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ORIGINAL ARTICLE

Pyrolysis of wheat straw pellets in a pilot-scale reactor: Effect of temperature and residence time

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Abstract

Pyrolysis of two types of pellets (T_1: 100% wheat straw, and T_2: 70% wheat straw; 10% sawdust, 10% biochar, and 10% bentonite clay) was performed in a pilot-scale reactor under a nitrogen environment at 20°C to 700°C. This was to investigate slow pyrolysis yields and gas composition as a function of temperature and residence time. The experimental data were obtained between 300°C and 600°C, with a residence time of 90 min, a nitrogen flow rate of 50 cm^3/min, and a heating rate of 20°C/min. The results indicated that the maximum pyrolysis temperature is 605°C with a residence time of 55 min. The product analysis showed that the proportion of gas was higher than that of biochar and bio-oil. The conversion efficiency increased with higher temperatures and varied between 66% and 76%. The results showed that carbon dioxide was the main component in the produced gas, and the maximum gas concentration was 63.6% at 300°C for T_1. The higher temperature and longer residence time increased the syngas ($CO + H_2$) composition for both T_1 and T_2 treatments. Nevertheless, the produced biochar had a high carbon content and retained a high calorific value, indicating slow pyrolysis is the ideal utilization route of wheat straw pellet biomass for biochar.

KEYWORDS

biochar, gas, pyrolysis, reactor, wheat straw pellet

1 | INTRODUCTION

Globally, crops produce vast residues, frequently accumulating in landfills.[1,2] Along with other residues, this accumulated waste is creating disposal problems. Some countries directly burn crop straws, which causes severe environmental pollution. Australian farmers often mix crop residues with soil. However, this concerns intensive (year-round crop cultivation) agriculture. It would be better that these crop residues address energy deficits and replace the consumption of fossil fuels and other nonrenewable resources. Agricultural waste biomass is the most promising source of energy production among all renewable sources (air, water, and solar) because it acts in a carbon-neutral manner.[3] Wei et al.[4] noted that biomass could fulfill about 9.7% of global energy and fuel demand through thermal and biochemical processes. Wheat straw, rice straw, rice husk, corncob, bagasse, cotton stalks, perennial grass, etc., are common agricultural residues.[5,6] The straw comprises 15%–40% of

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agricultural residues.[7] They are good candidates for bioenergy production due to their lower cost, availability, and enormous quantities. Currently, these materials are often used for heat generation and electricity production instead of open-burning.[8] This work chooses agricultural straws, particularly wheat straw, because they have great potential for energy production in both developed and developing countries.[6]

Energy (Syngas) production from biomass by thermochemical conversion (pyrolysis and gasification) has recently increased.[9] The yield and the composition of pyrolysis products depend on particle heating rate, gas residence time, and reactor temperature and pressure.[10] However, tar formation during the pyrolysis and gasification processes could degrade the producer's gas quality and restrict its use.[11] Therefore, the main obstacles are often determining how much tar to remove and maximizing the output of gaseous products from biomass.

Pyrolysis is a thermal decomposition process that transforms biomass into bioenergy without additional air (Table 1).[20] In addition, Snyder[21] noted that pyrolysis is a possible thermal conversion route from biomass to energy and offers good advantages. Because of the chemical nature of biomass, pyrolysis has been demonstrated to be a suitable method for converting biomass waste into more valuable materials.[22,23] Biomass pyrolysis can create three products: carbonaceous biochar, bio-oil (a complex mixture of liquid hydrocarbons), and syngas (mainly made of H_2, CO, and CH_4) (Figure 1).[24,25] The yield and quality of pyrolysis products vary depending on biomass type and characteristics, as well as operation factors like temperature, pressure, heating rate, residence time, and so forth.[26] Moreover, the reactor-related factors also influence reactor outputs.[27]

The thermal degradation method is linked to the weight loss profile concerning time and temperature.[28,29] This technique is strongly influenced by thermogravimetric tools or reactors.[30,31] Numerous researchers have investigated the impact of temperature on biomass pyrolytic products using different reactors.[5] Typical reactors such as screw reactors, fixed-bed reactors, tubular reactors, and rotary kilns were used to pyrolyze various types of biomass.[32] From the viewpoint of energy transformation, fixed bed pyrolysis is more attractive among different thermochemical conversion processes because of its simplicity and higher conversion capability of biomass and its solid wastes into a liquid product.

Many authors have studied the impact of operating conditions on pyrolysis products using experimental and numerical approaches.[33,34] The common parameters are residence time and temperature. Hossain et al.[35] experimented with the design, fabrication, and performance study of a biomass solid waste pyrolysis system for alternative liquid fuel production. They used the Devdaru (Polylthia Longifolia) seeds in pyrolytic oil in a fixed-bed reactor. It was found that parameters such as reactor bed temperature, running time, and feedstock particle influenced the product yield significantly. The obtained HHV of Devdaru seed oils was 24.22 MJ/kg. Tanoh et al.[9] examined the effects of temperature

TABLE 1 Summary of previous research on wheat straw pyrolysis conducted in various reactors.

Reactor types	Investing area	References
Screw reactor, Continuously fed bubbling	Pyrolysis mass yield, proximate and ultimate analysis, higher and lower heating value	Biswas et al.[12–14]
Fluidized bed reactor Fixed bed tubular reactor	Surface area, pH, and pore volume of the solid product (Biochar)	Min et al.[15–17]
Rotary kiln	The cation exchange capacity and electrical conductivity of char	Kloss et al.[18,19]

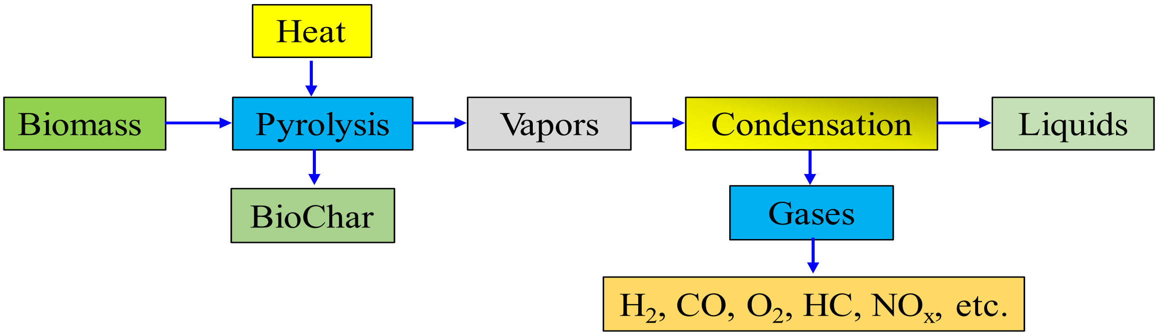


FIGURE 1 Process flow chart of the laboratory-scale pyrolysis along with the potential products.

(550–850°C) on wood pyrolysis products in a laboratory-scale rotary kiln. They assessed the product yields but did not analyze gas composition or tar content.

They focused mainly on mass yields, the proximate analysis, the ultimate analysis, higher heating value, and biochar product quality (surface area, biochar pore volume, PH, electric conductivity, cation exchange capacity, etc.). Most of these abovesaid studies focused on wood biomass and considered rotary types of reactors. In addition, straw pyrolysis has received interest in the literature shown in Table 1. However, recent research was done on wheat straw pyrolysis.[13,36] No studies were found in the literature about the pyrolysis of wheat straw pellets in fixed-bed small-scale kiln reactors. This type of oven primarily has the potential for biochar identification. Moreover, this kiln is low-cost and suitable for on-farm use.

Hence, this study aims to analyze wheat straw pellets' pyrolysis process to maximize the syngas yield on a small scale suitable for the on-farm level. The investigation was conducted in a pilot-scale fixed-bed oven to determine product yields, gas composition analysis, qualitative and quantitative analysis of tar, and char characterization.

Pyrolysis can be classified based on temperature and residence times (length of time biomass stays in the reactor's active zone) listed in Table 2.[38] Fast pyrolysis processes are designed to maximize the gas portion,[39] while intermediate pyrolysis focuses on the liquid part.[40] The fast and intermediate pyrolysis works in high temperatures (over 500°C) and less residence time (<1 min); hence, the process is complicated and cost involved. Also, this sophisticated system is suitable for the industrial scale. In addition, slow pyrolysis focuses on biochar production while having opportunities to produce bio-oil and biogas[41]). This pyrolysis operates at around 400°C with a long residence time (hours).[42] Based on the literature review, slow pyrolysis is widely accepted for small-scale reactors. Therefore, this study considered slow pyrolysis in a fixed-bed reactor to investigate the pellets.

2 | MATERIALS AND METHODS

2.1 | Feedstock and biochar analysis

A total of seven pellet types were manufactured with a focus on utilizing wheat straw, aiming to identify the most suitable pellet according to ISO standards.[43] However, this investigation used two distinct types of pellets as feedstock. First, the T_1 pellet was exclusively composed of 100% wheat straw, yielding the lowest quality rating as per the findings of Nath et al.[29,44] Conversely, the T_2 pellet was concocted from a blend comprising 70% wheat straw, 10% bentonite clay, 10% charcoal, and 10% sawdust, aligning with the predetermined suitability criteria stipulated by the ISO pellet standard. Consequently, for a comprehensive and insightful analysis, these particular pellets were selected as the focal points for examination and evaluation.

The pellets and biochar chemical analysis was conducted at the Feed Central Laboratory in Toowoomba, Queensland, Australia. Triplicate measurements were carried out for each sample to ensure accuracy and reliability.

The primary components of biomass, namely lignin, cellulose, and hemicellulose, were accurately determined using the acid detergent fiber (ADF), acid detergent lignin (ADL), and neutral detergent fiber (NDF) techniques.[44] These methods, which adhere to the Association of Official Analytical Chemists (AOAC) standard technique 973.18—Fiber (Acid Detergent)[45] and AOAC standard method 992.16,[46] are widely recognized for their accuracy and precision. The cellulose and hemicellulose content was then calculated from ADF and lignin, providing a comprehensive understanding of the biomass composition.[47]

The Hach method was employed to determine the moisture content and volatile compounds.[48] Additionally, AOAC standard method 942.05 was used to determine the total ash, where a 2–3 g sample was burned in a furnace above 700°C in an oxygen environment.[49] The remaining

TABLE 2 Conditions of the various pyrolysis processes and end products[37]

	Factors		Yield		
Type of pyrolysis	Temperature, °C	Residence time	liquid	Solid (char)	Gas
Slow	~400	Hours to days	75%	12%	13%
Intermediate	~500	~10–30 s	50% (in two phases)	25%	25%
Fast	~500	~1 s	30%	35%	35%
Gasification	750–900	-	5%	10%	85%
Torrefaction	~290	~10–60 min	5%	-	-

material after volatile matter loss was then measured. Finally, the fixed carbon (FC) content was calculated as FC (%) = 1 − Mass − Ash − Volatile Matter (dry basis) (ASTM 2013). The solid material's gross energy, or the gross heating value, was quantified as calories per gram. This was done in accordance with the ASTM D5865-03 standard (ASTM 2003), using the IKA C2000 basic oxygen bomb calorimeter for thermal analysis. The instrument was set to IKA's dynamic mode with an outer vessel temperature of 25°C. One gram of Parr standard benzoic acid was used to calibrate the calorimeter to ensure accuracy. For the gross energy determination, approximately 0.50 g of the sample was placed in the combustion capsule and inserted into the sample holder head of the bomb calorimeter, providing a precise measurement of the biomass's energy content.

An essential criterion for a material's combustibility is its gross calorific value (GCV), which is the amount of energy released per unit of fuel burned. Based on analytical techniques, three types of energy were determined from biomass: higher heating value (at constant volume—dry basis), lower heating value (at constant pressure—dry basis), and gross calorific value (at constant pressure —wet basis or as received).[50] This investigation used the gross calorific value for all pellets because it is the most practical measurement. Total organic carbon (TOC) and nitrogen were quantified using the CN628 carbon/nitrogen determinator. Additionally, AOAC 990.03 was followed for protein (Crude) determination in Animal Feed.[51] The sulfur content was determined using the CEM Application Notes for Acid Digestion method.[52] Finally, the oxygen content was calculated by the difference in elements.

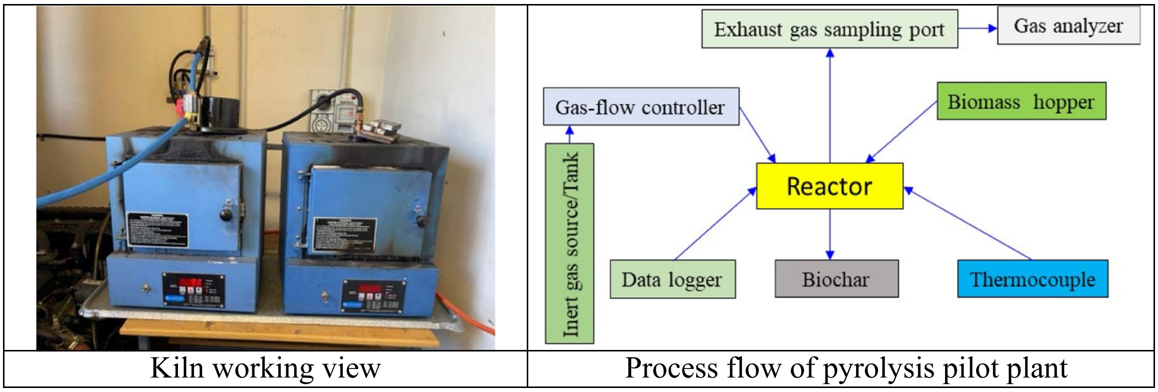
2.2 | Pyrolysis reactor

Pyrolysis experiments were conducted using a laboratory-scale kiln, specifically the Rio Grande PMC model #703-117, as illustrated in Figure 2. The primary components of the reactor unit comprise an electric heating system, a port for introducing air or nitrogen gas, an electrical data plate, and a feedstock burning chamber (source: https://products.riogrande.com/content/Instruction-Sheets/Rio-Grande-PMC-Kilns-With-AF4X-Controller-IS.pdf). Additionally, the unit is equipped with a measuring facility for monitoring gas and temperature, with its inner wall covered by ceramic-fiber-firing embedded elements. The firing internal chamber measures 20, 18.7, and 14.6 cm in width, length, and height, respectively, where the pyrolysis process occurs. The reactor is heated by electrical resistance at 11.4 A and 110 V. The reactor has a maximum working temperature of 1093°C, with a power output of 1370 Watts—the hopper, with a capacity of 2 kg, where biomass was firing. The reactor offering five programs for firing, the "PMC + ™ slow" option, was selected for this research to investigate pellets under slow pyrolysis conditions. The resulting gas exits the reactor from the upper part. The gas collection port or analyzer connection point is also in the upper part.

2.3 | Experimental procedures

Wheat straw pellets (WSP) were pyrolyzed in a small-scale kiln at varied temperatures (Table 3). Each pellet sample was approximately 500 gm in this study. For this research, nitrogen was introduced to the top of the reactor at a 50 cm^3/min flow rate as carrier inert gas. The experimental temperature and residence time were set using a controller programming display (P1 for Pyrolysis). All experiments were conducted twice to ensure repeatability, where the relative error was less than 5%, and all the samples were measured under the same conditions.[9]

The feedstock (pellet) was placed in a ceramic hopper inside the pyrolysis chamber. Due to the highly inhomogeneous composition and distinctive properties



Kiln working view

Process flow of pyrolysis pilot plant

FIGURE 2 Schematic diagram illustrating the laboratory-scale kiln setup. Kiln working view. Process flow of pyrolysis pilot plant.

TABLE 3 Experimental parameter.

Particular	Value
Pyrolysis	Slow
Temperature range, °C	25–700
Data recorded temperature, °C	300, 350, 400, 450, 500, 550, 600
Heating rate, °C/min	20
Nitrogen flow rate, cm^3/min	50
Pellet diameter, mm	7–8.2
Pellet length, mm	22–40
Biomass used, g	500
Residence time, min	90

of pellets,[53] resulting in process instabilities may lead to incomplete pyrolysis and other issues. The choice of burning agents (nitrogen, air, and steam) also influences the pyrolysis products. The outcome of pyrolysis also depends on the thermal behavior of WSP, with thermal conductivity and specific heat values of 0.13 W/m K and 1.63 kJ/kg K at 300 K, respectively.

The candidates' feedstock (pellet) average diameter and length were 35.0 and 8.0 mm, respectively. Correspondingly, the bulk density was measured at 244.79 and 607.40 kg/m^3 for the T_1 and T_2 pellets.

2.4 | Temperature and time measurement

The reactor has a temperature measurement facility and a data logger. The pyrolysis temperature was recorded as the biomass bed temperature inside the reactor, and this information was automatically stored. The pyrolysis process initiates at room temperature (25°C), with a heating rate of 20°C/min. The test continued for 90 min, reaching its peak temperature. According to the recommendation by Phounglamcheik et al.,[54] it is advisable to maintain residence times under 90 min to optimize biomass thermal conversion efficiency.

2.5 | Pyrolysis yield

This study used a laboratory-scale kiln to assess gas and char production. In the context of this pyrolysis process, the term "conversion" refers to the quantity of materials transformed into liquid or gaseous products, as defined by Ethaib et al.[55] The solid residue remaining in the reactor after the reaction is designated as biochar.

Bio-oil represents the condensable phase of a substance, consisting of water and vapors. This study measured the bio-oil at seven temperature levels (300, 350, 400, 450, 500, 550, and 600°C) in a nitrogen environment. The reference bio-oil information was aligned with Biswas et al.[13] and Paul et al.[56] Several equations (1–4) are used to calculate gas, biochar, bio-oil, and conversion:[57,58]

$$\text{Bio-oil yield, wt\%} = \frac{W_4 - W_3}{\text{Weight of feedstock}} \times 100, \tag{1}$$

$$\text{Biochar yield, wt\%} = \frac{W_2 - W_1}{\text{Weight of feedstock}} \times 100, \tag{2}$$

$$\text{Gas yield, wt\%} = 100 - (\text{Bio-oil yield} + \text{biochar yield}), \tag{3}$$

$$\text{Conversion, \%} = 100 - \text{Biochar yield, wt\%}, \tag{4}$$

$$\begin{aligned} &\text{where } W_1 = \text{Weight of empty reactor, g}, \quad W_3 = \text{Weight of emphty measuring cylinder g}, \\ &W_2 = \text{Weight of reactor after reaction, g}, \quad W_4 = \text{Weight of measuring cylinder with bio-oil, g}. \end{aligned}$$

Accurate measurement of experimental sample weight is crucial for obtaining reliable results. The specific procedure depends on the sample type, desired accuracy, and available equipment. Analytical balances are preferred for highly precise measurements due to their sensitivity to weight changes. An electrical balance (NMI 6/4C/280) was used to measure pellets, biochar, and reactors. Triplicate measurements were conducted for each sample to ensure accuracy and reliability.

2.5.1 | Gas sampling and analysis

The thermal gas is a condensable phase of pyrolysis vapor[59] and is released from the reactor as the pyrolysis process commences. The gaseous products were examined using Agilent 7820A gas chromatography (Agilent Technologies) (Figure 3). The thermal conductivity detector (TCD) for the gas chromatography was made up of three columns such as Ultimetal HayesepQ T 80/100 mesh (H_2, CO, and CH_4), Ultimetal Hayesep T 80/100 mesh (CO_2), and Ultimetal molsieve13 80/100 mesh column (N_2 and O_2).[60] The temperature for all columns was maintained at 60°C, with argon serving as the carrier gas.

At the top of the pyrolysis reactor, a gas sample port facilitates gas release for analysis. For gas analysis, the pyrolysis vapor was collected in a Tedlar bag and subsequently analyzed using Agilent gas chromatography. This study investigated the gas composition at temperatures 300, 350, 400, 450, 500, 550, and 600°C.

2.5.2 | Biochar measurement

Biochar is non-volatilized in biomass fraction.[59,61] This study investigates explicitly char reactivity at varying temperatures, namely 300, 350, 400, 450, 500, 550, and 600°C. Following the pyrolysis process, the reactor underwent a cooling period of at least 2 h in an oxygen-free environment, during which the pyrolysis products were released and subsequently weighed. After this cooling period, the residual material remaining in the oven was identified as biochar or carbon-enriched ash resulting from the pyrolysis process.

For the measurement of heating value, an IKA C2000 basic oxygen bomb calorimeter was employed, following the ASTM D5865-03 standard (ASTM 2003). The calorimeter underwent standardization using 1.00 g of Parr standard benzoic acid, with a dynamic mode and an outer vessel temperature of 25°C. For gross energy determination, approximately 0.50 g of the sample was placed in the metal combustion capsule and positioned in the sample holder in the bomb head.

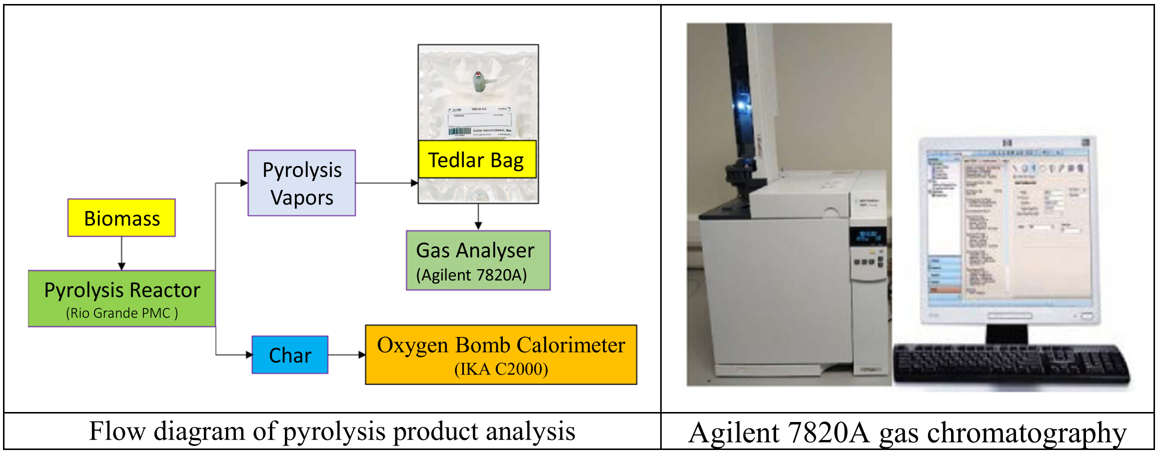
3 | RESULTS AND DISCUSSION

3.1 | Biomass fuel (pellet) characterization

Chemical analysis is important because biomass's mineral matter and organic composition significantly influence the distribution of pyrolysis products and their properties.[62] Table 4 presents assessments of the ultimate analysis, proximate analyses, and pellets' higher heating value.

Proximate analysis results are typically used to complete the energy balance of the pyrolysis process. In the case of the T_1 pellet, the proximate analysis indicates a higher volatile matter content (75.61%) than the T_2 pellets (53.03%). Conversely, the ash content of T_2 pellets is 11.87% higher than that of T_1 pellets (7.09%). The higher ash content in T_2 pellets may be attributed to including bentonite clay in the wheat straw mixture, as bentonite clay contains a substantial ash fraction (89.63%).[63] Feedstocks with higher volatile fractions and lower ash content tend to produce more syngas during pyrolysis and show lower biochar yields.[64] Additionally, the ash content in agricultural straw, such as wheat straw, is generally higher than in wood.[65] Consequently, a higher ash content in the product may increase the presence of solid particles, such as char and tar.

The higher heating value (HHV) of WSP is influenced by a higher carbon content, reflecting its energy content. Similar to other studies, the HHV for T_1 and T_2 pellets exceeded 17.0 MJ/kg.[66,67]



Flow diagram of pyrolysis product analysis

Agilent 7820A gas chromatography

FIGURE 3 Working principle of gas analyzer. Flow diagram of pyrolysis product analysis. Agilent 7820A gas chromatography.

The ultimate analysis provides insight into the carbon content and the nature of organic compounds in the feedstock. The ultimate analysis components for treatments T_1 and T_2 were remarkably similar, with carbon content of 44.32% and 45.87% for T_1 and T_2, respectively. Consequently, the pyrolysis yields may be comparable for both pellet types (Table 4).

TABLE 4 Chemical analysis of pellets.

	Pellets	
Features	T_1	T_2
Proximate analysis (wt% as received, db)		
Moisture	6.20 ± 0.12	3.50 ± 0.08
Volatile matters	75.61 ± 3.4	53.03 ± 4.71
Fixed carbon	11.10 ± 0.16	31.60 ± 2.35
Ash	7.09 ± 0.09	11.87 ± 1.25
Calorific value, HHV (MJ/kg)	17.02 ± 1.6	19.06 ± 1.03
Ultimate analysis (wt% as received, db)		
Carbon	44.32 ± 3.7	45.87 ± 6.1
Hydrogen	4.90 ± 0.06	6.30 ± 0.07
Nitrogen	0.56 ± 0.03	0.72 ± 0.39
Sulfur	0.11 ± 0.01	0.21 ± 0.6
Oxygen (by difference)	50.11 ± 5.23	46.9 ± 3.98
Composition (wt% as received, db)		
Hemicellulose	22.40 ± 3.1	23.30 ± 2.9
Cellulose	41.30 ± 5.07	30.00 ± 3.31
Lignin	7.00 ± 0.05	10.60 ± 0.67

Note: T_1: 100% WS; T_2: 70% WS + 10% SD + 10% BC + 10% BioC; ±: Standard Error.

Abbreviations: BC, bentonite clay; BioC, Biochar; db, dry basis; WS, wheat straw, SD, sawdust.

Wheat straw and straw pellets are classified as lignocellulosic biomass. Zaman et al.[68] noted that wheat straw typically contains an average lignin, cellulose, and hemicellulose content of 15%–20%, 33%–40%, and 20%–25%, respectively. As seen in Table 4, the T_1 pellet has lower lignin (binding agent) than T_2, as the additives in T_2 pellets increase lignin content. Khan and Mubeen[69] noted that wheat straw contains 11%–26% lignin, typically. The hemicellulose component decomposes between 220°C and 315°C temperatures, while cellulose pyrolysis occurs at a higher temperature range of 315°C to 400°C. Among these components, lignin is the most resistant to decomposition, encompassing a broader temperature range extending up to 900°C.[13]

3.2 | Temperature variation with time

Figure 4 illustrates the typical set of data (time vs. temperature) for pellet (T_1 and T_2) pyrolysis. A constant heating rate of 20°C/min was maintained throughout the current experiments, with a projected residence period of 90 min. Pyrolysis started at room temperature, and it was observed that the temperature increased steadily from the start of the experiments, reaching 605°C at the 55-min mark. Following this point, the temperature remained stable, sustaining this level for the remaining 35 min, thereby indicating that the maximum pyrolysis temperature attained was 605°C (Figure 4). Notably, these findings align with those of Tanoh et al.,[9] despite the different reactors and feedstock (wood pellet) utilized

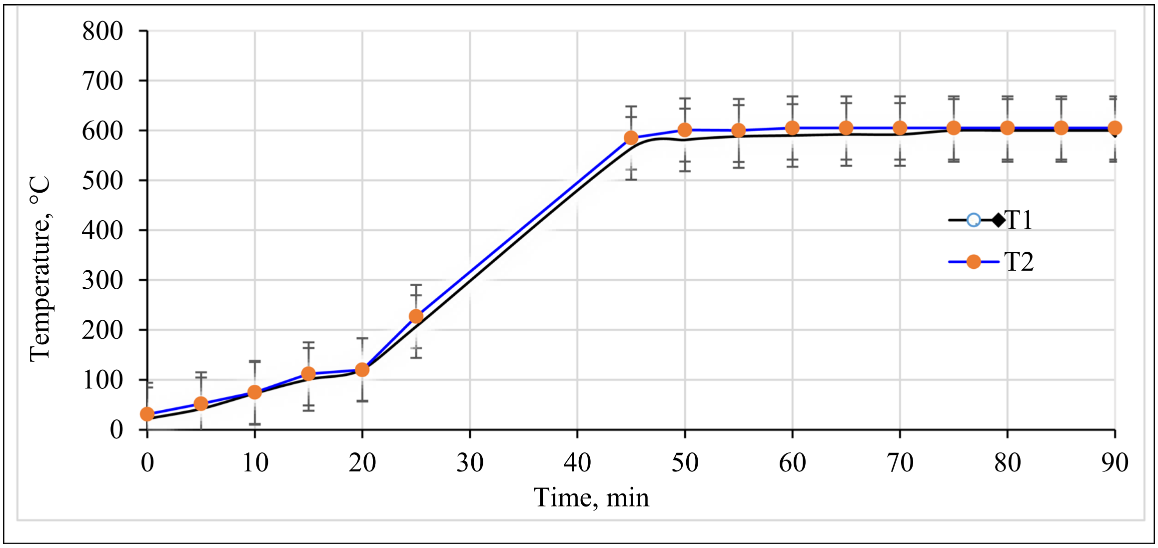


FIGURE 4 Correlation between pyrolysis temperature and duration over time.

in their study. In addition,[70] mention that the optimum pyrolysis temperature varied from 500°C to 700°C, which supported the present research.

Based on the finding above (maximum pyrolysis temperature), data relating to pyrolysis yields (char, gas, and bio-oil) were collected at temperature intervals of 300, 350, 400, 450, 500, 550, and 600°C, with sampling continuing for 50 min.

3.3 | Burning mechanism of pellets

The bulk density of pellets differed between T_1 and T_2, measuring 244.7 and 607.4 kg/m^3, respectively, with a slight variation in ignition time. Figure 5 illustrates the burning behavior of WSP over time. As time progresses, WSP gradually darkens and ultimately transforms into an ash color (Figure 5). Within the initial minutes of ignition, the emergence of gray smoke is observed, signifying the release of volatile matter. This stage is termed the initial gas phase.[71] During this gas phase, temperature gradients facilitate drying and devolatilization, initiating the respective reactions.[5] Subsequently, the surface temperature of the pellet increases, exposing the pellet to a heated environment. Through conduction and convection processes, heat is transmitted from the pellet surface to its center, releasing gas and forming remaining solid residues such as char and ash.[9] During burning, cellulose, hemicelluloses, and lignin generate water vapor and light gases like CO_2, CO, CH_4, and char.[13]

3.4 | Product (yield) distribution

The temperature has a noticeable effect on pyrolysis yields. In investigating pyrolysis products, temperatures ranging from 300°C to 600°C were considered within a nitrogen environment under atmospheric pressure. Zhang and Ma[72] have noted that wood pyrolysis typically initiates at temperatures between 200°C and 300°C. Consequently, the assumptions made in the present study align with the findings reported by Fahmy et al.[73] However, it's important to note that this specific temperature range can vary based on factors such as biomass type, reactor type, and the nature of the pyrolysis process.[36] Table 5 provides an overview of the pyrolysis product yields of pellets. Notably, the highest bio-oil yield, including water, was recorded at 43.61% at a temperature of 450°C, while the minimum yield was 30.24% at 600°C.[56]

In the examined reactor, the maximum biochar yield for T_1 and T_2 treatments was determined to be 33.9% and 32.5%, respectively, occurring at 300°C (Table 5). The observed reduction in biochar yield with increasing temperature may be attributed to heightened decomposition of primary products, such as hemicellulose and cellulose, or secondary degradation of char residues. These findings align with previous research studies.[12,74]

Table 5 shows the gas production potential of each biomass at specific pyrolysis temperatures. The highest gas yields for both pellets were observed at 600°C (T_2: 43.52%, T_1: 41.85%), consistent with the findings of Nanda et al.[7] This increase in gaseous product yield correlates with the secondary cracking of pyrolysis vapors at higher temperatures.[75] Additionally, the secondary decomposition of char at elevated temperatures may contribute to noncondensable gaseous products, further augmenting gas yield with increasing pyrolysis temperature. In summary, the rise in gaseous product yield is concurrent with decreasing oil and char yields as temperature increases. The total sum of all pyrolysis products falls within the range of 96.8% to 98.21%. This range is consistent with the literature findings of Sedmihradská et al.,[36] who employed wheat and barley straw as pyrolysis feedstock.

Before burning (Wheat straw pellets)

After 25 minutes

End of burning (90 minutes)

FIGURE 5 Observation of wheat straw pellet (T_2) burning processes. Before burning (wheat straw pellets). After 25 min. End of burning (90 min).

TABLE 5 Slow pyrolysis product yield.

Treatments	Temperature, °C	Bio-oil, %	Biochar, %	Gas, %	Total,[a] %	Loss, %
T_1	300	32.5	33.9	30.4	96.8	3.2
	350	36	32.1	29.93	98.03	1.97
	400	36.7	31.8	28.89	97.39	2.61
	450	43.61	30.3	23.79	97.7	2.3
	500	42.3	28.68	27.23	98.21	1.79
	550	35.61	26.81	35.57	97.99	2.01
	600	30.24	25.31	41.85	97.4	2.6
T_2	300	32.5	32.5	32.79	97.79	2.21
	350	36	32.2	29.0	97.2	2.8
	400	36.7	30.8	29.39	96.89	3.11
	450	39.61	29.6	28.59	97.8	2.2
	500	42.3	27.67	26.83	96.8	3.2
	550	35.61	25.78	36.58	97.97	2.03
	600	30.24	24.33	43.52	98.09	3.2

[a] *Note*: Total = Bio-oil + Biochar + Gas.

3.5 | Biomass conversion

This study estimated biomass conversion using Equation (4),[58] which is based on the remaining biochar after combustion. The conversion rate ranged from 67.5% to 75.67% for T_2 pellets at temperatures ranging from 300°C to 600°C, while for T_1, it varied from 66.1% to 74.69%. These results are consistent with the findings of Biswas et al.,[13] who investigated the pyrolysis of various agricultural biomass (corn cob, wheat straw, rice straw, and rice husk) and reported a conversion efficiency ranging from 64% to 76% for all biomass types. The observed trend indicates that the conversion rate increased with temperature, while the biochar yield decreased due to the primary decomposition of hemicellulose and lignin (Krishnamoorthy, Krishnamurthy & Pisupati 2019). Overall, the yield data from T_1 and T_2 pellets at various temperatures suggest that compositional variations in different pellets significantly impact product distribution (Figure 6).

3.6 | Characterization of produced gas

Burning of a material generates gases such as H_2, CO_2, O_2, N_2, CH_4, and CO, which can be quantified using a thermal conductivity detector (TCD) in an Agilent Gas Chromatograph.[60] This study specifically measured the primary components of the produced gas. Methane (CH_4), carbon monoxide (CO), hydrogen (H_2), and water (H_2O) were identified as the primary constituents of the pyrolysis gas, with only a small amount of oxygen (O_2) and nitrogen (N_2) present.[76]

3.6.1 | Relationship between pyrolysis temperature on gas concentration

The gas composition under different temperatures is shown in Figure 7. As observed, the predominant component of the gas was carbon dioxide (CO_2), primarily produced through the decarboxylation reaction at relatively low temperatures.[77] The CO_2 content ranged from 63.6% to 50.59% for T_1 pellets and 60.11% to 47.53% for T_2 pellets. A study on wheat and barley straw pyrolysis by Sedmihradská et al.[78] aligned with the present results, showing CO_2 percentages ranging from 61.26% to 39.46% with temperature variations between 400°C and 800°C.

In contrast, the carbon monoxide (CO) content varied from 23.3% to 34.6% for T_1 pellets, with increasing pyrolysis temperature enhancing the decarbonylation reaction. For T_2 treatment, CO sharply increased from 27.8% to 35.6% across the 300°C to 600°C. These values were consistent with the findings of Kern et al.[76] In rotary kiln pyrolysis of agricultural straw, they observed CO concentration at 34 vol% with a temperature variation from 450°C to 600°C.

The methane concentration (CH_4) decreased between 300°C and 400°C, increasing with further temperature elevation for T_1 pellets. Conversely, for T_2 pellets, the CH_4 variation ranged from 6.9% to 4.2%, with a

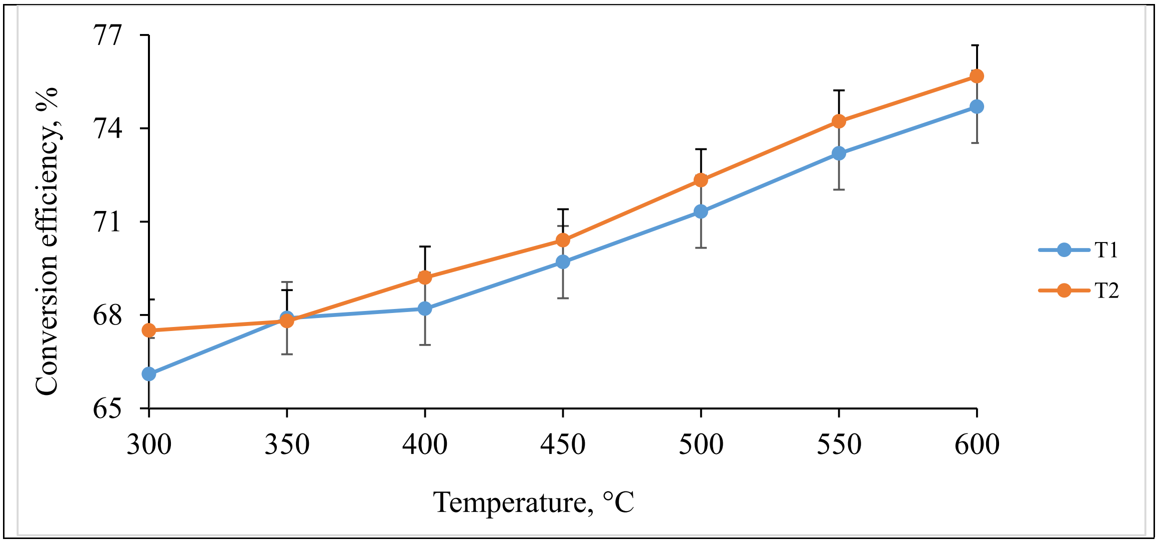


FIGURE 6 Pellets conversion across varying temperatures during pyrolysis.

temperature range of 400°C to 600°C. Other authors[59] confirmed this trend, though the CH_4 concentration (14%–17.7%) in their study was higher, possibly due to different agricultural residues such as pine sawdust. Moreover, the decline in CH_4 yield might be attributed to the reaction of CH4 and oxygen to produce acetylene at higher temperatures.[79]

The hydrogen (H_2) content increased for T_1 from 2.13% at 300°C to 9.36% at 600°C, generated by the cracking and rearrangement of aromatic bonds at higher temperatures (≥500°C).[80] On the other hand, H_2 content decreased between 300°C and 400°C but increased from 400°C to 600°C by 2.77% to 11.36%. These results aligned with the works of Sedmihradská et al.,[36] where a relatively similar amount of H_2 was found.

The content variation in gas composition agrees with the study of Khuenkaeo and Tippayawong,[81] who investigated the pyrolysis product yield from three biomasses: wood, coconut shell, and straw. High temperatures favored H_2 and CO production by dehydrating higher hydrocarbons, even though CO_2 concentration was prominent.

3.6.2 | Relationship between residence time on gas composition

This experiment produced biochar at temperatures of 300, 450, and 600°C. The CO_2 concentration sharply declined from 58.11% to 38.36% for the T_1 pellet, with 5 to 55 min of residence time. Alternatively, the CO_2 concentration dropped from 46.15% to 40.36% for the T_2 pellets within the same residence time. The results were consistent with the findings of Ningbo et al.,[59] even with different biomass and reactors. However, CO increased from 26.6% to 42.4% within 55 min of T_1 treatment. A similar trend was observed for the T_2 pellets, with the concentration increasing from 38.12% to 42.69% over the same duration. This upward trend indicates that a longer residence time allows for secondary cracking, leading to the decomposition of more macromolecular compounds and the release of CO.[82]

The H_2 content gradually increased from 5.9% to 11.87% with prolonged residence time in the case of the T_1 pellet. In the pyrolysis of pellet T_2, the H_2 concentration increased from 4.31% to 9.68% for equal residence time. As observed, CH_4 yield for both treatments had a slightly decreasing trend, with the concentration varying between 3% to 4%, indicating that solid residence time did not significantly influence the production of CH_4. Ningbo et al.[59] This trend was confirmed, although the gas concentration in their study was higher, likely due to differences in feedstock and reactor conditions.

Overall, Figure 8 shows that residence time enhances the generation of H_2 and CO, inversely decreasing the CO_2. The higher partial pressure of CO might favor H_2 production.[59]

3.6.3 | Biochar characterization

Biochar is a carbon-rich material produced by heating at different temperatures[83] This experiment produced biochar at 300, 450, and 600°C. Table 6 shows the basic properties of biochar and pellets.

Typically, biomass contains a certain amount of water or moisture, and the concentration of moisture plays a vital role in biochar generation.[84] The moisture present

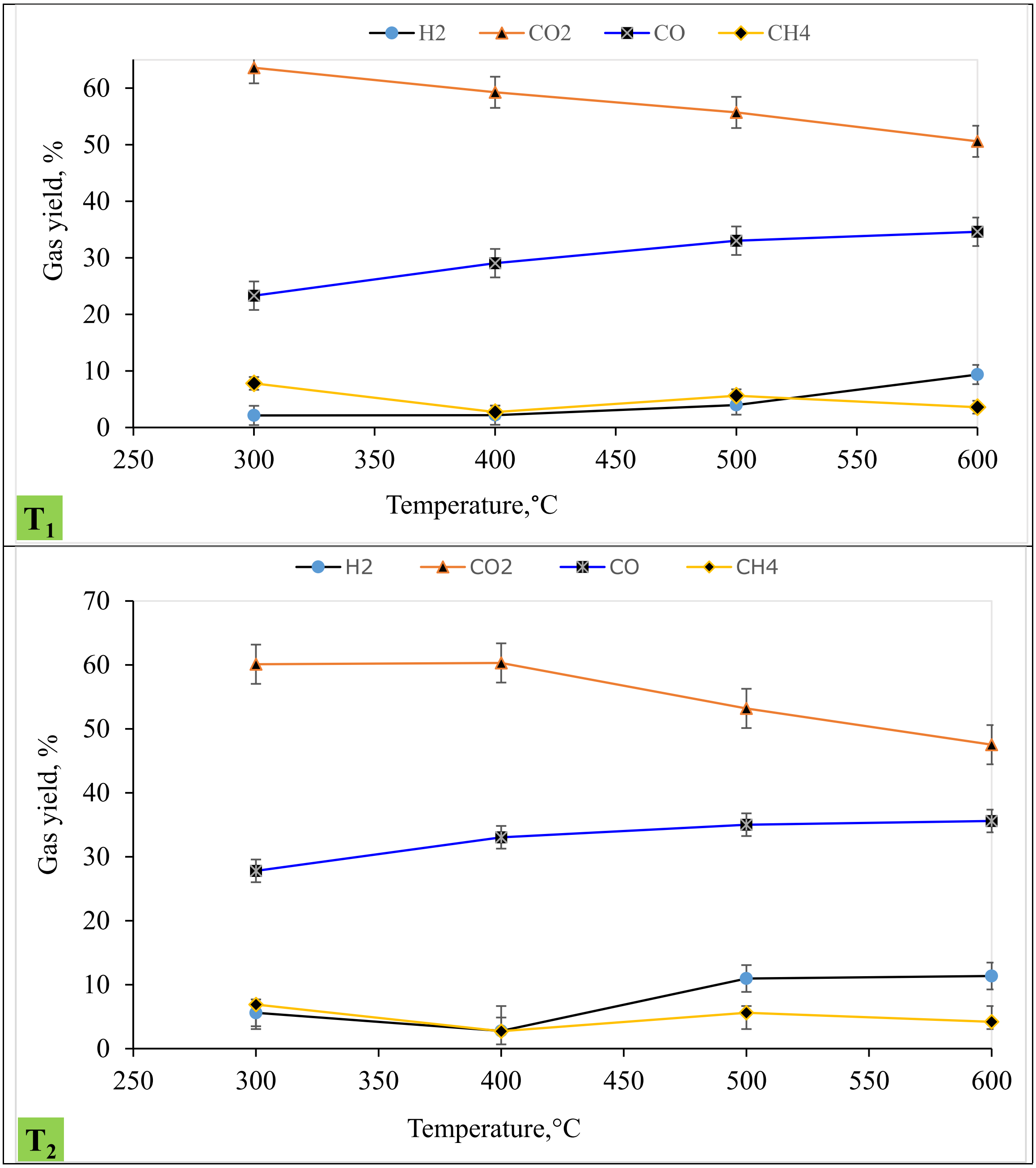


FIGURE 7 Correlation between temperature and gas yield (composition).

in biomass increases the energy required to reach the pyrolysis temperature and prevents char development.[83] The moisture content present in the biochar represents partial burning, and in this study, the moisture content for T_1 and T_2 treatments was 6.2% and 3.5%, respectively. However, the biochar's moisture content was lower than the raw materials, indicating that water evaporated during the heating process[85] (Table 6). Therefore, low moisture biomass is recommended for biochar production since it substantially reduces the energy requirements and time for pyrolysis.[86]

According to reports, the higher lignin concentration in plant biomass encourages carbonization and raises biochar ash and carbon contents.[87] As observed in Table 6, the ash content in the biochar was relatively lower than in the raw pellets. These findings closely align with those reported by Pérez-Maqueda et al.[88] However, Rafiq et al.[89] noted that increased pyrolysis temperature led to an increase of 5.7%–18.7% in ash content. This rise in ash content is attributed to the progressive concentration of inorganic constituents and the combustion residues of organic matter.[90]

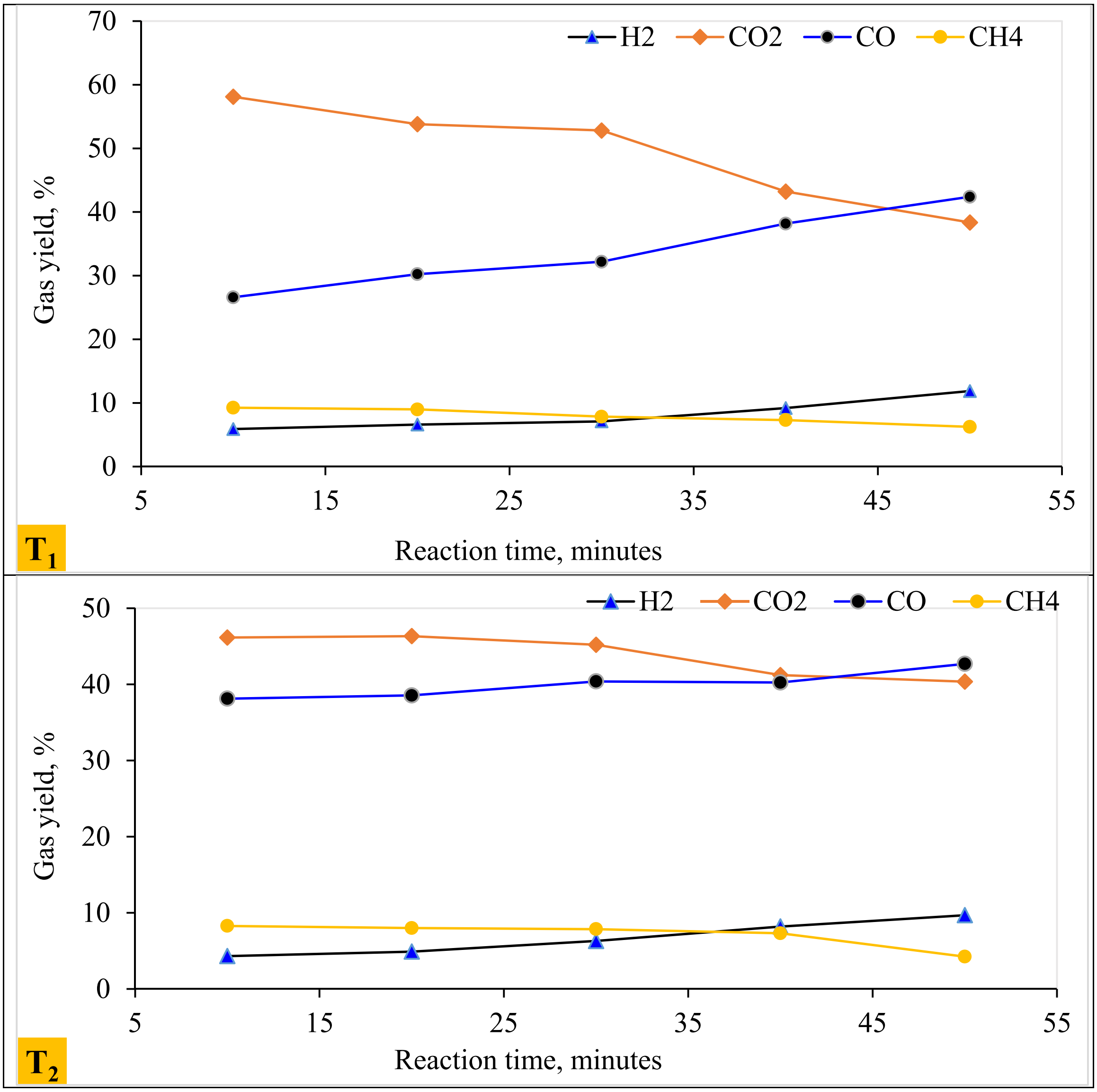


FIGURE 8 Relationship between residence time and gas yield composition.

TABLE 6 Characteristics of the pellet and biochar.

		T_2				T_1			
			Biochar				Biochar		
Property		**Pellets**	**300°C**	**450°C**	**600°C**	**Pellets**	**300°C**	**450°C**	**600°C**
Proximate analysis (% dry basis)	Moisture content	3.5	3.26	2.11	3.03	6.2	3.71	4.28	1.98
	Ash	11.87	11.45	10.39	8.71	7.09	7.02	6.31	5.85
	Volatile matter	53.03	44.51	35.66	32.77	75.61	58.31	45.92	41.39
	Fixed carbon	31.6	40.78	51.84	55.49	11.1	30.96	43.49	50.78
Higher heating value (dry basis, MJ/kg)		19.06	23.06	29.25	28.73	17.02	17.02	25.81	27.07

Note: T_1: 100% wheat straw and T_2: 70% Wheat straw, 10% sawdust, 10% biochar, 10% bentonite clay.

The volatile matter in biochar indicates incomplete thermal degradation during pyrolysis.[91] It can be observed from Table 6 that as the pyrolysis temperature increases, there leads to a decrease in volatile content from 44.51% to 32.77% for T_2 pellets and 75.61% to 42.39% for T_1 treatments. This decline occurs because the increasing temperature causes volatile fractions to continue breaking down into low-molecular-weight liquids and gases rather than forming charcoal.[92] These results are consistent with the findings of Tag et al.[93] Overall, the pyrolysis temperature influenced the biochar structure due to the release of volatiles.[94]

In addition, the volatile matter of biochar decreased, and fixed carbon showed an increase depending on both pyrolysis temperature and biomass type.[95] As the temperature increased from 300°C to 600°C, the fixed carbon content rose from 31.6% to 55.49% for T_1 and 11.1% to 50.78% for T_2 pellets. These might be due to the secondary cracking reaction increasing with the pyrolysis temperature rising, contributing to an increase in the combustible composition of the biomass.[59] In addition, the high carbon content indicates that biochar likely retains some plant residues, such as cellulose and lignin.[96] Therefore, biochar likely retains some plant residues, such as cellulose and lignin of Gabriela and Cora.[97]

The calorific value of biomass is important for the technological design of a pyrolysis plant. A Bomb Calorimeter determined the heating values of T_1 and T_2 samples and biochar. The maximum calorific value of biochar at 600°C was 27.73 MJ/kg for T_2 and 27.07 MJ/kg for T_1 pellets. The higher heating value (HHV) of the WSP biochar increased with the pyrolysis temperature. This increasing trend in HHV is consistent with the literature, such as Ningbo et al.,[59] where the heating value reached 27.54 MJ/kg at 900°C.

Overall, biochar quality, including heating value and fixed carbon, improves with longer residence time and higher temperature.[36] According to the European Biochar Certificate (EBC), the standard should contain more than 50% fixed carbon.[98] In the present study, the biochar from the T_2 pellet had more than 50% fixed carbon at temperatures ranging from 450°C to 600°C, while T_1 showed 50.78% fixed carbon at 600°C, which complies very well with the EBC standards. Therefore, biochar has a higher calorific value under different pyrolysis temperatures than raw materials, indicating the advantageous utilization of biomass through slow pyrolysis.

4 | CONCLUSIONS

In this study, the pyrolysis of two pellets (T_1 and T_2) was conducted in a laboratory-scale oven under an inert nitrogen environment. The primary objective was to gather fundamental insights for establishing a small fixed-bed pyrolysis plant that generates gaseous pyrolysis products, particularly biochar and gas, suitable for on-farm applications.

The findings demonstrated the significant impact of temperature and reaction time on total pyrolysis yields and gas composition. The distinct characteristics of the two pellet types, T_1 and T_2, led to variations in production depending on time and temperature. The optimal temperature for biochar production was identified as 300°C, while the maximum temperature for gas production was 600°C.

Importantly, the biochar produced under these temperature conditions met the European Biochar Certificate (EBC) standards, which require a carbon content exceeding 50% by mass. These results suggest that wheat straw pellets have considerable potential as a promising agricultural crop residue for bioenergy production. The insights from this study can serve as a foundation for developing small-scale pyrolysis facilities for on-farm use.

Overall, the study successfully analyzed syngas yield and other products from wheat straw pellet pyrolysis. A key limitation was the lack of in-depth investigation into ash and bio-oil, which prevented a complete understanding of the process. Therefore, further research is recommended, focusing on more experimentation to analyze the properties and potential applications of ash and bio-oil derived from wheat straw pyrolysis.

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DATA AVAILABILITY STATEMENT

The data presented in this study are available on request from the corresponding author.

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