Article

Study of Mass Loss and Elemental Analysis of Pine Wood Pellets in a Small-Scale Reactor

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Abstract: Studying the thermal decomposition of wood pellets is an important subject in order to understand the behavior of wood pellets during the combustion process. In fact, wood pellets have become an important fuel used in boiler combustion. The objective of this study is to investigate the mass loss and elemental analysis of pine wood pellets at various times and temperatures. Commercial pellets with a diameter of 6 mm were used. The experiment was conducted in the laboratory of the Engineering University of Minho. The pellets were burned in a small reactor of 1.36 kW with a maximum temperature range of 1150 °C. The data were observed at different temperatures: 264, 351, 444, 541, 650, and 734 °C, and at time intervals of 30, 60, 120, 180, 240, 300, 600, 900, 1200, and 3600 s. The results of the experiment revealed that the reaction rate increases with the temperature, and the higher the combustion temperature applied, the higher the mass loss of all substances observed. The remaining mass, as fixed carbon and ash or unburned substances, is about 3%. The residence time and temperature influence the species concentration of wood pellets.

Keywords: mass losses; temperatures; wood pellets; elemental analysis; residence time

1. Introduction

Biomass is one of the important renewable resources to be developed for energy utilization. The demand for biomass fuel is increasing due to its carbon-neutral and renewable properties [1,2] and the importance of the increased demand in the energy market [3]. Biomass is mainly composed of wood, short-rotation woody crops, agricultural residues, herbaceous species, municipal solid wastes, food processing wastes, aquatic plants, and animal wastes [4]. Woody biomass is densified into pellets from sawdust and waste wood after the milling, drying, and compression processes [1,5]. The purpose is to improve the physical and mechanical properties of wood biomass by producing a homogeneous quality regarding the particle density, moisture content, and uniform shape and size. Wood pellets have some advantages, such as being suitable for transport and storage, and their high energy density, durability, and use in a heating system [1]. As an interesting biomass fuel, the demand for wood pellets is predicted to increase continuously and become mostly supplied both domestically and internationally, which will also be increased in the coming years, such as in the EU countries [6].

The combustion of wood pellets is applied in a boiler or stove. Rabaçal et al. [7] investigated the combustion and emission characteristics of biomass as a function of thermal inputs using pine pellets burned in a domestic boiler. This study described the utilization of the typical operating conditions of a boiler to produce emissions with a specific biomass

material. Meanwhile, Bäfver et al. [8] applied pellet stoves and modern and old-type residential wood stoves to investigate the particle emissions produced from the combustion of wood logs and pellets. This study revealed that in terms of CO concentration, wood stoves emitted more CO than pellet stoves.

The composition of wood material is determined by the combustion characteristics of biomass. The nitrogen produced from the combustion of biomass is either from nitrogen in the air or in the fuel [9]. With proximate and ultimate analysis, the elemental of the biomass is identified [10]. The biomass of pine bark and wood was investigated by Saarela et al. [11], who compared their elemental contents in ashes. This study shows that the contents of inorganic elements and ash are mainly higher for bark than for wood. The determination of the calorific value, bulk density, durability, hardness, and equilibrium moisture content of torrefed pine was experimentally conducted by Järvinen and Agar [12]. This study also calculated the elemental analysis, including of ash, volatiles, fixed Carbon, Carbon, Hydrogen, Nitrogen, Oxygen, and Sulfur. Pine and beech wood were torrefied under the same conditions at different temperatures from 200 °C to 300 °C in a lab-scale device [13]. This study measured several parameters, including mass loss, temperature profile, and gas composition, and determined the elemental analysis of Carbon, Hydrogen, and Oxygen with different temperature values. It shows that thermal treatment leads to an increase in the Carbon content and a decrease in both the Hydrogen and Oxygen content. The elemental composition of several samples is presented in Table 1.

Thermal deformations of wood pellets during pyrolysis were conducted in an electrically heated horizontal tubular furnace with a temperature of 300 °C to 1000 °C in an inert atmosphere [14]. In this study, the center temperature of the pellet was also measured, and the results showed a reduction in the pellet's diameter when the heating temperature was increased. The single pellet of softwood and wheat straw was burned in the furnace at temperatures of 700 °C and 1000 °C [15]. This study reveals that single pellet combustion shows a similar release character of refractory and volatile elements as in grate boilers. The influences of the radiation temperature, moisture content, particle size, and biomass's physical properties on the heating rate during pyrolysis were investigated by Mehrabian et al. [16]. The Biot number and the thermal time constant were applied. This study shows that the effect of the Biot number on the heating rate is negligible compared to the thermal time constant. Thus, the thermal time constant can be applied to specify the heating rate regimes during pyrolysis. In addition, Miccio et al. [17] stated that, as an endothermic step, the devolatilization needs heat from the external environment and it can be either thermally or kinetically controlled, depending on particle size and shape.

Table 1. Proximate and ultimate analysis of several samples.

Sample	Proximate Analysis, (%)				Ultimate Analysis, (%)					CO_2 (kg/MJ)	LHV (MJ/kg)	References
	Moisture	VM	Ash	Fixed Carbon	C	H	N	S	O			
Pine pellets	6.90	77.80	0.60	14.70	50.80	5.39	1.55	0.037	42.22	-	17.10	Recent study
Whole-tree pine chips	7.89	83.2	0.5	15.8	49.7	6.2	0.17	-	43.6	-	17.13	[12]
Pine pellets	6.65	76.62	2.50	14.23	46.8	5.99	0.675	0.021	-	0.0979	17.53	[18]
Pine branches	12.5	63.7	2.6	21.2	46.6	6.3	0.9	<0.02	31.1	-	17	[19]
Pine pellets	7.3	80.5	1.3	10.9	46.0	6.2	0.5	<0.01	47.3	-	17.1	[20]

The combustion of wood pellets produces minor gas emissions into the environment [6], even though the gas emissions produced from the combustion need to be evaluated to understand their behavior and properties. This study investigates the mass loss and elemental analysis of wood pellets in a small reactor. The dry and wet pellets at different temperatures and times are also evaluated.

2. Materials and Methods

2.1. Properties of Pine Wood Pellets

Commercial pine wood pellets with characteristics according to the EN plus A1 Class standard were used [21]. The properties of the pine wood pellets applied in this study are presented in Table 2.

Table 2. Properties of pine wood pellets.

Proximate Analysis (wt.%, as Received)		Ultimate Analysis (wt.%, Dry Ash-Free)	
Moisture	6.90	Carbon	50.80
Volatile matter	77.80	Hydrogen	5.39
Ash	0.60	Nitrogen	1.55
Fixed carbon	14.70	Sulphur	0.037
Lower Heating Value (MJ/kg)	17.10	Oxygen	42.22

2.2. Experimental Apparatus and Procedure

The set of experiments was carried out in a small-scale reactor with a thermal capacity of 1.36 kW and a temperature range up to 1150 °C. This reactor is 100 mm in diameter and 170 mm in height. It contains a cup (made of stainless steel) with a diameter of 30 mm and 33 mm in height, and a scale with 100 mg accuracy. In addition, a dry pellet experiment was also conducted for the experimental comparison with wet pellets.

The pellet samples were heated in a constant temperature environment, and at a specific time, the sample for the mass loss and the elemental composition (based on the proximate and ultimate analysis) were measured. The data were obtained at different ambient temperatures: 264, 351, 444, 541, 650 and 734 °C and at time intervals of 30, 60, 120, 180, 240, 300, 600, 900, 1200, and 3600 s. A preliminary set of experiments was carried out in an alternative test facility, whose temperature was limited to a maximum of 250 °C. Although the scope of such experiments is limited, they provide the basis for fine tuning the procedure. In conducting the dry pellet tests, the pellets were dried at 105 °C for more than 24 h to remove the moisture content in a reactor with a temperature range of up to 250 °C. The samples were removed from water moisture by drying them according to Swedish standard SS 18 71 20 [22]. For both dry and wet pellet experiments, the procedure is schematically described in Figure 1.
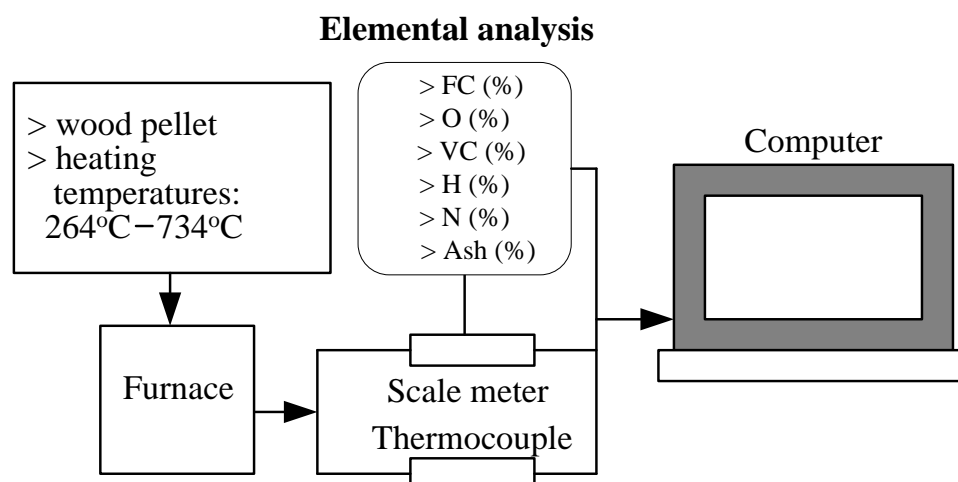

Figure 1. Experimental diagram.

In order to obtain the mass loss, the experiment was carried out in the following manner. The reactor and sample holder were preheated to the desired temperature. The

sample holder was removed from the reactor, weighed, and placed back in the reactor. In the meantime, the pellet was also weighted and placed inside the reactor, and the clock was set. Once the desired time was achieved, the sample/sample holder combination was removed from the reactor and weighed, and the mass loss was calculated from the weight difference. After the mass losses of pellets were obtained, the remaining masses of pellets were subjected to elemental analysis. The elemental analysis was conducted on the solid sample in order to understand the devolatilization of the pellets. In addition, the elemental analysis was conducted by using a CHN/CHNS Carbon/Hydrogen/Nitrogen/Sulfur/Oxygen determinator.

3. Results and Discussion

3.1. Mass Loss of Wood Pellets

The mass loss of wood pellets was calculated as the difference between the initial mass and final mass (Equation (1)).

$$m_l = m_i - m_f \qquad (1)$$

where m_l is a mass loss, m_i is the initial sample mass, and m_f is the final mass of the sample.

The mass loss as a function of time and temperature is presented in Figure 2. The temperature reported is the actual temperature inside the reactor, because it was observed that there was a gap between the setup and actual temperatures. The horizontal dotted line at 84.7% represents the percentage of volatile matter for the pine wood pellets on a wet basis (see Table 1). The data plotted show that the mass loss increases for any specific temperature; the higher the temperature, the faster the pellets volatilize. The results also show that most of the devolatilization occurs in the first 5 min of the process, depending on the temperature. For very low temperatures (264 °C), the mass loss occurs at a very slow rate, as the experiment was extended for over one hour. In any case, this temperature is above the one that defines the initial decomposition of biomass (204–261 °C) (including hemicellulose and lignin). This observation is in agreement with the TGA tests, which have shown that the reaction kinetics are very slow at such low temperatures. Because of the slow devolatilization process, at the lowest temperatures (264–351 °C), the data show a kink in the rate of mass loss, where a leveling of the mass precedes the devolatilization process. Because this stabilization is in the range of 7–8%, it suggests that the drying process is independent of the fuel devolatilization and that these are two distinct phases. At higher temperatures (which would induce a higher heating rate), such separation is not evident. The TGA data also corroborate this observation.

For the highest temperatures (650–734 °C), it is observed that most of the volatile matter is released within the first 2 min as the mass loss approaches the percentage of volatile matter, which is 84.7%. After approximately 1 h, the mass loss at 650 and 734 °C converges to the same value (~97%), which means that all the volatile gases have been released and the remaining substances are ashes, while the remaining unburned fixed Carbon is about 3%.

In order to investigate the influence of the initial moisture of the pellets and the transition between the drying and devolatilization phases, a set of tests was carried out with dry pellets. The experiments were limited to the initial 240 s of the process, and the results are presented in Figure 3. From the data, one may observe that the initial mass loss is due to the moisture released as the rate of mass is higher for wet pellets than for dry pellets. Water diffusion is more effective, which is more evident at low temperatures. In fact, for the lowest temperature (264 °C), the dry pellets do not lose any mass at all in the initial 240 s. Because this temperature is above the initial decomposition temperature, the fuel volatiles will eventually be released, as one can conclude from the data represented in Figure 2. As the pellet is brought to a higher temperature, the fuel volatiles are released, and the presence of water inside the pellet structure delays the fuel devolatilization due to the phase change enthalpy. With the temperature increase. the fuel devolatilization (mass loss observed in dry pellets) increases at a higher rate and earlier in the process. For the highest testing temperatures, the temperature rise is so fast (high heating rate) that dry

pellets appear to release the volatile matter faster than wet pellets even in the initial stages of reaction. Because of the sampling procedure, at 30 s (1st data point), we are not able to capture the initial moisture release from the biomass. This observation is in agreement with the results obtained with the TGA. Eventually, in the long run, all the samples (wet and dry) converge to the same rate of mass loss as all the moisture should have been released by this time. The mass loss at 734 °C and time 240 s is higher for wet pellets than for dry pellets, as at these conditions, the dry pellets almost devolatized completely and started with char combustion compared to the wet pellets. The temperature range between 400–700 °C includes the primary and secondary pyrolysis where volatile gases are released including water, tar, permanent gas, and char, while the range of 700–800 °C is referred to as the gasification phase [23].
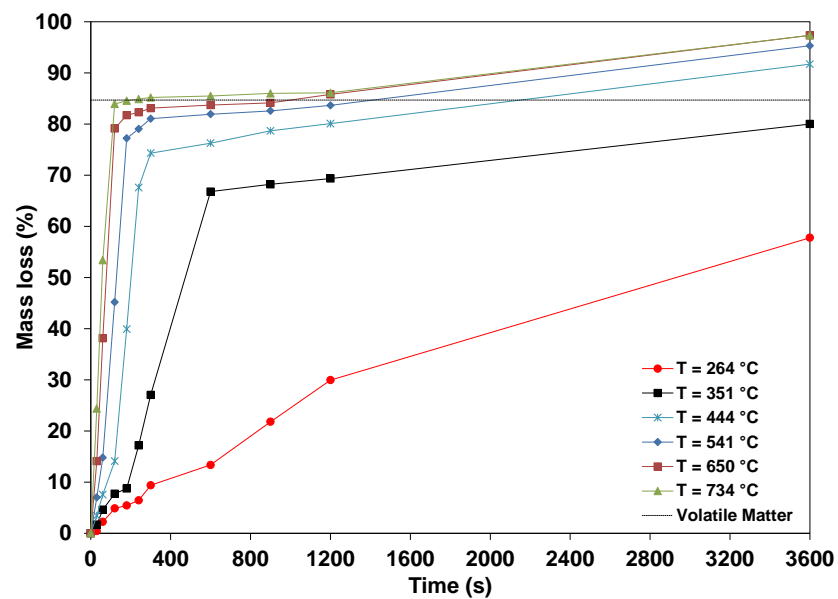

Figure 2. Mass loss of wood pellets vs time at temperature 264–734 °C.
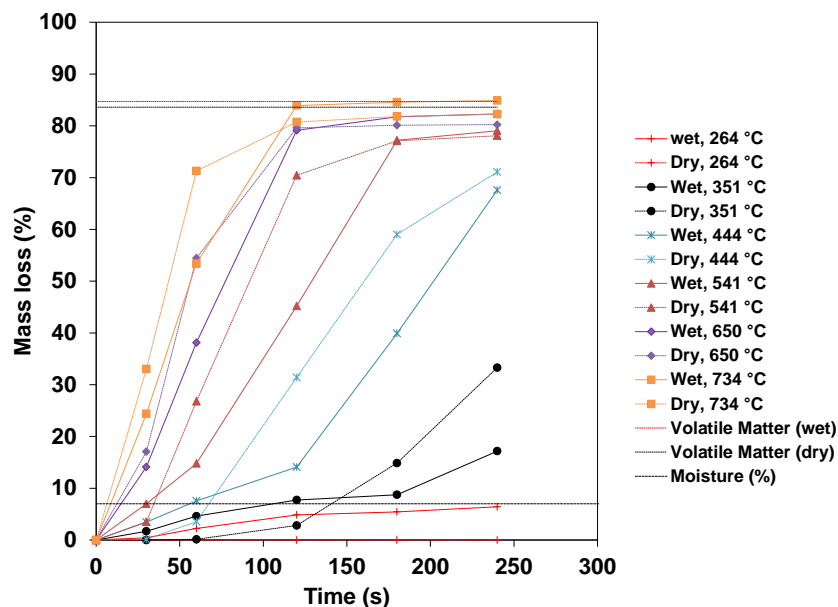

Figure 3. Mass loss of dry and wet pellets.

3.2. Elemental Analysis of Wood Pellets

The structure of biomass is composed of Cellulose (cell walls of biomass material), which represents 40 to 45% of the dry weight of wood; Hemicellulose represents 20 to 35% of the dry weight of wood, and Lignin represents 15 to 30% of the dry weight of wood [24]. The elemental analysis was conducted over time at different temperatures. By applying the same test to the mass loss experiment, the results of the elemental analysis can be obtained. Figure 4 presents the preliminary test to observe the behavior of the elemental composition during the combustion of wood pellets in a small-scale reactor with a maximum temperature range of 1150 °C, to determine the time length required for the other samples.
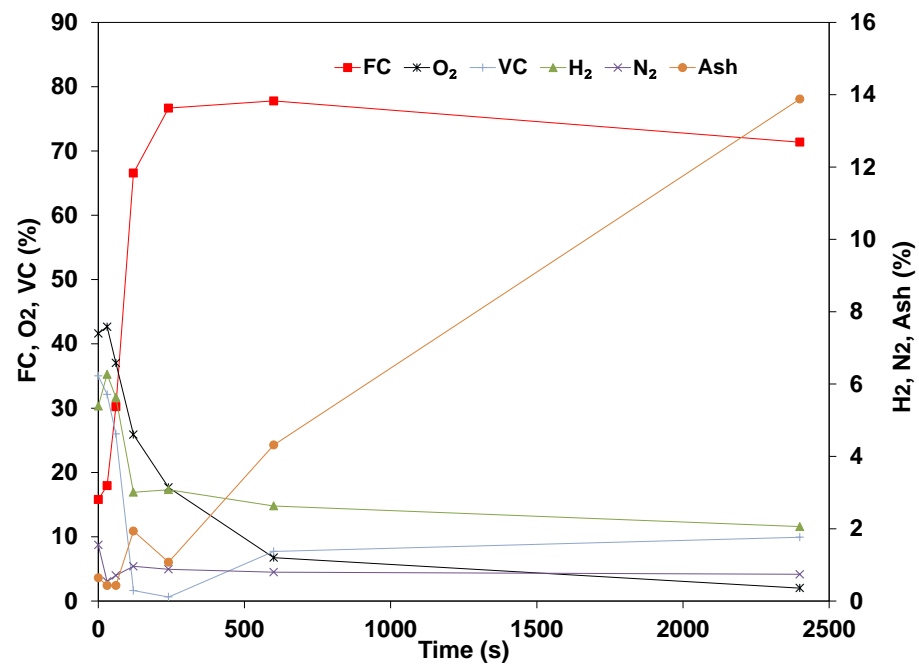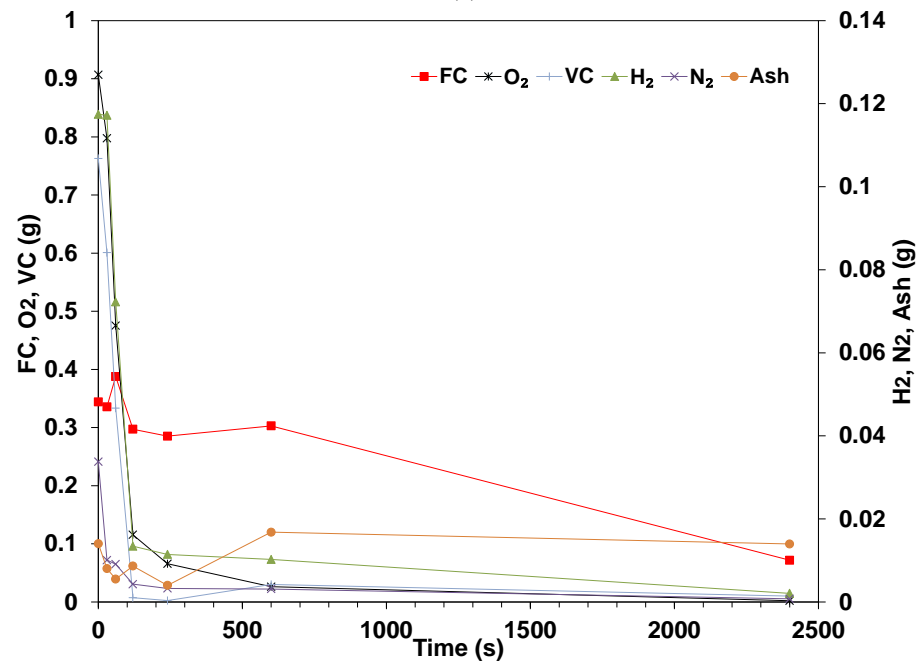
Figure 4. Elemental analysis of wood pellets at 650 °C: (**a**) in percentages and (**b**) in grams.

For each data point in the relationship between the mass loss and temperature, the composition of the remaining fuel was determined in terms of volatiles (C, H, N, O), fixed carbon, and ash. The results are presented in Figure 4 as a mass ratio (a) and as the total mass (b). The data in Figure 4b show that the volatile matter is released in a very short time (at this temperature, within the initial 4–5 min) while the ratio of fixed Carbon and ash steadily increases and, later in the oxidation process, constitutes most of the remaining matter in Figure 4a. After about 5 min, there are no significant changes in the gas composition, which means that only the char combustion is taking place. It is also observed that the rate of conversion for the volatile is similar for all compounds, although Nitrogen shows a higher rate of conversion.

After approximately 10 min, a steady reduction of volatiles is observed which, in the long run, approaches zero, coupled with a much slower oxidation of fixed Carbon. The ash content remains approximately constant, within the accuracy of the instrumentation.

Because the mass loss occurs in the early stages, all subsequent tests are analyzed for that period. Figures 5–10 depict the influence of temperature on the devolatilization process. In these, the time scale was adjusted according to the total reaction time in order to highlight the initial decomposition phase. As mentioned, the tests were carried out at 264, 351, 444, 541, 650, and 734 °C.
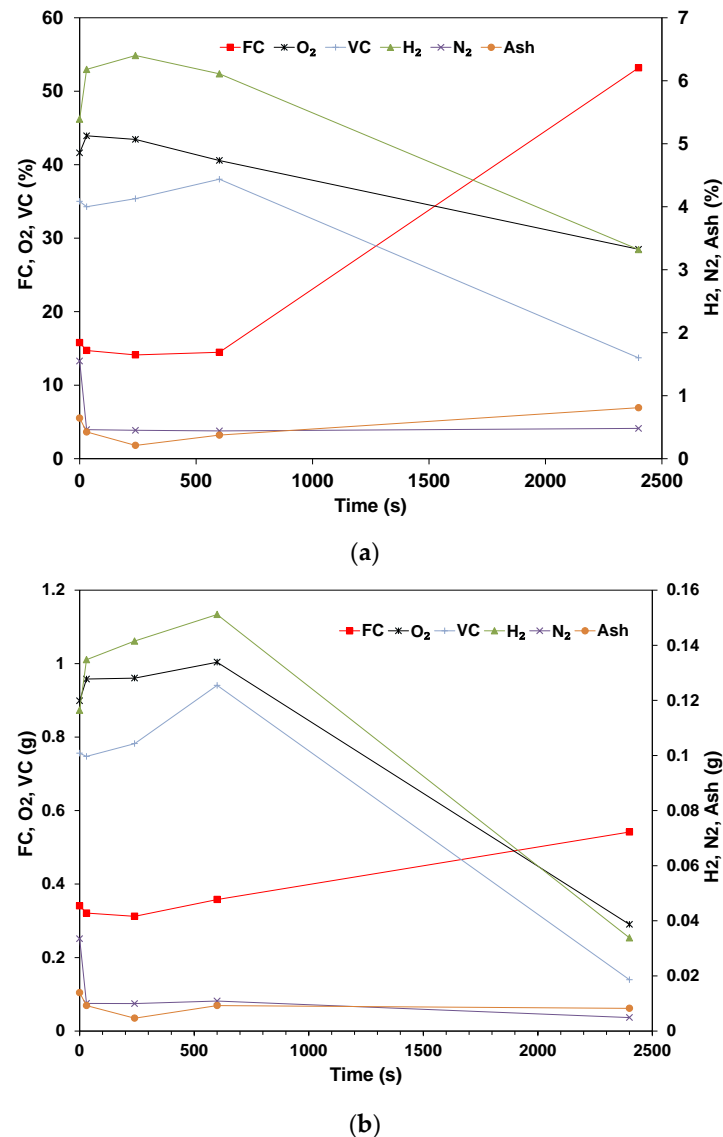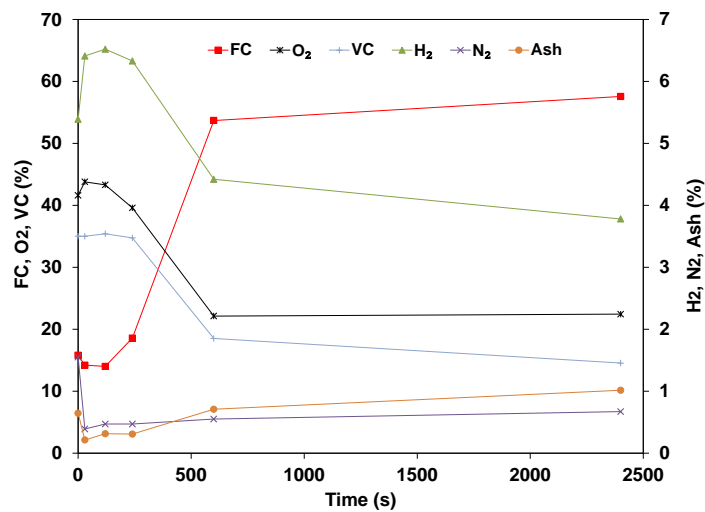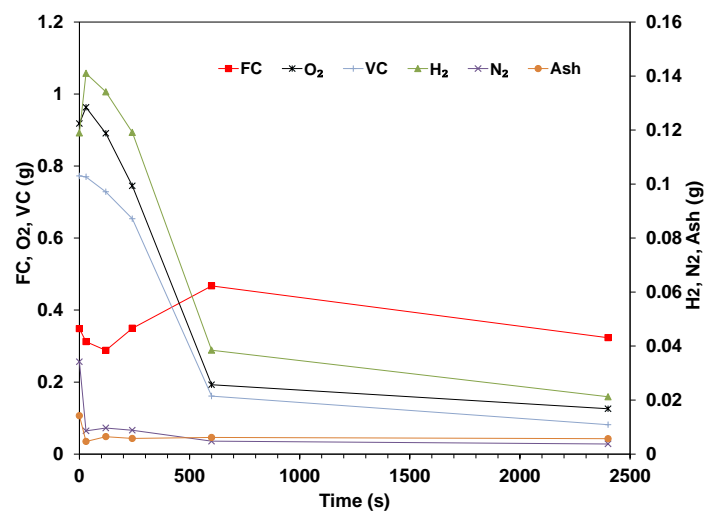

(a)

(b)

Figure 5. Elemental analysis of wood pellets at 264 °C: (**a**) in percentages and (**b**) in grams.
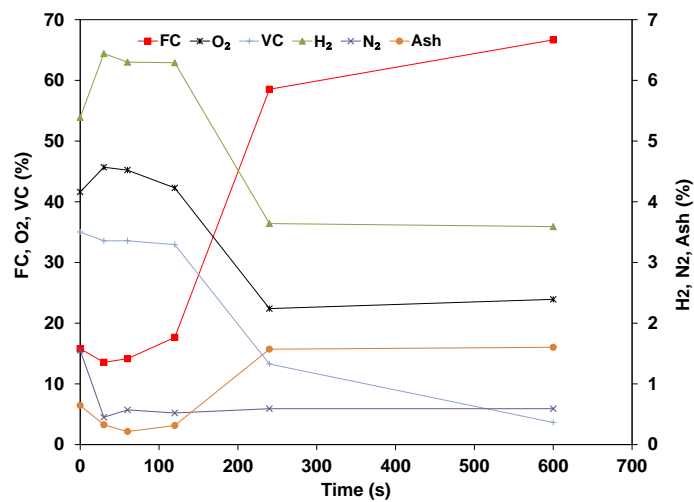
(**a**)

(**b**)

Figure 6. Elemental analysis of wood pellets at 351 °C: (**a**) in percentages and (**b**) in grams.

(**a**)

Figure 7. *Cont.*
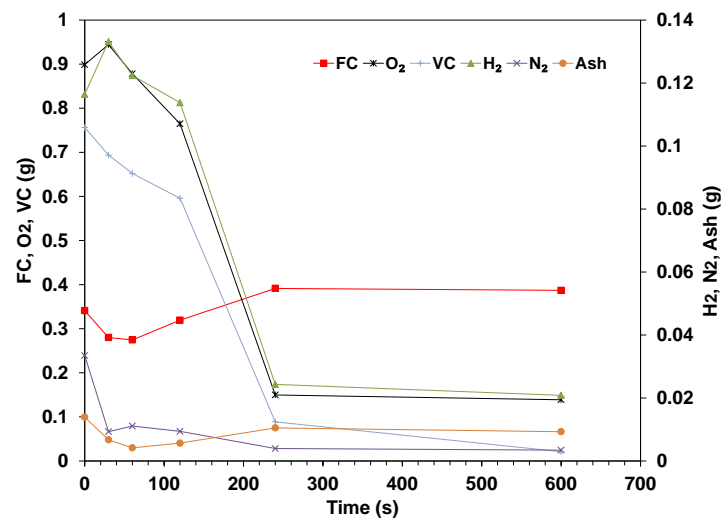
(**b**)

Figure 7. Elemental analysis of wood pellets at 444 °C: (**a**) in percentages and (**b**) in grams.
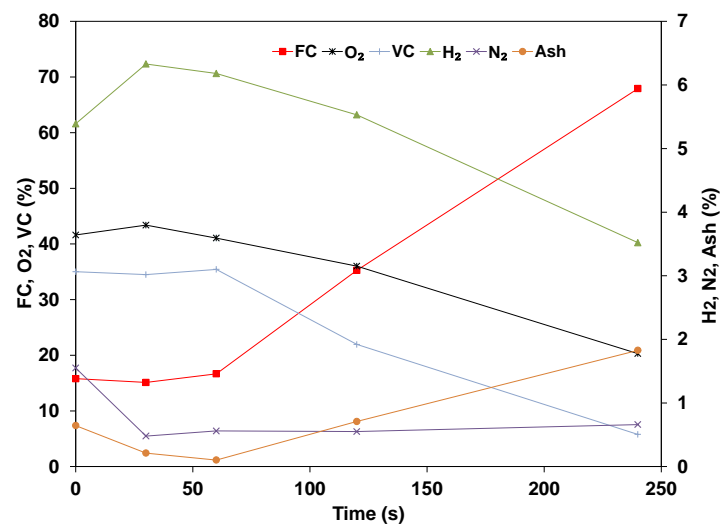
(**a**)

(**b**)

Figure 8. Elemental analysis of wood pellets at 541 °C: (**a**) in percentages and (**b**) in grams.

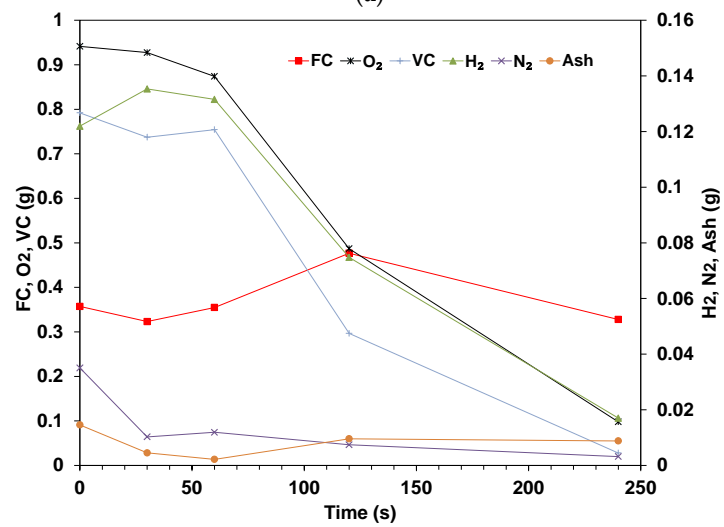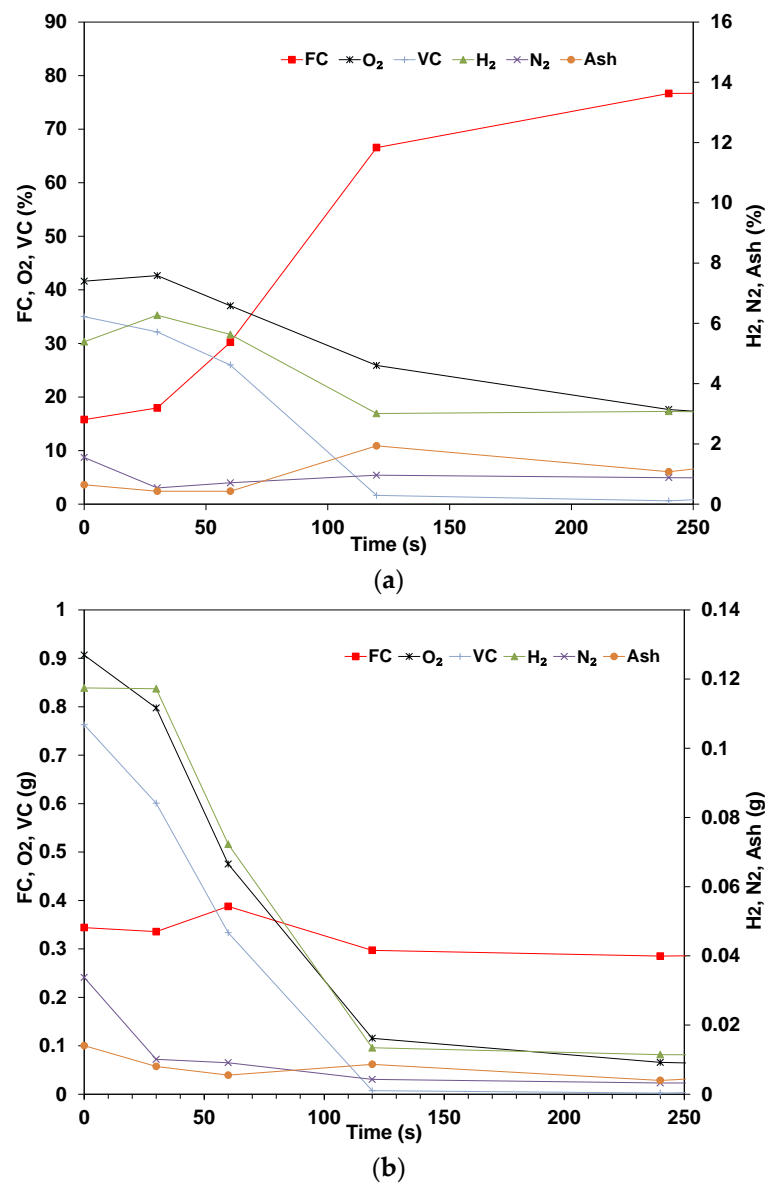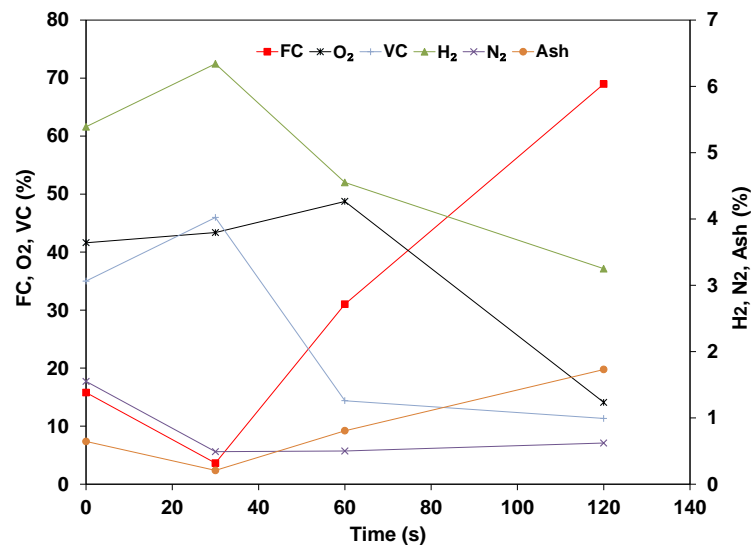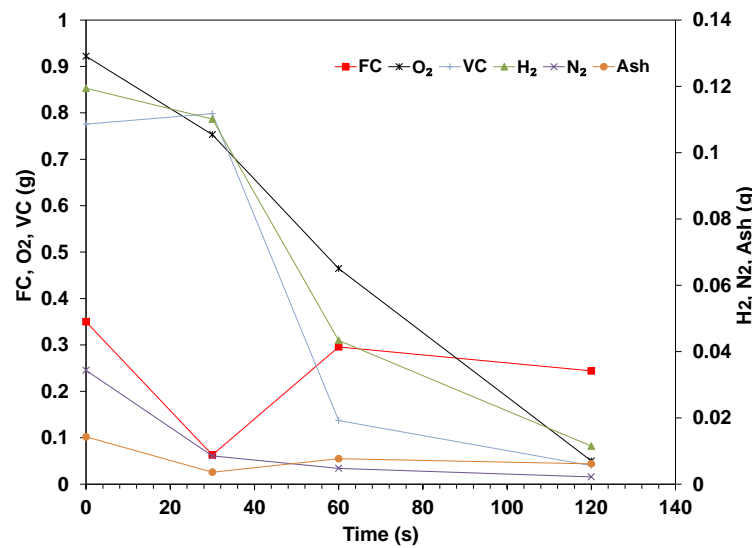**Figure 9.** Elemental analysis of wood pellets at 650 °C: (**a**) in percentages and (**b**) in grams.

From the data, it can be concluded that the rate of mass loss increases with the temperature. This increase in the reaction kinetics coefficient is in agreement with the results obtained with TGA. In all, the pattern is similar to that described above: the volatiles are released at a similar rate, although Nitrogen shows a higher diffusion coefficient. Looking at the data on a mass basis, it is observed that the mass of fixed Carbon increases during the initial devolatilization phase before its subsequent oxidation in the later stages of the devolatilization. This suggests that there is Carbon diffusion from the volatile fraction to the fixed fraction. This mechanism occurs over a period that is inversely proportional to the testing temperature. Taking into consideration that the heating rate is dependent upon the testing temperature, one may postulate that the diffusion between the volatile and fixed fractions of Carbon occurs at a low temperature. It can also be concluded that this time is directly correlated with the devolatilization of other volatiles, such as O, N, and H. Taking as an example, at a temperature of 444 °C (see Figure 7), the maximum mass of fixed Carbon occurs at approximately 240 s, which is the time frame for the nearly complete devolatilization of the volatile matter occurring at the same time. This is observed for all the other test conditions as well. This time is correlated with the testing temperature in Figure 11, where dT is the temperature difference between the reactor and the initial

temperature (20 °C). The data also suggest that the time for Carbon migrating to the fixed fraction tends to level off at high heating rates.

(**a**)

(**b**)

Figure 10. Elemental analysis of wood pellets at 734 °C: (**a**) in percentages and (**b**) in grams.

The total Carbon will decrease with time as it is oxidized. This is observed in Figure 12, where the total mass of Carbon is made dimensionless with the initial mass of Carbon. It shows that the mass loss of Carbon at different temperatures changes with time. This change corresponds to the initial loss of some low molecular weight of Carbon compounds present in the biomass (extractives) as well as to the thermal decomposition of hemicellulose and lignin, and at a higher temperature of the cellulose. According to Yeo et al. [25], these compounds start to decompose at 47 °C and undergo several different stages and temperature ranges. The decomposition of hemicellulose, cellulose, and lignin starts at a temperature between 227–327, 302–427, and 47–827 °C, respectively. The decomposition of these compounds is dependent on the temperature and residence time. When the temperature or residence time increases, the carbonaceous structure is more stable, and the ongoing bond cracking and subsequent mass loss are also observed at a slower rate. Madzaki et al. [26] also stated that the high volatile matter content of the biochar is due

to the decomposition of cellulose, hemicellulose, and lignin. Therefore, the data suggest that some of the Carbon released from hemicellulose and lignin is fixed into the cellulose structure. Figure 12 also shows that at 264 °C, the total mass of Carbon is increasing in the early stages. This may be explained by the short residence time dehydration reactions taking place, which contribute to the elimination of Oxygen and Hydrogen in particular. At higher temperatures, this effect is not noticeable as it is compensated by the faster elimination of fixed carbon.
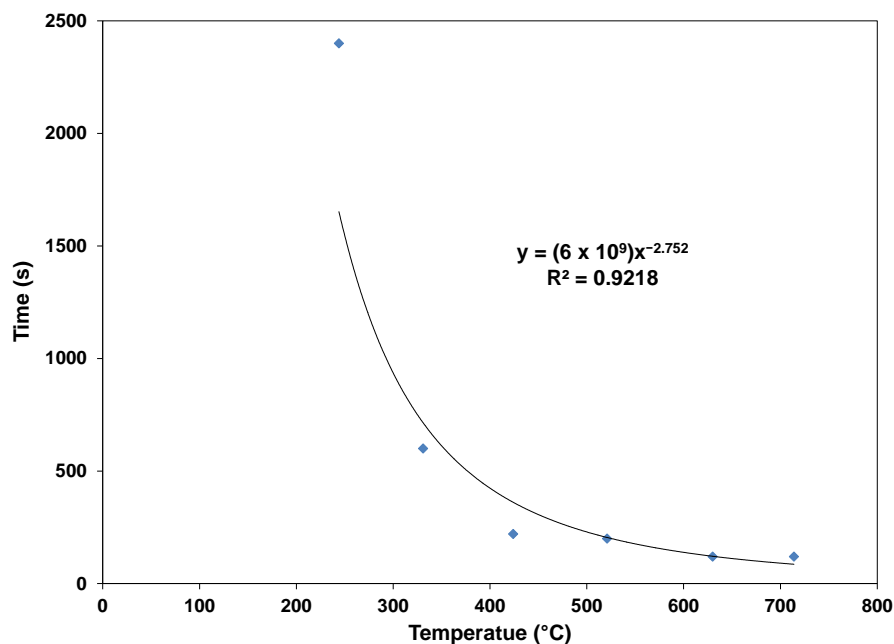

$$y = (6 \times 10^9)x^{-2.752}$$
$$R^2 = 0.9218$$

Figure 11. Fixed Carbon profile at different temperatures during the combustion.

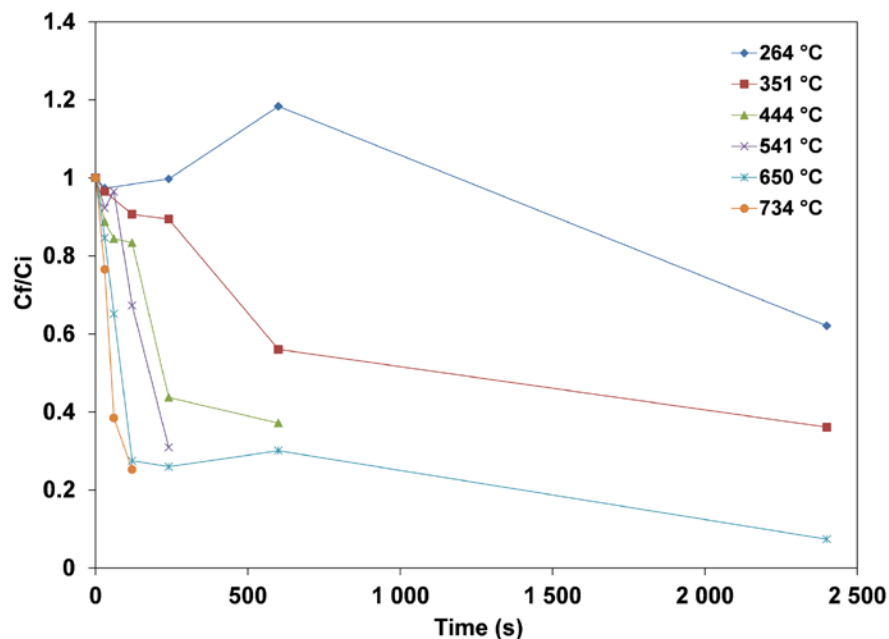**Figure 12.** Carbon ratio for different temperatures.

The data for species concentrations in the sample, presented as a fraction of the sample's mass, as shown in Figures 5a–10a, depict the variation in concentrations resulting from the species' diffusion out of the biomass. Therefore, some are expected to increase in concentration if their rate of volatilization is lower than others. However, if one looks

at the actual mass (Figures 5b–10b), it is observed that H and O show an increase in the total mass present in the sample in the early stages of the devotalization process, once the only source of Oxygen and Hydrogen is the moisture of biomass. Our hypothesis is that during the drying phase, the O and H from the water are integrated into the O and H in the biomass. The time scale for the H and O is the same; that is, the maximum mass occurs at approximately the same time. The maximum O and H mass occur within the time frame of the drying phase (see Figure 3).

To prove this hypothesis, a complementary set of tests was developed. In this, the tests were conducted at 264 °C and were limited in time to the initial 4 min of the devotalization process. The samples were removed from water moisture by drying them according to Swedish standard SS 18 71 20 [22]. The results are presented in Figure 13 in terms of species concentration (a) and on a mass basis (b). The analysis was limited to O and H. The results clearly show that with the dry pellets, the mass of H and O always decreases, which validates the hypothesis that in the drying phase, some of the O and H are transferred from the water to the biomass's structure.
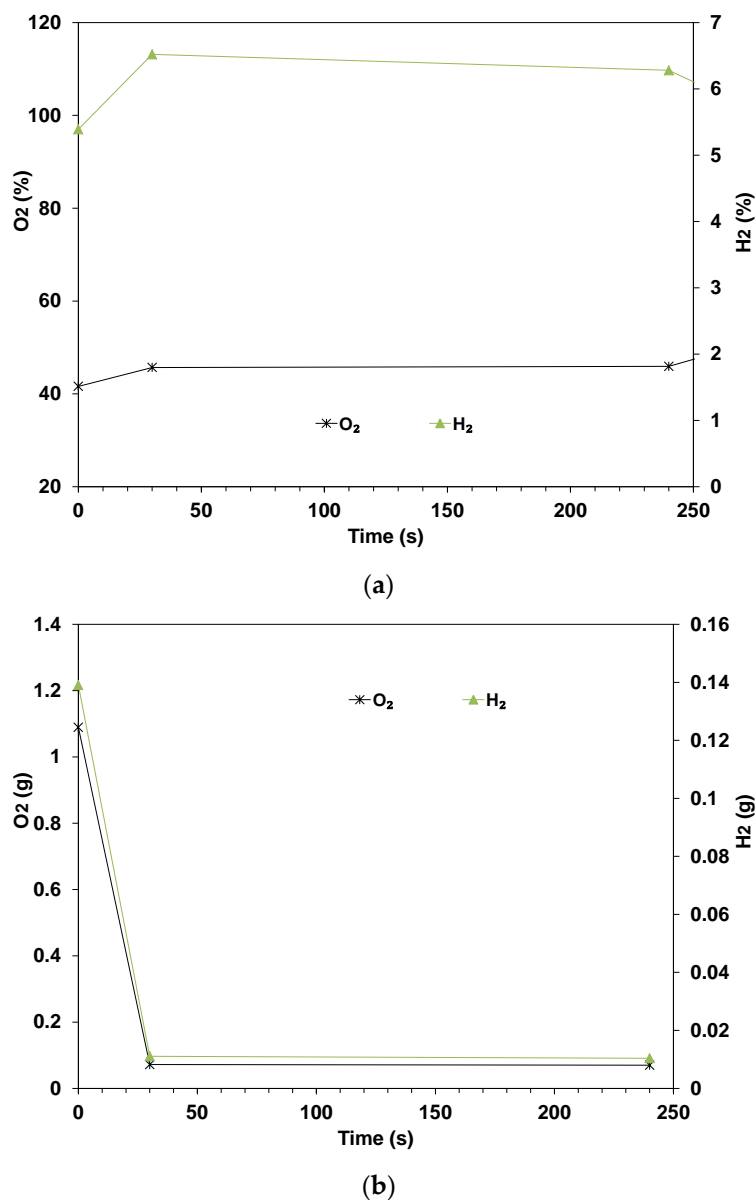

(a)

(b)

Figure 13. O_2 and H_2 concentration of dry pellets at 264 °C: (**a**) in percentages and (**b**) in grams.

4. Conclusions

A small reactor was developed, and an experimental procedure was developed to investigate the devolatilization process of pine wood pellets under controllable conditions. The results are concluded as follows:

- The mass losses increase for any specific temperature, and the higher the temperature, the faster the pellets release the volatile matter. Devolatilization occurs at a very slow rate at low temperatures. The maximum mass loss obtained at the highest temperatures (650 and 734 °C) for a 1-h test leveled off at about 97%, with the remaining substances, including fixed carbon and ashes, being about 3%. The mass loss of dry pellets increases with the temperature, and at a higher temperature, the dry pellets devolatilize faster than wet pellets.

- The elemental results show that the volatile matter is released in the early stages while the ratio of fixed carbon and ash steadily increases. The rate of conversion for the volatile is similar for all compounds, though Nitrogen shows a higher rate of conversion. The mass of fixed carbon increases due to the Carbon diffusion from the volatile fraction. The decomposition of these compounds is dependent on the temperature and residence time applied.

- The total Carbon decreases with time as it is being oxidized, and the species concentration in the sample is expected to increase in concentration if their rate of volatilization is lower than others. The results for dry pellets show that the mass of H and O always decreases, which indicates that in the drying phase, some of the O and H are transferred from the water to the biomass's structure. In addition, the decomposition of biomass is related to hemicellulose, cellulose, and lignin, which start at different temperatures and stages.

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

Abbreviations

Nomenclature
T Temperature (K)
m Mass (g)
t Time (s)
Subscripts
f Final
l Loss
i Initial

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