the ignition temperature, and α_f is the percentage of the remaining samples corresponding to burnout temperature. Moreover, burnout time (t_b) is measured as a vital combustion parameter, and t_b is the time for the dried fuel to begin losing weight until the end of combustion, when the weight stabilizes [42].

3. Results and Discussion

For the torrefaction experiment, results showed that the mass loss varies directly with torrefaction severity index SF, ranging from 10% for the treatment (230 °C, 10 min; SF = 4.8) to 20% for the treatment (250 °C, 30 min; SF = 5.9). By comparison, the severe torrefaction treatment (270 °C, 30 min; SF = 6.5) led to 38% mass loss. A bigger mass loss of 34% and 51% was reported in a study when softwood shavings were subjected to severe torrefaction for 60 min at 270 °C and 300 °C, respectively [20]. Results that are pertinent to pellet characteristics, and pyrolysis and combustion behaviour of torrefied pellets, are presented in the following sections.

3.1. Analysis of Pellet Characteristics

The effects of torrefaction on the bulk density before and after compaction are listed in Table 2. Loose bulk density refers to the density of biomass particles, both in their as-received state and after undergoing torrefaction treatment [30]. Due to pelletization, the density of biomass was significantly increased. For instance, untreated biomass typically possessed a bulk density of just 0.27 g/cm^3, whereas the density of individual pellets was higher, at 1.25 g/cm^3. The densities for SF = 5.9 and SF = 6.5 treatments dropped to 1.19 g/cm^3 and 1.17 g/cm^3, respectively. This is primarily due to the dry matter loss that

occurred during torrefaction, which is consistent with the single-pellet density reported by Li et al. [44]. Moreover, Peng et al. [24] observed that the density of torrefied sawdust pellets was lower than that of the control pellets produced under the same conditions, whereas torrefied sawdust before pelletization had a greater density than raw biomass.

Table 2. Loose bulk density and single-pellet density for untreated and torrefied samples.

SF	Loose Bulk Density (g/cm^3)	Single-Pellet Density (g/cm^3)
Untreated	0.31 ± 0.01	1.25 ± 0.01
4.8	0.30 ± 0.01	1.23 ± 0.02
5.0	0.30 ± 0.01	1.21 ± 0.04
5.3	0.30 ± 0.01	1.21 ± 0.03
5.4	0.28 ± 0.01	1.22 ± 0.02
5.6	0.29 ± 0.02	1.22 ± 0.03
5.9	0.29 ± 0.02	1.19 ± 0.04
6.5	0.21 ± 0.02	1.18 ± 0.02

Figure 2 shows the single-pellet durability of the untreated pellets and treated (torrefied) pellets. Data analysis using *t*-test (Microsoft Excel V16.71) assessed significant variations in pellet durability across torrefaction treatments. The analysis assumed equal variances between samples, with a significance level of 0.05. Results showed that the durability of all treated pellets was less than that of the pellets from untreated biomass (93 ± 4%), and the differences in durability are statistically significant (*p*-value < 0.05). At torrefaction temperatures of 230 °C and 250 °C, as residence time increased from 10 min to 30 min, pellet durability decreased from 63 ± 4% to 50 ± 5%, and from 63 ± 6% to 53 ± 5%, respectively, illustrating that increased treatment time led to a decrease in the single-pellet durability.

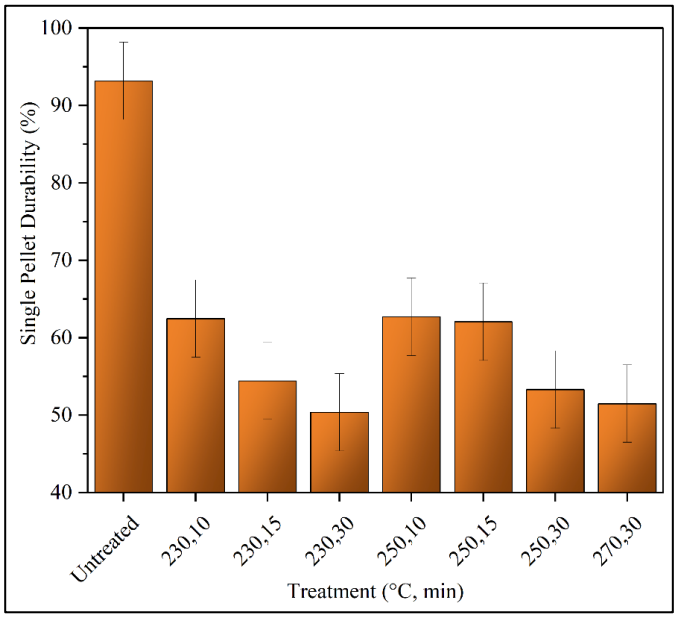


Figure 2. Single-pellet durability for untreated and torrefied pellets.

Interestingly, a significant difference in durability (*p*-value < 0.05) was noted between samples torrefied at 250 °C, 10 min; SF = 5.4 vs. 230 °C, 30 min; SF = 5.3. This difference aligned with broader peaks at 2990–3670 cm^{-1} (indicating hydroxyl -OH groups), where samples torrefied at SF = 5.4 exhibited higher durability from FTIR analysis. This suggests that sufficient -OH groups remained on the wood polymer chains to form strong inter-

particle bonds during pelletization. The higher pellet durability observed at SF = 5.4 is also supported by the crystallinity index (*CrI*), where samples treated at this condition had lower *CrI* values than those torrefied at SF = 5.3, suggesting the presence of amorphous cellulose can facilitate pelletization by enhancing bonding. Our results agree with those of Stelte et al. [21], in that improved pelletizing properties can be achieved when biomass (wheat straw) was torrefied at temperatures up to 250 °C. However, heating above this temperature did not enhance pelletizing of torrefied biomass, coinciding with the reported trend of pellet properties by Liu et al. [43].

The response surface methodology (RSM) was used to further explore the interactions between torrefaction temperature and time and their effects on pellet durability. A first-order equation (Equation (17)) was generated to predict single-pellet durability (DB), based on the two key factors: torrefaction temperature (X_1) and treatment time (X_2). Analysis of variance (ANOVA) was performed to assess the statistical significance of the factors and the predicted durability. Results as summarized in Table 3 include the sum of squares, degrees of freedom (df), mean squares, *F*-values, and *p*-values for both responses.

$$DB(\%) = 56.72 + 1.79X_1 - 5.12X_2 \quad (17)$$

where *DB* is single-pellet durability, while X_1 and X_2 are temperature and time. respectively.

Table 3. ANOVA analysis for the response surface method (RSM) model for single-pellet durability as a function of torrefaction temperature and time.

Source	Sum of Squares	df	Mean Squares	*F*-Value	*p*-Value
Equation (17)	132.9	2	66.5	11.53	0.0391
X_1-Temperature	19.33	1	19.33	3.35	0.1644
X_2-Time	113.6	1	113.6	19.71	0.0212

The ANOVA for pellet durability showed that the first-order equation can predict single-pellet durability effectively. Torrefaction time significantly impacted pellet durability, with a *p*-value of 0.0212, suggesting that longer torrefaction times significantly reduce pellet durability. However, torrefaction temperature did not show a significant effect (p = 0.1644), indicating that temperature variations within the tested range did not strongly influence the pellet durability.

The durability required by the international standard "ISO/TS 17225-2" [45] for conventional wood pellets is based on measurements using a "tumbler durability tester". No such standard has been established for torrefied biomass pellets, and the single-pellet durability tester is not recognized in an international standard. Hence, the measured single-pellet durability (*DB*) needs to be checked against the ISO standard requirements for conventional wood pellets. To this end, a linear regression equation (R^2 value of 0.94) that was developed by Yu et al. [26] in the BBRG Laboratory for tumbler durability vs. single-pellet durability of wood pellets is applicable. As shown in Table 4, based on calculations using the equation, the tumbler durability for untreated loblolly pine pellets was 96.8%, while the mildly treated loblolly pine pellets ranged from 82.6% to 86.7%. These calculated tumbler durability values for pellets with 12% wb moisture content are in line with the measured values of 80–90% reported by Larsson et al. [46] for pellets with moisture content of 11–15% wb. The calculated durability values are also comparable with the experimental results of tumbler durability (>85%) observed by Rudolfsson et al. [47] for pellets with 12–14% wb moisture content.

Table 4. Single-pellet durability and tumbler durability of pellets made from mildly torrefied loblolly pine residues.

SF	Single-Pellet Durability (%) [a]	Tumbler Durability (%) [b]
Untreated	93.2	96.8
4.8	62.5	86.6
5.0	54.5	84.0
5.3	50.4	82.6
5.4	62.7	86.7
5.6	62.1	86.5
5.9	53.3	83.6
6.5	51.5	83.0

[a] Data obtained from experimental results of this study. [b] Calculated values using linear regression equation [32].

Figure 3 shows the moisture uptake of untreated and torrefied pellets at different torrefaction operation conditions. The lower the moisture uptake, the higher the moisture resistance. The resistance against moisture represents the hydrophobicity of fuel pellets. During the experiment, the untreated pellets were observed to enlarge and distort quickly in the humidity chamber. In contrast, the torrefied pellets retained good firmness, but, eventually, they also swelled and began to disintegrate after several hours in the humid chamber.

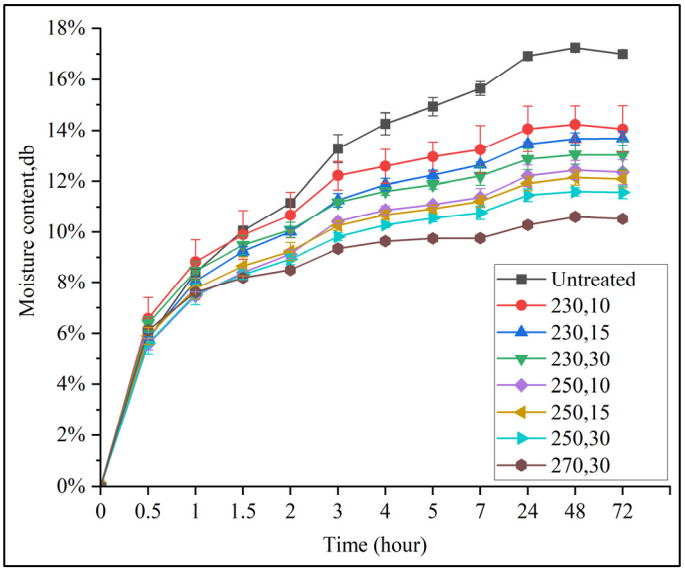


Figure 3. Moisture uptake of untreated and torrefied pellets at 90% relative humidity and 40 °C temperature in a humidity chamber.

A similar magnitude of adsorption was observed for the first 4 h, where the moisture uptake was significantly high and reached around 87–93% of equilibrium moisture content (EMC). The moisture content of biomass continued to increase over the next 4 h, reaching around 97–99% of their EMC. The biomass moisture content at 48 h was essentially the EMC, as the increase became almost negligible beyond that time. EMC is expressed in [%db (dry basis)] unit. During the moisture uptake test, the mass of the solid remained constant; hence, any change in moisture content directly corresponds to the mass of water adsorbed. Pellets made from biomass torrefied at the lowest severity, SF = 4.8 experienced a 17% reduction in EMC, reaching 14%db, which corresponds to 0.14 g of water absorbed per gram of dry biomass. By comparison, the hydrophobicity of the pellets increased significantly, with more than 25% reduction in EMC, reaching 12% for pellets made from biomass torrefied at SF = 5.4. These reductions indicate that the hydrophobicity of biomass

pellets was improved, owing to the increase in treatment severity. The primary reason for this is that many functional groups decomposed during the torrefaction process, as confirmed by FTIR analysis. The torrefied pellets exhibited a significant reduction in the number of polar and strongly absorbent oxygen-containing groups (such as O-H, C-O, and C-O-C) that are present in the biomass [12]; subsequently, the moisture adsorption capacity of the torrefied pellets decreased. The data obtained highlights not only increased energy density, but also enhanced storage stability. The findings are in agreement with those reported by other researchers [9,10,40].

3.2. Pyrolysis Behaviour of Torrefied Pellets

The derivative thermogravimetric (DTG) curves (Figure 4) show the pyrolysis behavior of untreated and torrefied loblolly pine pellets, highlighting how increasing torrefaction severity affected thermal degradation patterns. The pyrolysis characteristic parameters of untreated and torrefied loblolly pine pellets, as listed in Table 5, were determined from the DTG curves. The residue weight at 800 °C exhibited a progressive increase with the severity of torrefaction. Untreated samples had the lowest residue weight, at 15.5%. This value increased to 19.4% and 22.7% for samples torrefied at SF = 4.8 and SF = 5.4, respectively. The highest residue weight of 31.4% was observed in samples torrefied at SF = 6.5. This trend suggested that higher torrefaction intensity increased thermal stability, resulting in greater char residue due to cross-linking reactions [48–50].

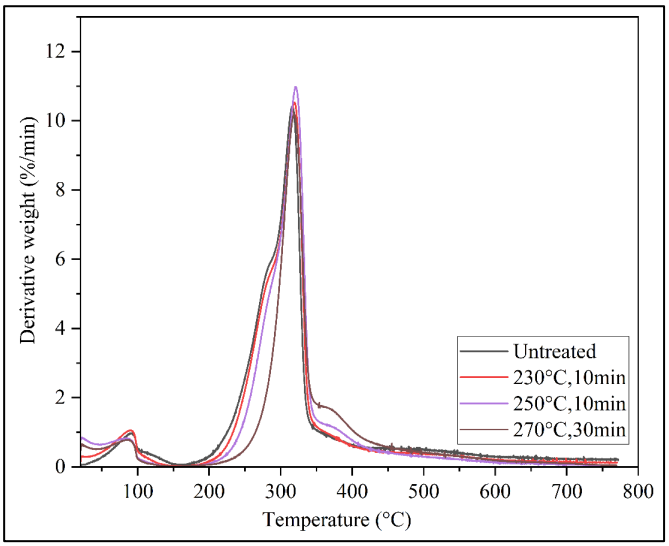


Figure 4. Derivative thermogravimetric (DTG) curves to represent degradation rate of untreated and torrefied loblolly pine pellets in an inert (N_2) environment.

Table 5. Pyrolysis characteristics of untreated and torrefied loblolly pine pellet.

SF	Pyrolysis Parameters					Kinetics Parameters		
	Residue Weight % at 800 (°C)	D_{max} (%/min)	T_{max} (°C)	T_i (°C)	CPI ($\times 10^{-4}$%/(min °C^2))	E (kJ/mol)	A ($\times 10^5$/min)	R^2
Untreated	15.5	9.8	315.5	214.3	1.6	27.0	0.2	0.9879
4.8	19.4	10.3	318.4	234.6	2.0	30.9	0.5	0.9902
5.0	21.0	10.5	323.5	247.8	2.3	36.1	1.6	0.9938
5.3	22.5	11.2	324.4	251.2	2.4	36.7	1.8	0.9886
5.4	22.7	11.0	325.2	251.3	2.4	36.4	1.7	0.9892
5.6	22.9	12.1	322.7	258.0	2.7	39.9	3.6	0.9892
5.9	24.6	11.6	324.3	254.5	2.8	39.7	3.3	0.9813
6.5	31.4	10.0	319.5	265.7	3.0	43.0	6.0	0.9808

The peak intensity at 234 °C indicated the maximum degradation rate of hemicellulose. This DTG hemicellulose peak decreased in intensity for torrefied biomass pellets, signifying substantial destruction of the hemicellulose structure after torrefaction. This shoulder was eliminated in severely torrefied biomass. The initial devolatilization temperature (T_i) exhibited a clear trend of increasing with increasing torrefaction severity, attributed to the reduction in volatile hemicellulose components. For pellets torrefied at SF = 4.8, T_i was higher, at 234.6 °C, compared to 214.3 °C for untreated pellets. The highest T_i, observed at the most severe torrefaction level (SF = 6.5), reached 265.7 °C. This increase showed that deeply torrefied biomass required a higher temperature to initiate decomposition. Ru et al. [51] studied the pyrolysis behaviour of torrefied fast-growing poplar at 200–300 °C for 30 min; they also observed an increase in T_i of the samples with increasing severity of torrefaction.

The highest peak of the DTG curve corresponds to cellulose decomposition that occurred at 275–350 °C. The D_{max} value, which represents the peak decomposition rate of cellulose, first increased with mild torrefaction. Starting at 9.8%/min for untreated pellets, D_{max} rose to 10.3%/min and 11%/min at SF = 4.8 and SF = 5.4, respectively, as the proportion of cellulose content increased due to hemicellulose degradation. The decomposition rate peaked at 12.1%/min for pellets torrefied at SF = 5.6, before descending, indicating that moderate torrefaction enhanced the breakdown rate by exposing reactive sites. T_{max} gradually increased with torrefaction severity, moving from 315.5 °C for untreated pellets to a range of 318–325 °C for mildly torrefied samples, while it was 319.5 °C for SF = 6.5.

This was confirmed by a reduction in the crystallinity index (*CrI*) from 36.2% at SF = 4.8 to 34.4% at SF = 5.4. The cellulose peak intensity then increased again at SF = 5.6, where *CrI* also rose to 38.5%, indicating a structural change in the remaining cellulose. This observation is similar to those made by Yu [52]. Moreover, Li et al. [36] observed a similar trend for torrefied pine pellets with higher severity (300 °C), having the lowest D_{max} and T_{max} reported as a result of the formation of active cellulose with a lower degree of polymerization at this temperature.

The comprehensive pyrolysis index (CPI) of loblolly pine pellets (Table 5) was promoted by torrefaction, which agrees with findings by Li et al. [30]. The CPI value increased with torrefaction, indicating that the pyrolysis reaction progresses more rapidly and efficiently as torrefaction temperature and time increase. Untreated pellets had a CPI of 1.6×10^{-4}%/(min°C^2), which rose to 2×10^{-4}%/(min°C^2) and 2.4×10^{-4}%/(min°C^2) for pellets treated at 230 °C and 250 °C for 10 min, respectively. The CPI steadily increased to 2.8×10^{-4}%/(min°C^2) for pellets treated at (250 °C, 30 min; SF = 5.9), suggesting that torrefied materials decomposed more readily under pyrolysis conditions. By comparison, the CPI of pellets treated at 270 °C, 30 min; SF = 6.5 was 3×10^{-4}%/(min°C^2), which is comparatively higher than that of mildly torrefied pellets.

The kinetic parameters during the pyrolysis process are listed in Table 5, and are calculated for the main temperature range 190–315 °C [35]. Activation energy (E) is a fundamental kinetic parameter that quantifies the minimum energy required to initiate decomposition reactions within biomass during thermal degradation [38]. For untreated biomass, the E value was 27 kJ/mol, whereas biomass treated at SF = 4.8 showed a 14.4% increase in E, reaching 30.9 kJ/mol. As the torrefaction temperature and residence time increased, the E value increased further, to 30.9–39.7 kJ/mol. The highest E value of 43 kJ/mol was observed for biomass treated at SF = 6.5, indicating that the reactive torrefied biomass would require more energy to decompose, due to its carbonized structure. The pre-exponential factor (A) followed a similar trend, increasing with higher torrefaction temperatures and longer residence times. Its value was 0.2×10^5 min^{-1} for untreated pellets and 0.5×10^5 min^{-1} for treatment at SF = 4.8, and it increased further with longer

durations. When increasing the temperature from 230 °C to 250 °C for 30 min, the pre-exponential factor almost doubled, and the highest value of 6.0×10^5 min^{-1} was observed for the most severely torrefied pellets, at SF = 6.5. This trend supported the increase in reactivity associated with higher CPI values.

However, there was a slight dip in the E and A values for pellets torrefied at SF = 5.4. This could be due to the larger breakdown of the crystalline region and cleavage of glycosidic bonds during this torrefaction treatment, thus facilitating subsequent pyrolysis [53]. The regression coefficient (R^2) values were consistently high (0.98–0.99), affirming good fit for the kinetic model across torrefaction treatments. Lu et al. [48] reported that the activation energy for non-torrefied *Cryptomeria japonica* wood was 76.8 kJ/mol, compared to the range of 105–109 kJ/mol for torrefied samples at 250 and 300 °C for 1 h.

Ren et al. [49] observed similar results in their study of the thermal decomposition behaviour of torrefied Douglas fir sawdust pellets at 250–300 °C for 10 min. They reported that T_i increased with increasing torrefaction temperature, but the height of the peak for torrefied biomass revealed the decreasing trend with the severity of torrefaction. Moreover, they reported that the activation energy values for torrefied Douglas fir sawdust ranged from 198 to 195 kJ/mol, which was a bit lower than the 204 kJ/mol observed for non-torrefied biomass, and which are at least 3–4 times the activation energy in our study. This difference may stem from variations in feedstock reaction models and their associated kinetic parameters [54].

3.3. Combustion Behaviour of Torrefied Pellets

The untreated and torrefied pellets were subjected to non-isothermal thermogravimetric tests in air, to evaluate their combustion behavior (Figure 5). All the combustion DTG curves displayed a similar peak distribution. A sharp peak in the DTG curve indicated maximum weight loss, followed by a decline as the reaction concluded. During the early stages of combustion, the thermal decomposition of hemicellulose released volatiles, which superimposed with cellulose degradation, creating a peak between 280 and 310 °C. This observation is consistent with Jia [55], who reported a prominent peak in the DTG curves of five different biomass pellets.

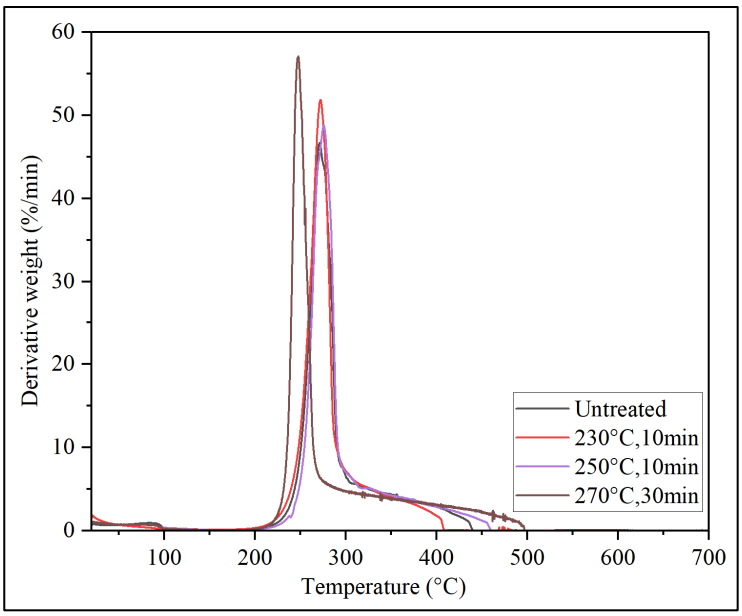


Figure 5. DTG curves to represent degradation rate of untreated and torrefied loblolly pine pellets in an oxidative environment.

The volatile ignition consumed heat and influenced the combustion process. The mean and maximum combustion rates primarily occurred within the volatile matter combustion region [56]. The DTG curve for pellets torrefied at (270 °C, 30 min) was slightly shifted to a lower temperature, while the other samples had similar positions. Bach and Tran [57] observed that spruce torrefied at (275 °C, 60 min) also shifted to a lower temperature in the DTG curve, which may result from partial degradation of cellulose and a lower devolatilization peak.

Table 6 summarizes the combustion behavior parameters of untreated and torrefied loblolly pine residues. The combustion ignition temperature (T_i) for mildly torrefied loblolly pine pellets remained relatively consistent, ranging from 264 °C to 268 °C, compared to the untreated sample, at 263 °C. Generally, T_i was higher than for the untreated sample, except for the pellets torrefied at 250 °C and 270 °C for 30 min, which had T_i values of 260 °C and 252 °C, respectively. Junga et al. [58] also observed that T_i of the raw material was comparable to that of nearly all torrefied samples.

Table 6. Combustion characteristics of untreated and torrefied loblolly pine pellet.

SF	T_i (°C)	T_f (°C)	R_{mean} (%/min)	R_{max} (%/min)	Burnout Time (min)	S_n ($\times 10^{-5}$%^{2}/(min^2°C^3))
Untreated	262.9	407.2	12.7	56.7	26.7	2.6
4.8	267.5	411.4	12.7	52.2	26.5	2.3
5.0	267.9	427.8	8.2	45.9	30.3	1.1
5.3	264.0	446.4	9.8	44.4	28.8	1.4
5.4	266.5	437.9	10.6	46.8	28.0	1.6
5.6	266.0	454.9	9.7	48.9	29.0	1.5
5.9	259.3	469.7	8.6	45.2	29.9	1.2
6.5	251.6	498.5	7.4	56.9	30.8	1.3

The burnout temperature (T_f) increased with torrefaction severity, particularly at higher torrefaction temperatures and longer durations, as compared to the untreated pellet. Specifically, at 30 min residence time, T_f increased by approximately 10% at 230 °C, 15.3% at 250 °C, and 22.4% at 270 °C, compared to the untreated sample. This trend highlights the combined effect of temperature and time on the combustion characteristics as the biomass becomes more thermally stable. A previous study also reported that torrefied biomass had higher ignition and burnout temperatures than untreated biomass, reducing the risk of spontaneous combustion and spoilage while enhancing combustion stability in biomass fuel [59]. Under inert conditions, increasing torrefaction severity facilitates C-H bond polymerization into C-C bonds and the transformation of small reactive aromatics into larger, more stable structures. This enhanced carbon chain length and biomass stability, leading to delayed ignition and burnout temperatures during combustion [59].

In general, burnout time was also found to be longer with greater torrefaction severity, as indicated by the increase in T_f, suggesting that the biomass becomes more thermally stable and takes longer to burn completely. Untreated biomass had the shortest burnout time of 27 min, whereas the most severe condition (SF = 6.5) resulted in the longest burnout time of 31 min. Moreover, the maximum burning rate (R_{max}) decreased with higher torrefaction temperatures and longer durations, though an exception was noted at SF = 6.5, whereby R_{max} increased to 56.9%/min, which was also observed for the untreated sample. Liu et al. [43] demonstrated that torrefaction of wheat straw for high-quality pellet production led to a continuous decrease in R_{max} with increasing temperature, indicating progressive hemicellulose decomposition.

The combustion characteristic index, S_n, accounts for the difficulty of ignition, pace of combustion, and extent of burnout [43]. A higher S_n index, which indicates more robust

combustion and faster sample consumption, was obtained for the untreated biomass pellets, with a value of $2.6 \times 10^{-5}\%^2/\text{min}^2\ ^\circ\text{C}^3$. For pellets torrefied at SF = 4.8, the S_n decreased to $2.3 \times 10^{-5}\%^2/\text{min}^2\ ^\circ\text{C}^3$, compared to untreated pellets. However, treatments beyond this severity resulted in more than a 42% drop in combustibility, with S_n values ranging from $1.1 \times 10^{-5}\%^2/\text{min}^2\ ^\circ\text{C}^3$ to $1.6 \times 10^{-5}\%^2/\text{min}^2\ ^\circ\text{C}^3$ after being torrefied with various combinations of temperature and time. Although no specific trend was observed regarding the effect of torrefaction temperature and time on S_n, the most significant reductions in S_n were generally observed at higher temperatures and longer durations.

Park et al. [60] conducted torrefaction on three types of agricultural and forestry biomass to enhance fuel and combustion characteristics, and reported that increasing torrefaction temperature led to a decline in the combustion index, indicating a deterioration in combustion performance with progressive torrefaction. The reactivity of combustion is influenced by the interplay between chemical reactions and diffusion control. Elevated torrefaction temperatures improve the pore structure of torrefied biomass, facilitating diffusion control. However, this also leads to the development of microcrystalline structures and a reduction in active sites, which ultimately decreases the combustion rate. Results of our study on torrefied loblolly pine pellets' combustion behaviour agreed with those of Sarker et al. [10], who reviewed the combustion parameters of torrefied biomass and concluded that, compared to untreated biomass, torrefied biomass exhibited higher ignition and burnout temperatures, a prolonged burnout time, and reduced reactivity.

4. Conclusions

This study investigated the effects of mild torrefaction temperature (230 °C and 250 °C) and shorter residence times (10, 15, and 30 min) on the mechanical durability, hydrophobicity (moisture resistance), and fuel characteristics of pellets made from loblolly pine residue. The goal was to minimize the mass loss associated with torrefaction while producing pellets, without using external binders or adjusting pelletization parameters. The key findings may be summarized as follows.

Mass loss, which ranged from 10 to 20% among the torrefaction treatments, was less than the typical mass loss of more than 25–30%, when raw biomass was subjected to severe torrefaction conditions. The hydrophobicity of the pellets increased significantly, as reflected in the large reduction in the equilibrium moisture content (EMC). This is important for storage and transportation when pellets are exposed to humid environmental conditions, as it reduces the risk of pellet degradation and spoilage. Thermogravimetric analysis of the pyrolysis behaviour of torrefied pellets revealed a clear trend of increasing initial devolatilization temperature and higher comprehensive pyrolysis index (CPI) with torrefaction severity, indicating that the pyrolysis reaction progresses more efficiently. Moreover, the thermal stability of the pellets was enhanced with reduced risk of spontaneous combustion, as evidenced by the prolonged burnout time and reduced combustion characteristic index (S_n) of the torrefied pellets. Residence time had a more significant impact on pellet durability than torrefaction temperature; however, the durability of the pellets made from torrefied biomass under the various torrefaction process conditions was lower than that of the pellets made from untreated biomass. Overall, the study demonstrated that mildly torrefied biomass could produce pellets that have some significant advantages over untreated pellets. Further research is needed to explore the feasibility of producing binder-free durable pellets from mildly torrefied biomass.

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editing. S.S.: Conceptualization, project administration, resources, supervision. All authors have read and agreed to the published version of the manuscript.

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Abbreviations

CrI	Crystallinity index
CPI	Comprehensive pyrolysis index
db	Dry basis
DTG	Derivative thermogravimetry
df	Degrees of freedom
EMC	Equilibrium moisture content
FTIR	Fourier-Transform Infrared
HHV	Higher heating value
MC	Moisture content
RSM	Response surface methodology
RH	Relative humidity
SF	Severity factor
S_n	Combustion characteristic index
TGA	Thermogravimetric analysis
wb	Wet basis
XRD	X-ray diffraction

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