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Quality Properties and Torrefaction Characteristics of Pellets: Rose Oil Distillation Solid Waste and Red Pine Sawdust

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Abstract: Two different biomass types, rose oil (*Rosa damascena* Mill.) distillation solid wastes (RDWs) and red pine sawdust (RPS), were pelletized in this study at different moisture and additives. The prepared pellets were also torrefied. This study revealed that the strength of the RPS and RDW pellets decreased as their moisture content increased in both their raw and torrefied forms. However, the tensile strength of the torrefied pellets increased with the increased binder ratio, which is similar to raw pellets. Compared to their raw form, the torrefied pellets generally had higher ash contents, fixed carbon contents, and higher heating values. As a result of torrefaction, the higher heating value of the RPS pellets increased from 17.51–18.80 MJ/kg to 20.20–21.73 MJ/kg, while the higher heating value of the RDW pellets increased from 17.42–18.54 MJ/kg to 19.13–20.92 MJ/kg. For the torrefied RPS and RDW pellets in this study, there was no statistically significant difference between initial moisture content and energy efficiency, energy density, or mass yield. On the other hand, energy density ratios in both the torrefied RPS and torrefied RDW pellets generally increased with increasing binder content. Furthermore, the torrefied pellets were found to be more stable in moisture absorption than the raw pellets.

Keywords: rose pulp; torrefaction; biomass; pellet; biofuel

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1. Introduction

The population growth, as well as the improvement of living standards as a consequence of technological development, has boosted energy consumption worldwide [1]. Based on the Energy Information Administration (EIA), the global energy requirement will increase by 50% and reach almost 900 quadrillion Btu by 2050 [2]. Herein, it is imperative to meet the increasing energy demands to satisfy the estimated global energy requirements in the future. Today, global energy demands comprise oil (30.9%), coal (26.8%), natural gas (23.2%), nuclear energy (5%), and renewable energy (14.1%) [3]. The percentage distribution values of these sources are expected to be altered due to the beginning of conventional fossil fuel depletion. Fossil fuels contribute to atmospheric pollution, respiratory tract disease, and global warming driven by greenhouse gas emissions (GHGs). Further, approximately 80%, 50%, and 30% of existing coal, gas, and oil reserves, respectively, should remain under the soil to achieve one of the Paris Agreement targets, maintaining the global mean temperature limit to increase 2 °C [4]. Furthermore, one of the goals of the European Union is to increase the proportion of renewable energy sources to 20% by 2020 [5]. Therefore, biomass could be one of the good alternatives among energy sources to maintain this temperature increase below 2 °C due to being carbon neutral and meeting energy needs [6].

Biomass is a continuous energy source due to its abundant quantity and easy-to-reach sources in nature [7,8]. Additionally, there is no extra initial investment cost compared to fossil fuels. Biomass includes agricultural residues, wood and wood wastes, municipal solid wastes, and animal manure. Since it has high moisture content and dust levels and low bulk density, direct burning of biomass is not economically feasible [7]. Moreover, improper storage of agricultural residues and wastes leads to environmental pollutants (soil, air, water, and visual) and odor problems [9]. Pelletization is the first step to decrease the moisture content and density of raw biomass. However, torrefaction applied after pelletization is a promising approach to minimize these disadvantages and provide direct use from conventional heating systems to large-scale energy generation power plants [10]. Torrefaction is the heat treatment process of pellet samples at 150–280 °C temperature ranges through the dehydration and decarboxylation reaction and a result of the conversion of raw biomass to commercial biofuels [11]. Many scientific studies indicated that raw biomass fuel properties, combustion, and emissions characteristics were improved with the torrefaction process [12,13].

Türkiye is the leader in the world's rose production, comprising 65% of the global rose oil production. In the last year, 19,879 tons of rose oil were manufactured in 3317 ha in 2022 in Isparta, Türkiye's center of rose production [14]. A total of 18,235 tons of RDW were annually produced due to the distillation process of rose flowers, corresponding to about twice the amount of rose flowers [15]. These wastes and residuals are dumped into the areas or stream beds located close to the distillation plants. This disposal contaminates the surface and underground waters of Isparta as well as causes nuisance odors and aesthetic problems [16]. Additionally, the red pine (*Pinus brutia* Ten.) is the most widespread tree species in Türkiye, covering an area of 56,10,215 hectares [17]. In 2020, about 850,000 m^3 of wood residues from forest products were produced in Türkiye [18]. At this point, improving the quality of biomass by producing biofuels provides not only the current national biomass potential but also reduces agricultural waste and is strategically important for our country, which is dependent on foreign energy sources.

Although there are various studies on the determination of combustion/co-combustion [19,20], pyrolysis [21–24], pelletization [25], and fire resistance [26] properties of *Pinus brutia* Ten., gasification potential [27], and pelletization properties of raw rose oil distillation solid wastes [9], no studies so far have been found in the literature on the pelletization of torrefied rose pulp. Therefore, this study aims to appraise two types of biomasses, namely rose oil (*Rosa damascena* Mill.) distillation solid waste (RDW) and red pine sawdust (RPS), which were pelleted with different moistures and additive ratios and then torrefied at 270 °C for 1 h. Various fuel properties of prepared raw and torrefied pellets, mass, diameter, length, proximate analyses, higher heating values (HHVs), tensile strength, and water uptake resistances were investigated within the scope of this study.

2. Materials and Methods

In this study, two types of biomass, RDW obtained from a rose oil factory and red pine sawdust (*Pinus brutia* Ten.) (RPS) from a sawmill in Isparta, were used for the production of pellets. The as-received moisture content of the RDW used in this study was 68 ± 5.14%. Therefore, due to the uncontrolled microbial degradation problem of RDW, the waste was dried by laying at room temperature and then dried in an oven (3 h at 70 °C), and its moisture content decreased to 6.25 ± 2.17% for further experiments. Since the moisture content (<10%) of RPS obtained from the sawmill was low, no extra drying step was carried out.

The proximate and elemental analysis results of the samples are given In Table 1. Standard test methods, ASTM D-871-82-2019, ASTM E-872, and E-1755-01, were used for the moisture content (MC), volatile matter (VM), and ash content (AC) of the samples, respectively. The fixed carbon (FC) amount was also calculated using the difference [19]. The correlations developed for raw biomasses (Equation (1)) [28] and torrefied biomasses [29]

(Equation (2)) provided in the literature were used to calculate the higher heating values (HHVs) of the raw and torrefied samples.

$$\text{HHV}_b = -10.81408 + 0.3133\,(\text{VM} + \text{FC}) \tag{1}$$

$$\text{HHV}_t = 0.1846\ \text{VM} + 3.3525\ \text{FC} \tag{2}$$

where HHV_b and HHV_t are the HHV (MJ/kg) of the raw and torrefied samples, respectively, and VM and FC refer to the volatile matter (%) and fixed carbon contents (%) of the samples.

Table 1. Elemental and proximate analysis results of the biomass samples.

	Moisture (%)	**Volatile Matter (%)**	**Fixed Carbon (%)**	**Ash (%)**	**HHV (kcal/kg)**
RPS *	8.20	85.15	5.45	1.20	4261.47
RDW **	6.25	77.45	9.56	6.75	4153.91
	C%	**H%**	**N%**	**S%**	**O% *****
RPS	48.18	6.40	-	-	44.22
RDW	45.58	6.02	4.14	0.29	37.22

* as-received, ** after being dried at 70 °C, *** ash-free.

After drying, the biomass samples were grounded with a ball mill, and then the samples were sieved between 2 mm and 250 µm for the pelletization process. Sieve analysis results of the samples are given in Table 2.

Table 2. Sieve analysis results of the biomass samples.

Ranges	**RDW (g)**	**RPS (g)**
2 mm	0.00	0.00
1.7 mm	36.35	103.91
1.18 mm	49.90	40.60
850 µm	31.36	28.34
600 µm	42.07	9.41
425 µm	18.23	3.03
250 µm	16.19	11.64
<250 µm	5.54	5.22

2.1. Pelletization of the Samples

In this study, pellets with initial moisture content close to the moisture contents of RDW, which were produced in the rose oil production facilities in Isparta province, were prepared to be able to be used in these facilities with their RDW in pellet production with a small capacity pelletizing machine without any drying processing. Accordingly, pellets were prepared by using a pelletizing machine (3 kW) with a flat die (11 mm × 25 mm) and a roller with a capacity of 15–20 kg/h. Corn starch was used as a binder during the experiments, and four different (0%, 2%, 4%, and 6%) binder contents and four different (50%, 55%, 60%, and 65%) initial moisture contents were used for the preparation of pellets. However, as the moisture content exceeded 60%; it caused the holes of the existing pellet machine to become clogged. Furthermore, RDW pellets with 60% moisture content could not be prepared without binder addition. Therefore, 65% moisture content was not used in the experiments. After the pellets were produced, they were dried at room temperature.

2.2. Torrefaction of the Pellets

The RDW and RPS pellets were also torrefied to understand the effect of thermal treatment on the physical and mechanical characteristics of the prepared pellets. Ceramic crucibles of 50 cm^3 with lids were used, and the pellets were torrefied in a muffle furnace at a torrefaction temperature of 250–300 °C, similar to previous studies with biomass in the literature [30–32].

In the preliminary experiments, three different holding times (15 min, 30 min, and 60 min) and three different torrefaction temperatures (250 °C, 270 °C, and 290 °C) were examined, and it was found that the optimum operating conditions that had the highest energy density were 270 °C for 1 h [33]. Therefore, in this study, the RDW and RPS pellets were torrefied at 270 °C for 1 h.

2.3. Calculation of Mass Yield, Energy Yield, and Energy Density Ratio of the Pellets

Based on Equations (3)–(5), the mass yield, energy yield, and energy density ratio of the torrefied samples were calculated. The dry ash-free mass of the biomass that is still present after torrefaction is expressed as a mass yield [33].

$$\text{Mass yield} = \frac{M_t}{M_b} \tag{3}$$

where M_b is the biomass before torrefaction and M_t is the amount of torrefied biomass at time t (g).

Equation (4) was used to determine the energy yields of the samples. Energy yield measures how much of the total chemical energy of the initial dry biomass was retained in the torrefied biomass [34]. As a result, it establishes how much energy was left over after sample burning.

$$\text{Energy yield} = \frac{M_t \times HHV_t}{M_b \times HHV_b} \tag{4}$$

Equation (5) was used to determine the energy density ratio of the samples, which is the ratio of the amount of energy released from the torrefied product when completely combusted to its original energy amount [34].

$$\text{Energy density ratio} = \frac{HHV_t}{HHV_b} \tag{5}$$

2.4. Determination of Tensile Strength, Impact, and Water Intake Resistance of the Pellets

In this study, the tensile strength of the pellets was also measured using Ertest, ADCON-1 Unconfined Pressure Test Set as a Newton (N). Five pellets were randomly selected from each mixture for the measurement of the tensile strength of the pellets. The vertical tensile strength of the pellets was calculated by using the following formula [9]:

$$\sigma_y = \frac{F}{\pi d^2} \tag{6}$$

where d and σ_y are the diameter of the pellets (m) and the vertical tensile strength (Pa), respectively. F is the maximum applied compressive force until the pellet is broken. Furthermore, to determine the impact resistance of the pellets, 10 randomly selected pellets from each mixture were first weighed and then dropped from a height of 1.85 m to the hard ground 4 times. After dropping 4 times, the pellets were sieved through a 3.15 mm diameter sieve and reweighed. By using the initial and final mass of the pellets, the mass loss (%) was calculated to determine the impact resistance of both raw and torrefied pellets [35].

$$\text{Mass loss } (\%) = \left(1 - \frac{m_A}{m_E}\right) \times 100 \tag{7}$$

where m_A is the pellet weight before test (g) and m_E is the pellet weight after test (g).

To determine the water intake resistance, firstly, the pellets were weighed and then immersed in water at room temperature for two hours. After two hours, the pellets were removed from the water and weighed every hour until they reached a constant weight [36]. Furthermore, IBM SPSS Statistics 20.0 was used to analyze the data set statistically. A bivariate analysis was applied to find the association between two variables in this study.

3. Results and Discussion

3.1. Proximate Analysis of the Raw and Torrefied RPS and RDW Pellets

In this study, the moisture contents of RPS pellets were 6.92%, 10.04%, and 9.56% for 50M + 0B, 55M + 0B, and 60M + 0B, respectively (Table 3). Torrefaction improves the physical properties, chemical composition, and energy and storage properties of biomass. The moisture content of the torrefied material is about 1–3% on a wet basis (w.b.) [37]. In this study, the moisture contents of the torrefied RPS pellets were 1.37%, 0.35%, and 0.94% for 50M + 0B, 55M + 0B, and 60M + 0B, respectively (Table 4). Similarly, the moisture contents of the RDW pellets were 8.51% and 8.61% for 50M + 0B and 55M + 0B, respectively (Table 3). Torrefaction also decreased the moisture content of the RDW pellets, and the final moisture contents of the torrefied RDW pellets were found to be 0.05% and 0.37% for 50M + 0B and 55M + 0B, respectively (Table 4). The torrefied pellets had considerably lower moisture content than the raw ones, even though there were no statistically significant differences ($p > 0.05$) between the initial moisture content and the final moisture content of the RPS and RDW pellets. In the ISO 17225-1:2021 standard, the moisture and ash contents of woody pellets prepared for industrial use are M10 $\leq$ 10% and A3.0 $\leq$ 3.0%, respectively [38]. Therefore, the moisture contents of the RPS and RDW pellets met the limit value given in the standard (Table 3). However, although the RPS pellets met the ash restriction, the ash content of the RDW pellets was approximately 3–4 times higher than the limit value given in the standard (Table 3). Similar findings were also observed for the torrefied pellets. On the basis of the ISO 17225-8:2016 torrefied pellet standard, the moisture and ash contents of the agricultural waste are M10 $\leq$ 10% and A5.0 $\leq$ 5.0%, respectively [39]. Although the ash content of the torrefied RDW pellets was higher than the limit value, the torrefied RPS pellets met the standard value for ash content.

The torrefaction process produces a uniform solid product with lower moisture and higher energy content than the raw biomass. During torrefaction, moisture and some volatile organic compounds in the biomass become volatile [37]. Similarly, in this study, AC, FC, and HHV values of the torrefied pellets generally increased compared to their raw form. However, in general, MC and VM decreased. In the literature, it is also stated that the VM of the biomass decreased with the torrefaction process while the FC increased [40,41]. The calorific value of torrefied biomass is higher because it has more C-C and C-H bonds capable of releasing more energy than the O-H and C-O bonds in the raw biomass. Therefore, the torrefied biomass moves toward the coal side in the Van Krevelen diagram [42]. Accordingly, in parallel with the increase in the FC as a result of torrefaction, the HHV of the RPS pellets increased from 17.51–18.80 MJ/kg to 20.20–21.73 MJ/kg, and the HHV of the RDW pellets increased from 17.42–18.96 MJ/kg to 19.13–20.92 MJ/kg.

3.2. Fuel Properties of the Raw and Torrefied RPS and RDW Pellets

According to both ISO 17225-1 (solid biofuels—fuel specifications and classes standard) and ISO 17225-8 (solid biofuels—fuel specifications and classes—part 8: graded thermally treated and densified biomass fuels for commercial and industrial use), the diameter and length of both raw and torrefied biomass pellets should be >6 $\pm$ 1 mm and 3.15 mm < L < 40 mm for industrial pellet boilers, respectively. All raw and torrefied RPS pellets appear to meet the standard, but RDW pellets did not (see Tables 5 and 6). When the lengths of the pellets are examined, it is seen that all the raw and torrefied pellets met the standard. In general, the particle density of both the RPS (825–1402 kg/m^3) and RDW (1038–2127 kg/m^3) pellets increased with increasing binder content. In other words, adding binders to the mixture generally improved the density of the pellets [43]. On the other hand, the initial moisture content of the pellets did not show a noticeable increase or decrease trend in particle density. However, with thermal treatment, the particle density of both the torrefied RPS (811–1404 kg/m^3) and RDW pellets (986–1731 kg/m^3) decreased. Because various tiny voids occur inside the pellets due to mass loss caused by the thermal decomposition of biomass, lower densities are detected for the torrefied pellets [44]. The calculated particle densities were found to be comparable to those reported for agricultural pellets (950–1400 kg/m^3) and wood pellets (1056–1500 kg/m^3) [9,45–49].

3.3. Abrasive Resistance and Tensile Strength of the Raw and Torrefied RPS and RDW Pellets

Tensile strength refers to the force required to break the pellets [50]. It is desirable that the pellets do not crumble and show higher strength values while being transported or stored. When the tensile strengths of the RPS and RDW pellets are examined, it was observed that the tensile strength of the RPS pellets increased with the increase in the binder content, but there is no significant increase in the tensile strength of the RDW pellets (Table 7). Binders are used to enhance the mechanical properties of the pellets. Furthermore, binders can also lower the energy needed for pelletization [51].

It was also determined that the resistance of the pellets decreased as the moisture content increased in both the raw and torrefied RPS and RDW pellets. Therefore, the optimum moisture content was identified as 50% and 55%. Although the strength of the RPS pellets increased as the binder content increased, the resistance of the RDW pellets did not increase at these two moisture contents. The tensile strength of the torrefied pellets also increased with increasing binder content. However, the optimum binder content was determined to be 2% for both pellet types in order to obtain pellets with higher calorific value by using more waste and meet the ISO 17225-1 standard. After torrefaction, pellet strength decreases due to a combination of wood polymer (i.e., hemicellulose and cellulose) degradation and weakening of the bonds among the particles [50]. Accordingly, the decrease in the tensile strength of the torrefied RPS pellets ranged from 8.40% (50M + 4B) to 73.21% (50M + 0B) and was found to be 35.94% on average. The decrease in tensile strength of the torrefied RDW pellets was between 9.92% (50M + 6B) and 57.48% (50M + 0B), with an average decrease of 35.09%. Therefore, the average decrease in the tensile strength of both types of biopellets was quite similar despite the differences in their cellulose, hemicellulose, and lignin contents. Although it has been reported in the literature [50] that there is a decrease in pellet strength (4.36% vs. 25.38%) with torrefaction, it is seen that this decrease is less than the decreases in the conducted study.

Average mass losses of the RPS pellets at 50M, 55M, and 60M regardless of binder ratios were 0.11%, 0.07%, and 0.12%, respectively. The average mass losses of the RDW pellets were found to be 0.03%, 0.03%, and 0.06% for 50M, 55M, and 60M, respectively, regardless of binder ratios (Table 8). In other words, the RPS pellets were found to be 2.4–6 times more prone to breakage and crumbling at the same moisture content than the RDW pellets. The average mass losses of the torrefied RPS and RDW pellets for 50M, 55M, and 60M were 0.25%, 0.34%, and 0.32% and 0.25% 0.22%, and 0.32%, respectively (Table 8). Therefore, there was not a big difference in breaking and crumbling between the torrefied RPS and RDW pellets in terms of mass loss. On the other hand, the torrefied RPS pellets were found to be 2–4.7 times more brittle compared to the RPS pellets, while the torrefied RDW pellets were found to be 6.4–12 times more brittle compared to the RDW pellets. After torrefaction, the fibers between the biomass particles are broken and the particles become shorter and spherical, and a more brittle structure is formed [52–54]. Furthermore, different initial moisture contents did not have a significant effect on the abrasive resistance of both the raw and torrefied pellets. Nevertheless, it is seen that the obtained results are compatible with the literature and the RPS, RDW, torrefied RPS, and torrefied RDW pellets maintain their integrity at an average of 99.89%, 99.96%, 99.72%, and 99.74% (impact resistance), respectively [55].

Table 3. Proximate analysis of the RPS and RDW pellets (mean ± standard deviation).

Mixes	MC (%)		VM (%)		FC (%)		AC (%)		HHV (MJ/kg)	
	RPS	RDW	RPS	RDW	RPS	RDW	RPS	RDW	RPS	RDW
50M + 0B	6.92 ± 0.07	8.51 ± 0.37	83.58 ± 4.23	67.05 ± 6.45	7.48 ± 1.03	17.49 ± 2.67	2.03 ± 0.15	6.96 ± 0.67	18.07 ± 0.45	18.54 ± 1.13
50M + 2B	10.56 ± 0.59	8.94 ± 0.58	77.50 ± 5.54	66.16 ± 3.32	10.75 ± 2.56	17.53 ± 1.98	1.18 ± 0.11	7.38 ± 0.56	18.10 ± 0.34	18.39 ± 0.20
50M + 4B	9.18 ± 0.67	9.14 ± 0.45	76.46 ± 3.24	68.74 ± 2.26	12.29 ± 1.95	15.76 ± 0.67	2.07 ± 0.09	6.36 ± 0.72	18.45 ± 1.06	18.24 ± 0.35
50M + 6B	11.62 ± 0.89	9.10 ± 0.23	76.38 ± 7.34	68.06 ± 5.56	9.82 ± 0.89	16.05 ± 0.34	2.19 ± 0.19	6.89 ± 0.86	17.56 ± 1.79	18.22 ± 0.19
55M + 0B	10.04 ± 1.01	8.61 ± 0.45	77.17 ± 2.23	65.32 ± 6.34	11.12 ± 1.19	18.36 ± 2.23	1.67 ± 0.09	7.71 ± 0.82	17.81 ± 0.06	18.53 ± 1.01
55M + 2B	10.13 ± 0.94	9.18 ± 0.38	79.01 ± 5.56	68.74 ± 0.98	10.10 ± 1.63	14.09 ± 0.79	0.75 ± 0.06	7.99 ± 1.09	18.15 ± 0.02	17.66 ± 0.05
55M + 4B	10.80 ± 0.56	8.10 ± 1.10	77.51 ± 6.34	69.39 ± 1.14	10.97 ± 1.39	15.20 ± 0.15	0.72 ± 0.02	7.40 ± 0.06	18.18 ± 0.45	18.17 ± 0.34
55M + 6B	10.53 ± 0.23	10.83 ± 0.97	77.59 ± 6.78	66.61 ± 0.56	11.29 ± 1.17	15.57 ± 1.01	0.71 ± 0.02	7.00 ± 0.67	17.75 ± 0.37	17.78 ± 0.13
60M + 0B	9.56 ± 0.45	-	75.28 ± 7.23	-	10.26 ± 1.04	-	4.90 ± 1.12	-	17.51 ± 0.57	-
60M + 2B	9.85 ± 0.56	7.38 ± 0.07	81.40 ± 4.14	65.84 ± 2.95	8.17 ± 0.67	19.31 ± 0.89	0.58 ± 0.01	7.47 ± 0.45	17.91 ± 0.23	18.96 ± 1.12
60M + 4B	9.28 ± 0.34	8.32 ± 0.23	77.98 ± 5.56	65.91 ± 3.34	11.19 ± 0.39	18.20 ± 2.24	1.55 ± 0.04	7.57 ± 0.96	18.34 ± 0.15	18.58 ± 0.45
60M + 6B	9.29 ± 0.23	12.63 ± 1.19	76.57 ± 4.32	65.04 ± 4.68	13.23 ± 0.49	15.35 ± 0.19	0.91 ± 0.02	6.98 ± 0.37	18.80 ± 1.09	17.42 ± 0.23

M: moisture content (%), B: binder content (%).

Table 4. Proximate analysis of the torrefied RPS and RDW pellets (mean ± standard deviation).

Mixes	MC (%)		VM (%)		FC (%)		AC (%)		HHV (MJ/kg)	
	RPS	RDW	RPS	RDW	RPS	RDW	RPS	RDW	RPS	RDW
50M + 0B	1.37 ± 0.31	0.05 ± 0.01	80.24 ± 2.26	65.86 ± 6.04	16.73 ± 1.41	24.55 ± 7.94	1.66 ± 0.26	9.54 ± 0.42	20.71 ± 0.32	20.81 ± 1.68
50M + 2B	0.86 ± 0.28	0.76 ± 0.23	77.09 ± 5.46	65.20 ± 8.2	20.98 ± 4.91	24.6 ± 7.90	1.08 ± 0.07	9.44 ± 1.21	21.63 ± 0.13	20.71 ± 1.25
50M + 4B	0.55 ± 0.52	0.03 ± 0.12	77.74 ± 10.2	65.18 ± 7.15	18.95 ± 5.78	25.2 ± 7.65	2.76 ± 0.99	9.59 ± 0.67	21.03 ± 0.15	20.92 ± 1.37
50M + 6B	1.02 ± 0.63	0.48 ± 0.43	75.81 ± 7.02	64.17 ± 7.15	17.60 ± 6.66	25.48 ± 8.21	1.57 ± 0.17	9.87 ± 0.18	20.20 ± 1.12	20.83 ± 1.57
55M + 0B	0.35 ± 0.08	0.37 ± 0.01	77.22 ± 8.30	65.06 ± 1.38	21.21 ± 5.12	22.42 ± 1.00	1.22 ± 0.36	12.14 ± 1.32	21.73 ± 1.21	19.91 ± 0.13
55M + 2B	1.24 ± 0.72	0.51 ± 0.18	75.65 ± 8.5	65.72 ± 0.21	21.89 ± 5.51	22.82 ± 1.36	1.22 ± 0.12	10.96 ± 0.67	21.68 ± 0.12	20.18 ± 0.52
55M + 4B	0.81 ± 0.19	0.25 ± 0.05	77.91 ± 0.60	66.20 ± 9.35	20.33 ± 2.18	22.57 ± 3.18	0.95 ± 0.11	10.97 ± 7.30	21.55 ± 0.34	20.18 ± 0.60
55M + 6B	1.02 ± 0.18	0.70 ± 0.07	76.41 ± 8.02	67.82 ± 5.28	20.70 ± 7.52	18.74 ± 2.86	1.88 ± 0.57	11.74 ± 2.48	21.40 ± 0.42	19.13 ± 0.38
60M + 0B	0.94 ± 0.26	-	75.94 ± 3.30	-	21.59 ± 3.81	-	1.13 ± 0.11	-	21.63 ± 0.26	-
60M + 2B	0.97 ± 0.03	0.55 ± 0.08	79.28 ± 4.92	65.3 ± 1.86	18.26 ± 2.10	23.08 ± 2.09	1.49 ± 0.21	10.57 ± 0.99	21.07 ± 0.24	20.19 ± 0.39
60M + 4B	0.95 ± 0.12	0.55 ± 0.01	75.60 ± 2.27	65.92 ± 0.57	20.99 ± 1.30	21.14 ± 1.36	2.06 ± 0.12	12.40 ± 0.87	21.35 ± 0.14	19.62 ± 0.42
60M + 6B	0.97 ± 0.05	0.06 ± 0.01	78.93 ± 3.30	65.17 ± 1.64	19.25 ± 3.93	22.61 ± 1.86	0.85 ± 0.11	12.16 ± 0.56	21.36 ± 0.15	20.00 ± 0.70

M: moisture content (%), B: binder content (%).

Table 5. Mass, diameter, length, and bulk density of the RPS and RDW pellets (mean ± standard deviation).

Mixes	Mass (g)		Diameter (mm)		Length (cm)		Particle Density (kg/m^3)	
	RPS	RDW	RPS	RDW	RPS	RDW	RPS	RDW
50M + 0B	0.518 ± 0.02	0.591 ± 0.04	6.6 ± 0.51	4.20 ± 0.42	2.06 ± 0.13	5.00 ± 0.00	938 ± 169	2127 ± 398
50M + 2B	0.663 ± 0.05	0.571 ± 0.03	6.7 ± 0.48	4.60 ± 0.51	2.40 ± 0.13	2.45 ± 0.09	996 ± 177	1812 ± 402
50M + 4B	0.631 ± 0.07	0.626 ± 0.04	6.00 ± 0.00	5.10 ± 0.31	2.62 ± 0.12	2.59 ± 0.13	1402 ± 73	1623 ± 247
50M + 6B	0.643 ± 0.02	0.625 ± 0.04	6.00 ± 0.00	5.05 ± 0.51	2.31 ± 0.12	2.60 ± 0.13	1234 ± 44	2045 ± 440
55M + 0B	0.620 ± 0.05	0.539 ± 0.04	7.00 ± 0.00	5.00 ± 0.00	2.4 ± 0.15	2.61 ± 0.19	825 ± 32	1316 ± 43
55M + 2B	0.736 ± 0.03	0.539 ± 0.01	6.60 ± 0.51	5.01 ± 0.42	2.64 ± 0.15	2.68 ± 0.07	1037 ± 168	1431 ± 328
55M + 4B	0.721 ± 0.06	0.519 ± 0.05	6.00 ± 0.00	5.00 ± 0.00	2.66 ± 0.14	2.71 ± 0.23	1198 ± 81	1221 ± 47
55M + 6B	0.671 ± 0.07	0.504 ± 0.05	6.00 ± 0.00	5.00 ± 0.00	2.5 ± 0.6	2.59 ± 0.22	1186 ± 57	1241 ± 62
60M + 0B	0.643 ± 0.03	-	6.40 ± 0.51	-	2.52 ± 0.13	-	1015 ± 186	-
60M + 2B	0.634 ± 0.04	0.446 ± 0.04	6.80 ± 0.42	5.00 ± 0.42	2.46 ± 0.15	2.67 ± 0.24	896 ± 114	1176 ± 209
60M + 4B	0.576 ± 0.07	0.420 ± 0.03	6.50 ± 0.52	5.00 ± 0.00	2.45 ± 0.25	2.58 ± 0.23	901 ± 151	1038 ± 30
60M + 6B	0.591 ± 0.05	0.509 ± 0.04	6.80 ± 0.42	5.00 ± 0.00	2.48 ± 0.20	2.84 ± 0.31	830 ± 122	1145 ± 47

Table 6. Mass, diameter, length, and bulk density of the torrefied RPS and RDW pellets (mean ± standard deviation).

Mixes	Mass (g)		Diameter (mm)		Length (cm)		Particle Density (kg/m^3)	
	RPS	RDW	RPS	RDW	RPS	RDW	RPS	RDW
50M + 0B	0.398 ± 0.02	0.420 ± 0.02	6.00 ± 0.00	4.20 ± 0.42	2.15 ± 0.10	2.50 ± 0.14	822 ± 53	1555 ± 277
50M + 2B	0.541 ± 0.04	0.412 ± 0.02	5.70 ± 0.48	4.00 ± 0.00	2.42 ± 0.18	2.39 ± 0.15	1116 ± 188	1719 ± 779
50M + 4B	0.637 ± 0.02	0.479 ± 0.03	5.00 ± 0.00	4.40 ± 0.51	2.70 ± 0.04	2.67 ± 0.18	1404 ± 45	1532 ± 346
50M + 6B	0.455 ± 0.02	0.446 ± 0.02	5.00 ± 0.00	4.00 ± 0.00	2.35 ± 0.15	2.57 ± 0.09	1236 ± 54	1731 ± 95
55M + 0B	0.456 ± 0.06	0.389 ± 0.03	5.30 ± 0.48	4.10 ± 0.31	2.5 ± 0.30	2.41 ± 0.17	1054 ± 167	1551 ± 206
55M + 2B	0.433 ± 0.02	0.365 ± 0.02	5.90 ± 0.31	4.00 ± 0.00	2.35 ± 0.15	2.43 ± 0.18	852 ± 123	1496 ± 45
55M + 4B	0.479 ± 0.02	0.359 ± 0.02	5.70 ± 0.48	4.50 ± 0.42	2.35 ± 0.16	2.47 ± 0.15	1021 ± 185	1345 ± 221
55M + 6B	0.554 ± 0.05	0.414 ± 0.02	6.00 ± 0.00	4.20 ± 0.42	2.58 ± 0.12	2.5 ± 0.14	951 ± 87	1532 ± 252
60M + 0B	0.492 ± 0.03	-	5.90 ± 0.31	-	2.44 ± 0.18	-	936 ± 173	-
60M + 2B	0.463 ± 0.03	0.351 ± 0.03	5.90 ± 0.31	4.10 ± 0.31	2.53 ± 0.11	2.45 ± 0.21	845 ± 117	1374 ± 154
60M + 4B	0.517 ± 0.02	0.305 ± 0.01	6.00 ± 0.47	4.60 ± 0.51	2.45 ± 0.13	2.42 ± 0.12	950 ± 158	986 ± 236
60M + 6B	0.446 ± 0.03	0.308 ± 0.01	5.90 ± 0.31	4.30 ± 0.48	2.55 ± 0.17	2.44 ± 0.14	811 ± 122	1121 ± 209

Table 7. Tensile strength of the raw and torrefied pellets (MPa) (mean ± standard deviation).

Mixes	RPS	RDW	Torrefied RPS	Torrefied RDW
50M + 0B	1.12 ± 0.05	1.27 ± 0.05	0.30 ± 0.02	0.54 ± 0.05
50M + 2B	1.16 ± 0.05	1.23 ± 0.03	0.83 ± 0.05	0.75 ± 0.03
50M + 4B	1.19 ± 0.02	1.29 ± 0.01	1.09 ± 0.04	0.83 ± 0.01
50M + 6B	1.53 ± 0.04	1.31 ± 0.02	1.21 ± 0.05	1.18 ± 0.02
55M + 0B	1.00 ± 0.01	1.01 ± 0.01	0.46 ± 0.01	0.56 ± 0.01
55M + 2B	1.04 ± 0.01	0.98 ± 0.01	0.89 ± 0.04	0.59 ± 0.01
55M + 4B	1.88 ± 0.04	0.99 ± 0.04	1.21 ± 0.03	0.53 ± 0.04
55M + 6B	2.63 ± 0.05	1.29 ± 0.04	1.90 ± 0.01	0.75 ± 0.04
60M + 0B	0.76 ± 0.01	-	0.45 ± 0.05	-
60M + 2B	0.93 ± 0.03	0.54 ± 0.01	0.46 ± 0.04	0.46 ± 0.01
60M + 4B	1.03 ± 0.01	0.58 ± 0.01	0.62 ± 0.01	0.40 ± 0.01
60M + 6B	1.07 ± 0.01	0.59 ± 0.01	0.67 ± 0.01	0.44 ± 0.01

Table 8. Mass loss (%) of the raw and torrefied pellets (mean ± standard deviation).

Mixes	RPS	RDW	Torrefied RPS	Torrefied RDW
50M + 0B	0.08 ± 0.03	0.01 ± 0.02	0.25 ± 0.20	0.29 ± 0.12
50M + 2B	0.13 ± 0.08	0.03 ± 0.03	0.26 ± 0.16	0.27 ± 0.11
50M + 4B	0.13 ± 0.17	0.04 ± 0.01	0.24 ± 0.01	0.25 ± 0.11
50M + 6B	0.08 ± 0.07	0.02 ± 0.03	0.26 ± 0.02	0.17 ± 0.13
55M + 0B	0.08 ± 0.07	0.01 ± 0.08	0.38 ± 0.33	0.28 ± 0.01
55M + 2B	0.11 ± 0.18	0.04 ± 0.03	0.38 ± 0.01	0.19 ± 0.09
55M + 4B	0.04 ± 0.03	0.02 ± 0.02	0.36 ± 0.35	0.21 ± 0.06
55M + 6B	0.04 ± 0.05	0.03 ± 0.03	0.22 ± 0.19	0.20 ± 0.06
60M + 0B	0.12 ± 0.03	-	0.24 ± 0.02	-
60M + 2B	0.10 ± 0.08	0.06 ± 0.04	0.20 ± 0.04	0.32 ± 0.14
60M + 4B	0.11 ± 2.80	0.07 ± 0.48	0.28 ± 0.01	0.37 ± 0.15
60M + 6B	0.14 ± 0.03	0.04 ± 0.02	0.23 ± 0.07	0.27 ± 0.49

3.4. Mass Yield, Energy Yield, and Energy Density Ratios of the Torrefied RPS and RDW Pellets

In the torrefaction process, the hemicellulose is mainly responsible for the mass loss of biomass [56]. Hemicellulose in biomass depolymerizes during the torrefaction process and releases volatile compounds with lower energy values. Therefore, the solid matter obtained after torrefaction always has high energy content [57]. An energy density above 1 indicates an energy gain per unit mass. Therefore, pellets with an energy density above 1 mean that the torrefaction process increases the net usable energy of the pellet [58]. In this study, similar to the literature, the average energy density ratios of the RPS pellets for 50M, 55M, and 60M, regardless of binders, were found to be 1.13, 1.13, and 1.15, respectively. Similarly, the energy density ratios of the RDW pellets were found to be 1.02, 0.93, and 1 for 50M, 55M, and 60M, respectively (Table 9). Therefore, for the same moisture content and different starch additives, a statistically significant difference could not be observed ($p > 0.05$) for both the RPS and RDW pellets.

Biomass maintains about 90% of its energy density with the torrefaction process [59]. However, the energy yield of biomass depends on its content and the energy yield of the remaining solid decreases with increasing temperature and residence time. Increasing carbon content and decreasing hydrogen and oxygen content cause HHVs to increase because the energy contained in the C–C bond is higher than in the C–H or C–O bonds [60]. The average energy yield of the torrefied RPS pellets for 50M, 55M, and 60M were 0.82, 0.81, and 0.81, respectively. On the other hand, the average energy yield of the torrefied RDW pellets for 50M, 55M, and 60M was found to be 0.75, 0.64, and 0.72, respectively. The energy yield of the torrefied both RPS and RDW pellets increased significantly ($p < 0.05$) with the increasing binder content in the mixture. In the literature, the energy yield of Adansonia digitata varied between 70.74% and 74.96% for different torrefaction temperatures [61]. Phanphanich and Mani (2011) showed that the mass yield, energy yield, and energy density of woody biomass (pine chips and log residues, torrefaction temperature 225/250/275/300 °C, 0.5 h) were 52–89%, 71–94%, and 1.05–1.39%, respectively [62]. Pimchuai et al. (2010) reported that the mass yield, energy yield, and energy density of agricultural residues (rice husk, peanut husk, pulp, and water hyacinth, 250/270/300 °C torrefaction temperature, 1/1.5/2 h residence time) were 41–79%, 55–98%, and 1.08–1.66.

Table 9. Mass yield, energy yield, and energy density ratio of the torrefied pellets (mean ± standard deviation).

Mixes	Mass Yield		Energy Yield		Energy Density Ratio	
	RPS	**RDW**	**RPS**	**RDW**	**RPS**	**RDW**
50M + 0B	0.75 ± 0.01	0.75 ± 0.03	0.81 ± 0.04	0.75 ± 0.10	1.08 ± 0.04	1.00 ± 0.09
50M + 2B	0.79 ± 0.01	0.72 ± 0.02	0.89 ± 0.08	0.76 ± 0.08	1.13 ± 0.09	1.01 ± 0.08
50M + 4B	0.71 ± 0.01	0.71 ± 0.02	0.81 ± 0.04	0.73 ± 0.04	1.14 ± 0.06	1.03 ± 0.08
50M + 6B	0.67 ± 0.00	0.73 ± 0.03	0.78 ± 0.07	0.75 ± 0.09	1.16 ± 0.11	1.02 ± 0.09
55M + 0B	0.78 ± 0.01	0.68 ± 0.05	0.85 ± 0.04	0.61 ± 0.04	1.09 ± 0.06	0.89 ± 0.01
55M + 2B	0.69 ± 0.01	0.68 ± 0.01	0.79 ± 0.06	0.64 ± 0.01	1.14 ± 0.01	0.94 ± 0.03
55M + 4B	0.71 ± 0.03	0.68 ± 0.02	0.80 ± 0.03	0.64 ± 0.04	1.14 ± 0.04	0.94 ± 0.03
55M + 6B	0.71 ± 0.03	0.67 ± 0.02	0.81 ± 0.10	0.65 ± 0.00	1.13 ± 0.00	0.96 ± 0.02
60M + 0B	0.71 ± 0.07	-	0.83 ± 0.10	-	1.15 ± 0.04	-
60M + 2B	0.77 ± 0.02	0.70 ± 0.05	0.87 ± 0.03	0.69 ± 0.03	1.12 ± 0.07	0.98 ± 0.02
60M + 4B	0.66 ± 0.01	0.73 ± 0.01	0.75 ± 0.01	0.69 ± 0.01	1.14 ± 0.03	1.00 ± 0.00
60M + 6B	0.70 ± 0.03	0.71 ± 0.01	0.81 ± 0.11	0.72 ± 0.05	1.15 ± 0.11	1.01 ± 0.05

Mass yield is dependent on raw biomass type, torrefaction temperature, residence time and reactor type, etc., regardless of energy yield or energy density [63]. On the other hand, agricultural residues appear to have a higher mass loss and energy density due to their relatively higher volatile and hemicellulose content [64]. In this study, the mass yield did not change significantly with the moisture content of the pellets, and the average mass yields of the torrefied RPS pellets were 0.73, 0.72, and 0.70 for 50M, 55M, and 60M, respectively. The average mass yields of the torrefied RDW pellets for 50M, 55M, and 60M were 0.73, 0.68, and 0.71, respectively. While there was a significant ($p < 0.05$; $r^2 = 0.665$) relationship between average mass yield and average energy yield for the RPS pellets, no significant relationship was found between average mass yield and average energy density ($p > 0.05$; $r^2 = 0.025$). On the other hand, in addition to a significant relationship between average mass yield and energy yield ($p < 0.05$; $r^2 = 0.797$), a significant relation between average mass yield and energy density ($p < 0.05$; $r^2 = 0.548$) of the RDW pellets was found in this study.

3.5. Water Intake Resistance of the Raw and Torrefied RPS and RDW Pellets

One of the enormous problems during the storage of pellets is the re-absorption of moisture and the following microbiological degradation [65]. Therefore, as emphasized in the literature, the torrefaction process can convert the hydrophilic structure of the biomass to a hydrophobic structure via the elimination of hydroxyl groups in the biomass [42]. It took longer for the raw pellets to obtain a constant weight compared to the torrefied ones in this study (Figures 1–4). Furthermore, the time to reach a constant weight of the RPS, RDW, torrefied RPS, and torrefied RDW pellets was about 21, 20, 10, and 9 h, respectively. Therefore, the torrefied pellets were found to be more stable regarding water intake than the raw pellets.

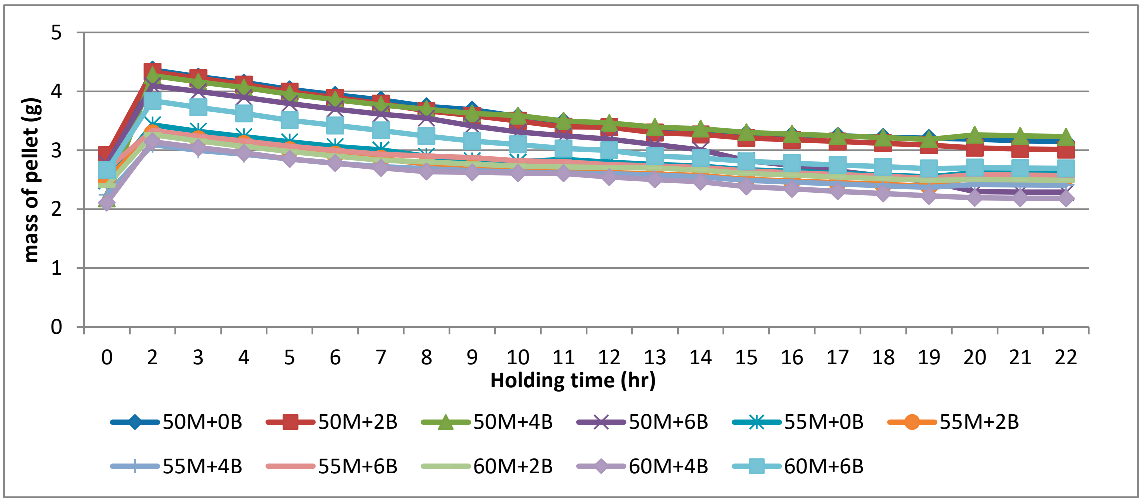

Figure 1. The water intake resistance of the RPS pellets as a function of holding time.

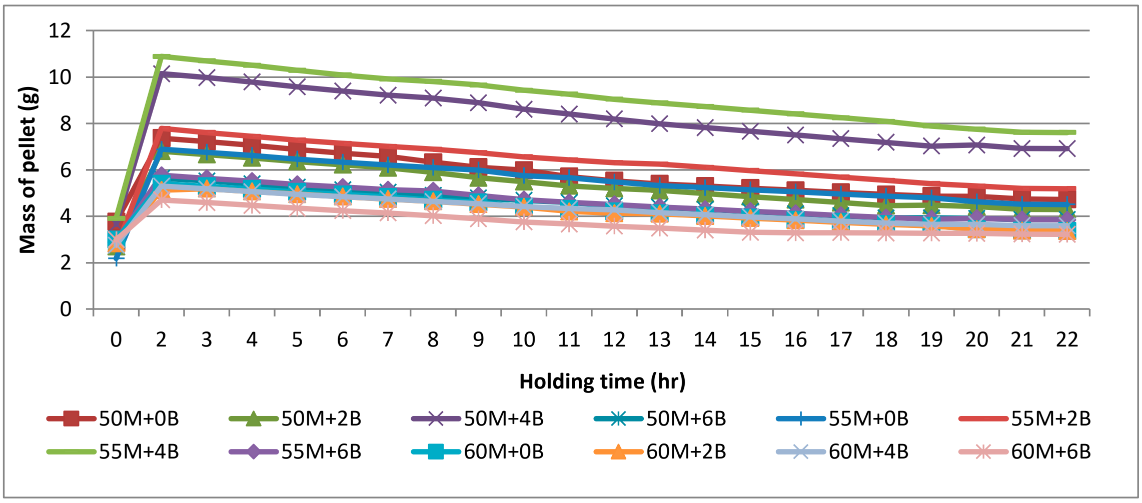

Figure 2. The water intake resistance of the RDW pellets as a function of holding time.

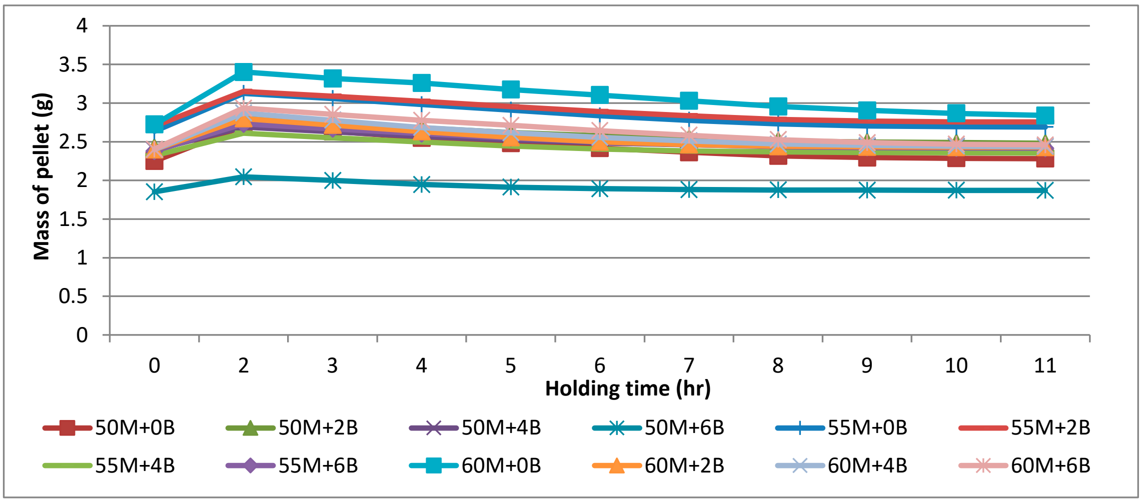

Figure 3. The water intake resistance of the torrefied RPS pellets as a function of holding time.

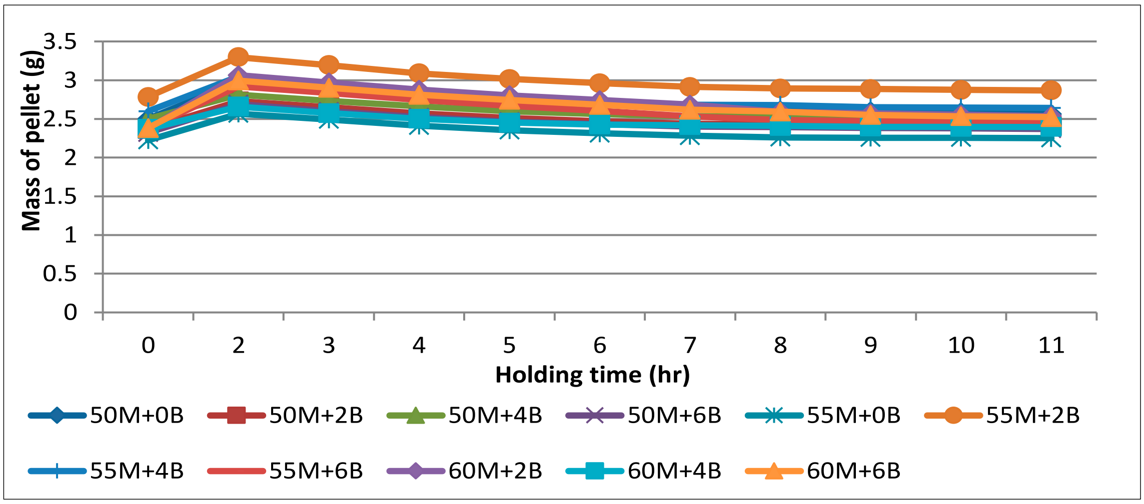

Figure 4. The water intake resistance of the torrefied RDW pellets as a function of holding time.

At the end of 2 h, the mass of the RDW pellets increased by 19.53% (50M + 0B) to 44.36% (60M + 6B). The masses of the RPS pellets increased from 57.19% (60M + 6B) to 198.36% (55M + 4B) after 2 h. On the other hand, water intake of the torrefied RDW pellets after 2 h was between 13.72% (50M + 0B) and 21.86% (60M + 6B) for different mixtures, while the moisture absorption amount of the torrefied RPS was between 19.32% (45M + 0B) and 33.41% (50M + 10B). Therefore, it has been observed that both the raw and torrefied RDW pellets had higher water intake resistance compared to the RPS pellets, and the torrefaction process increased the water intake resistance of the biopellets. As a result, it was clearly seen in our study that the torrefied RPS and RDW pellets resisted water uptake and further degradation. In addition, this study observed that the raw pellets broke down very quickly, and a brown color was observed in the water. In contrast, the torrefied pellets remained harder and retained their original shape compared to the raw pellets, and the color of the water was much lighter than the raw pellets.

Similar results were also obtained in the literature, and water intake of the torrefied pellets decreased between 7% and 20% [66,67]. In a study, pellets torrefied at 200 °C had a water intake of less than 40% of their initial mass in the first 10 min. However, a further increase in torrefaction temperature decreased the water intake. Furthermore, when raw pellets were exposed to water, their weight significantly increased in the first 10 min and reached 165% of their initial mass [68].

4. Conclusions

In this study, the quality of the raw and torrefied pellets blending with RDW and RPS having different moisture and binder content was investigated. Based on our findings, the main conclusions can be listed as follows.

- ✓ The contrary of findings in the literature, the ash content, fixed carbon, and higher heating values of the torrefied pellets were generally higher than their raw form. However, their moisture and volatile matter content were lower. In line with the increase in the fixed carbon content as a result of torrefaction, the higher heating values of the RPS and RDW pellets increased.
- ✓ Although the initial moisture content of the pellets did not show a significant variation in the particle density, the particle density of both the RPS and RDW pellets decreased after the torrefaction process.
- ✓ The tensile strength of the raw RPS increased with increases in the binder content, but there was no significant increase in the tensile strength of the RDW pellets with the binder content. Furthermore, the average decreases in the tensile strength of both the torrefied RPS and RDW were found to be close.

- ✓ The average energy density ratios of the RPS and RDW pellets were above one, while the torrefied RPS and RDW pellets were just below one, but their values improved significantly with the increasing binder content of the mixture.
- ✓ The relationship between average mass yield and average energy yield regardless of binder ratios was significant for RPS but not RDW. For the torrefied RPS and RDW pellets, there was no statistically significant difference between moisture content and energy efficiency, energy density, or mass yield.
- ✓ As a result of torrefaction, the increase in the higher heating values of the RPS pellets was higher than the RDW value.
- ✓ The RPS pellets were found to be more prone to breakage and chipping at the same moisture content than the RDW pellets at rates ranging from 2.4 to 6 times. Furthermore, the torrefaction process increased the breakage of the pellets, causing them to crumble.
- ✓ Both the raw and torrefied RDW pellets had higher water intake resistance than the RPS pellets. The torrefaction process increased the water intake resistance of the biopellets, resulting in the torrefied RPS and RDW pellets resisting water uptake and further degrading. Accordingly, torrefied pellets were more stable in moisture absorption than the raw pellets, and they can be stored in the open air longer than the raw pellets.

Therefore, when using the RDW and RPS pellets as alternatives to raw materials, torrefaction is a good alternative because it has high calorific values, water intake resistance, and a longer open storage time. It was also concluded that the quality of both the RDW and RPS pellets could be optimized for thermochemical systems as a fuel depending on their moisture and binder contents.

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