

Article

Influence of Reaction Temperature and Heating Rate on the Pyrolysis Products of Rice Husk

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Abstract

Rice cultivation generates a substantial amount of rice husks (RH) as agricultural waste from rice milling, which is not effectively utilized for environmentally friendly products. This study investigates the product yields and chemical characteristics of RH pyrolysis conducted at temperatures between 400 and 600 °C, at 50 °C intervals, with heating rates of 5, 10, and 20 °C/min. The highest liquid product yield (30.57%) was achieved at 600 °C under a heating rate of 20 °C/min. Gas chromatography–mass spectrometry (GC–MS) characterization revealed that the liquid product consisted predominantly of complex mixtures, with C₆ compounds dominating the carbon number distribution, followed by C₈ and C₅ compounds. During the pyrolysis process, CO₂ and CO were the main components of the non-condensable gases, whereas total hydrocarbons (THC) were generated at pyrolysis temperatures of 500 °C and above. The carbon content of biochar increased as the pyrolysis temperature increased, while the oxygen and hydrogen contents decreased. These findings elucidate the influence of temperature and heating rate on the chemical properties of pyrolysis products derived from RH. In addition, the kinetic analysis of RH pyrolysis showed that the estimated activation energy varies depending on the applied method. Therefore, the combined use of multiple methods is considered desirable for providing a more reliable kinetic interpretation of RH pyrolysis.

Keywords: rice husk; pyrolysis; product yield; chemical characterization; kinetic analysis

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1. Introduction

Rapid industrialization and global population growth have led to a substantially increased energy demand, thereby intensifying the global energy crisis. Moreover, the continued reliance on fossil fuels has resulted in rising greenhouse gas emissions and severe environmental impacts [1]. It is projected that worldwide energy demand will reach approximately 17,487 million tons of oil equivalent (Mtoe) by 2040, growing at a yearly rate of 1.1% from 2015–2040 [2]. In 2022, energy-related carbon dioxide (CO₂) emissions exceeded 36.8 gigatons (Gt), representing a 0.9% rise over the previous year. It was reported that CO₂ emissions from energy combustion increased by approximately 423 million metric tons, or above 1% [3]. The large amount of greenhouse gases, especially CO₂ and methane (CH₄), generated from the use of fossil fuels for energy and power generation, is the

foremost contributor to air pollution. Such emissions have given rise to major environmental issues, among them climate change and global warming [4].

To achieve net-zero emissions by 2050, renewable energy is considered an effective way to reduce CO₂ and CH₄ emissions and keep global warming within 1.5 °C [5]. Among renewable energy sources, biomass is considered a significant alternative for replacing fossil fuels while mitigating greenhouse gas emissions, thereby contributing to sustainable development [6,7]. Specifically, rice husks (RH) are one of the most abundant and promising biomass resources, especially in rice-growing countries, owing to their high availability and energy potential. It is estimated that global rice cultivation covered around 165 million hectares in 2022 [8]. Rice farming generates significant agricultural waste in many countries, including rice husks, which are byproducts of the milling process that removes the husk from the grain [9,10]. RH are highly abundant agricultural residues, as rice is a primary food source for billions of people [11]. Each year, rice production yields more than 750 million tons of grain, together with approximately 150 million tons of RH [12]. RH accounts for roughly 20% of the total weight of rice grains after the milling process [13–15].

Across many developing Asian countries, agricultural residues are frequently disposed of through open burning or landfilling, emitting CO₂ and other greenhouse gases that contribute to environmental problems and necessitate compliance with global environmental regulations [16]. In RH, cellulose and hemicellulose are each more abundant than lignin, with approximate content ranges of 28.6–41.5%, 14.0–28.65%, and 20.4–33.7%, respectively [14]. However, RH is unique not only as a renewable resource but also because of its abundant silica content, which makes it suitable for manufacturing construction materials while reducing dependence on natural resources in the construction industry [17]. Pyrolysis offers a simple, efficient, and cost-effective approach for the thermal conversion of biomass and can accommodate a wide range of biomass feedstocks [18]. During pyrolysis, lignocellulosic biomass undergoes thermal decomposition in the absence of oxygen, producing crude bio-oil, biochar, and non-condensable gases [19,20]. The resulting products have broad applications, such as turbines, combustion systems, boilers, and power generation [21,22]. Additionally, fuels derived from pyrolysis are considered more environmentally friendly since they release lower emissions of CO₂, SO_x, and NO_x than conventional fossil fuels [23]. Nevertheless, this process can affect product yield through several factors, including heating rate, temperature, feedstock type, and reactor design. Pyrolysis can be carried out using a variety of systems and reactor designs. According to [24], a fixed-bed reactor is a particularly suitable choice for small-scale power applications, as it offers a simple and practical route to producing fuels. Existing studies have investigated the thermochemical conversion of RH through pyrolysis under various operating conditions. Ji-lu [25] evaluated the characteristics of bio-oil produced from RH fast pyrolysis in a fluidized-bed reactor under a nitrogen atmosphere, with emphasis on the effects of reaction temperature. Tsai et al. [26] investigated the effects of operating conditions on product yield distribution and the chemical characterization of bio-oil in a fixed-bed reactor. This study reported a maximum bio-oil yield of approximately 40% at 500 °C when the heating rate exceeded 200 °C/min. Heo et al. [27] also investigated RH pyrolysis in a fluidized-bed reactor and demonstrated that reaction temperature strongly affected the properties of bio-oil. In another study, Weldekidan et al. [28] characterized the properties of pyrolysis gas, bio-oil, and biochar using solar radiation at a heating rate of 160 °C/min. Their results indicated that the high oxygen content and acidic properties of bio-oil require further upgrading, such as hydrothermal or catalytic treatment, before being used as transportation fuels.

However, numerous studies have mainly focused on individual aspects, such as the yields of specific products, liquid product properties, and biochar characteristics. Com-

prehensive evaluations of the pyrolysis characteristics of RH, including the effects of pyrolysis temperature and heating rate in a fixed-bed reactor on the yields of all pyrolysis products (gas, biochar, and liquid products) and the characteristics of each product, as well as the kinetic behavior of the pyrolysis reaction, remain limited. Moreover, comprehensive studies on the activation energy of RH pyrolysis using differential and integral methods are still limited in the literature. Therefore, the main objectives of this study were to investigate the product yields and chemical characteristics of RH pyrolysis conducted at various temperatures and heating rates using a range of analytical tools. In addition, thermogravimetric and kinetic analyses were performed to investigate the decomposition behavior of RH during pyrolysis, providing fundamental data for kinetic interpretation.

2. Materials and Methods

2.1. Materials

RH particles were used as the raw material for pyrolysis in this study. The RH was sieved to obtain particle sizes in the range of 0.60–1.70 mm. Moisture content was reduced to below 1% by drying the sieved material in an oven at 105 °C for 24 h.

2.2. Experimental Procedure

The pyrolysis of RH was conducted in a fixed-bed reactor consisting of a quartz tube (550 mm in length and 60 mm in internal diameter), placed in an electric furnace with a heating zone of 450 mm. The temperature and heating rate were controlled by a PID controller. A two-stage condenser employing cooling water and a refrigerant was installed at the outlet of the reactor to collect liquid products, while non-condensable gases were discharged. The experimental apparatus is illustrated schematically in Figure 1, as reported previously [29]. In each run, pyrolysis experiments were repeated three times under the same conditions to ensure accuracy and minimize errors in experimental results. Raw RH samples of 15 g were placed in ceramic boats and loaded at the center of the reactor. Nitrogen was supplied at a flow rate of 4 L/min to remove residual oxygen from the reactor. The reactor was then heated to the pyrolysis temperature at 400, 450, 500, 550, and 600 °C with three different heating rates of 5, 10, and 20 °C/min. Based on the TGA analysis in Section 3.4, the pyrolysis experiments were conducted over a temperature range of 400–600 °C at 50 °C intervals, where most of the pyrolysis-induced mass loss occurred, and nearly complete decomposition was achieved. The heating rates were set at 5, 10, and 20 °C/min due to the operational limitations of the reactor. After reaching the target temperature, the reactor was maintained at this temperature for an additional 30 min to ensure complete pyrolysis. Liquid oils were recovered in the condenser during the pyrolysis process. In this study, the liquid products were classified into low-viscosity liquid product (LVLP) and high-viscosity liquid product (HVLP) based on their flowability. LVLP, which exhibited sufficient flowability, was directly recovered from the condenser installed in the cooler (No. 10 in Figure 1). In contrast, HVLP, which had very limited flowability and existed in the form of wax at the outlet section exposed to room temperature (No. 9 in Figure 1) of the reactor (No. 5 in Figure 1), was recovered after the completion of the reaction by removing the reactor and separately heating the reactor until the product became sufficiently fluid for collection. Upon completion of the pyrolysis, the reactor was allowed to cool before the biochar was removed from the ceramic boats. All the product yields (HVLP, LVLP, and biochar) were weighed, recorded, and stored for further analysis.

The yields of each product were calculated using the following equations:

$$\text{Biochar yield, (\%)} = \frac{\text{Mass of bio char (g)}}{\text{Mass of raw RH (g)}} \times 100 \quad (1)$$

$$\text{Liquid product yield, (\%)} = \frac{\text{Mass of liquid product (g)}}{\text{Mass of raw RH (g)}} \times 100 \quad (2)$$

$$\text{Gas yield, (\%)} = 100 - (\text{Y, biochar} + \text{Y, liquid product}) \quad (3)$$

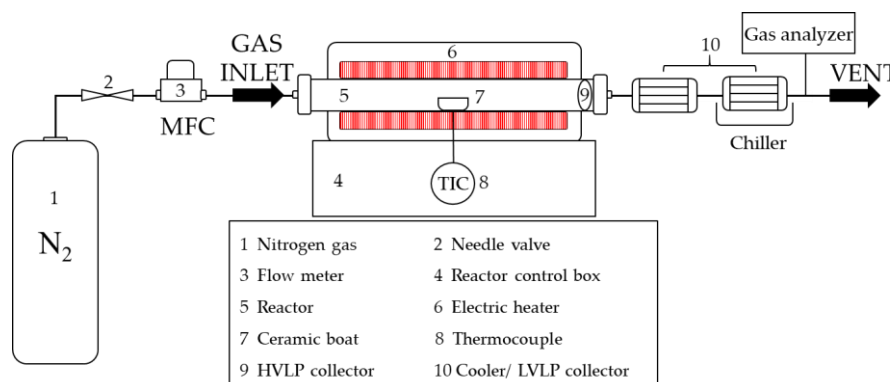


Figure 1. Schematic illustration of the laboratory-scale fixed-bed reactor.

2.3. Analytical Methods

2.3.1. Proximate and Ultimate Analyses

The percentages of volatile matter (VM), fixed carbon (FC), and ash content (AC) in the raw RH material were determined via proximate analysis according to ASTM D3174-89 [30] and ASTM D3175-89 [31], following the procedure described in [32]. Ultimate analysis was conducted to determine the elemental composition, including carbon (C), hydrogen (H), nitrogen (N), and oxygen (O), of raw RH and derived biochar using an elemental analyzer (Flash 2000, Thermo Fisher Scientific, Oxford, UK).

2.3.2. Gas Product Analysis

Non-condensable gases were analyzed using a gas analyzer (ECOM-MK 6000+, Ecom GmbH, Iserlohn, Germany) to measure the concentrations of CO, CO₂, and total hydrocarbons (THC) generated during RH pyrolysis. The gas analyzer was connected to the outlet of the condenser, as shown in Figure 1. The experiments were conducted at 500, 400, and 600 °C with a heating rate of 10 °C/min. Additional experiments at 500 °C were performed using heating rates of 5 and 20 °C/min. CO₂ and total hydrocarbons (THC) were measured using a non-dispersive infrared (NDIR) sensor, and CO concentrations were analyzed using an electrochemical (EC) sensor.

2.3.3. Liquid Product Analysis

The liquid products derived from RH pyrolysis were analyzed using a gas chromatography–mass spectrometer (GC–MS) (QP2010 Ultra, SHIMADZU, Kyoto, Japan) to determine their chemical composition. GC–MS analysis was performed using an SH-Rxi-1ms capillary column (30 m × 0.25 mm i.d., 0.25 µm film thickness). Helium was used as the carrier gas at a column flow rate of 1.15 mL/min. The injector temperature was set at 250 °C with a split ratio of 500:1. The column oven was initially held at 40 °C for 3 min before being heated to 300 °C at 10 °C/min, followed by a final hold of 10 min.

2.3.4. Biochar Analysis

The biochar produced from RH pyrolysis was characterized using X-ray fluorescence (XRF), Fourier-transform infrared (FTIR) spectroscopy, and X-ray diffraction (XRD). The oxide compositions were determined using a wavelength-dispersive X-ray fluorescence (WD-XRF) spectrometer (ZSX Primus, Rigaku, Tokyo, Japan) operated at an acceleration

voltage of 50 kV. FTIR analysis was performed to investigate changes in the chemical structures of raw RH and resulting biochar obtained at different temperatures and a heating rate of 10 °C/min. For this purpose, an FTIR spectrometer (Spectrum 100, PerkinElmer, Beaconsfield, UK) equipped with attenuated total reflectance (ATR) was used to collect spectra over the range of 4000–500 cm^{−1} with a resolution of 2 cm^{−1}. XRD analysis was performed to investigate changes in the crystalline structures of raw RH and biochar obtained from pyrolysis. An XRD instrument (MiniFlex 600, Rigaku, Tokyo, Japan) equipped with a D/teX Ultra detector was used, with measurements conducted over a 2θ range of 5–80° at a scan rate of 3°/min using Cu Kα radiation.

2.4. Thermogravimetric and Kinetic Analysis

2.4.1. Thermogravimetric Analysis

Thermogravimetric analysis (TGA) was performed to evaluate the thermal decomposition behavior and mass loss of RH samples using a thermogravimetric analyzer (NETZSCH TG 209 F1 Libra, NETZSCH-Gerätebau GmbH, Selb, Germany). The samples were heated from ambient temperature (30 °C) to 800 °C at heating rates of 5, 10, 20, 30, and 40 °C/min under an inert N₂ atmosphere.

2.4.2. Kinetic Analysis

Kinetic analysis was conducted to determine the activation energy associated with thermal decomposition. The activation energy values were calculated using the integral and the differential method based on TGA data, which have been widely applied in kinetic studies of pyrolysis reactions [33]. In general, the reaction rate of thermal decomposition is expressed by the Arrhenius equation (Equation (4)), which has been widely applied in kinetic analyses of biomass pyrolysis [34,35]:

$$\frac{d\alpha}{dt} = A e^{-\frac{E}{RT}} (1 - \alpha)^n \quad (4)$$

where A is the pre-exponential factor (min^{−1}), E is the activation energy (kJ/mol), n is the reaction order, R is the gas constant (0.008314 kJ/mol·K), T is the reaction temperature (K), and α is the degree of conversion. The degree of conversion in Equation (4) is calculated from the weight loss data obtained from TGA as follows:

$$\alpha = \frac{m_o - m_t}{m_o - m_f} \quad (5)$$

where m_o is the initial mass of the sample, m_t is the mass at a given time, and m_f is the final mass. By introducing the constant heating rate β (K/min) into Equation (4), it can be rearranged to yield Equation (6):

$$\frac{d\alpha}{(1 - \alpha)^n} = \frac{A}{\beta} e^{-\frac{E}{RT}} dT \quad (6)$$

Applying an integral approximation to Equation (6) leads to Equations (7) and (8), which are known as the Coats–Redfern method [36]:

$$\ln \left\{ \frac{1 - (1 - \alpha)^{1-n}}{T^2(1-n)} \right\} = \ln \left\{ \frac{AR}{\beta E} \left(1 - \frac{2RT}{E} \right) \right\} - \frac{E}{RT} \quad n \neq 1 \quad (7)$$

$$\ln \left\{ \frac{-\ln(1 - \alpha)^{1-n}}{T^2} \right\} = \ln \left\{ \frac{AR}{\beta E} \left(1 - \frac{2RT}{E} \right) \right\} - \frac{E}{RT} \quad n = 1 \quad (8)$$

Therefore, the activation energy values can be determined by assuming the reaction order and calculating the slopes obtained from the linear plots of Equations (9) and (10):

$$- \ln \left\{ \frac{1 - (1 - \alpha)^{1-n}}{T^2(1-n)} \right\} \text{ vs } \frac{1}{T} \quad n \neq 1 \quad (9)$$

$$- \ln \left\{ - \frac{\ln(1 - \alpha)}{T^2} \right\} \text{ vs } \frac{1}{T} \quad n = 1 \quad (10)$$

Also, the activation energy can be obtained from the reaction rate expression derived from the differential form of Equation (4). That is, taking the logarithm of both sides of Equation (4) yields Equation (11). Accordingly, the activation energy can be determined from the slope by plotting $\ln(d\alpha/dt)$ versus $1/T$ at each conversion. This method corresponds to the Friedman method.

$$\ln\left(\frac{d\alpha}{dt}\right) = \ln\{A(1-\alpha)^n\} - \frac{E}{RT} \quad (11)$$

3. Results and Discussion

3.1. Characteristics of Raw RH

Table 1 presents the ultimate and proximate analyses of RH used in this study. RH was composed of carbon (C, 37.24%), oxygen (O, 31.23%), hydrogen (H, 4.91%), and nitrogen (N, 0.67%), with the remaining 25.92% consisting of other elements. As shown in Table 1, the nitrogen content was low. Nitrogen contained in the fuel typically enhances the formation of nitrogen oxides (NO_x), which are environmental pollutants, during combustion; therefore, a low nitrogen content is considered desirable for cleaner combustion [37,38]. In addition, the raw RH exhibited volatile matter (VM), fixed carbon (FC), and ash content (AC) of 57.30%, 21.63%, and 21.06%, respectively. Feedstocks that contain high volatile matter are more appropriate for biofuel production, and a high percentage of fixed carbon can be considered as activated carbon processing [39]. Table 2 presents the eight elemental and oxide components identified in the raw RH by XRF analysis. The other constituents in Table 1 are mainly known as ash-forming materials, such as silicon, aluminum, potassium, calcium, phosphorus, and other heavy metals [40]. The major element was silicon (Si) at approximately 68.02%, followed by potassium (K) at 13.05%, which is consistent with the previous study [41]. The chemical composition of RH is mainly attributed to several factors, including climate, paddy type, soil chemistry, type of fertilizer, and sample preparation [42]. The ash and Si contents determined in the present study closely matched the values reported in earlier studies [43], in which RH exhibited ash contents of approximately 19–24% and silica concentrations in the range of 68–91%.

Table 1. Proximate and ultimate analysis of raw RH.

Proximate analysis (%)	VM	57.30
	FC	21.63
	AC	21.06
Ultimate analysis (wt.%)	N	0.67
	C	37.24
	H	4.91
	O	31.23
	Others	25.92

Table 2. Chemical composition of raw RH obtained via XRF analysis.

Elemental components (wt.%)	Si	Al	P	S	K	Ca	Mn	Fe
	68.02	0.31	5.12	2.18	13.05	8.95	1.13	1.20
Oxide components (wt.%)	SiO ₂	Al ₂ O ₃	P ₂ O ₅	SO ₃	K ₂ O	CaO	MnO	Fe ₂ O ₃
	81.81	0.33	4.71	2.12	5.79	4.17	0.45	0.52

3.2. Product Yields from RH Pyrolysis

Figure 2 and Table 3 show the product yields of RH pyrolysis at various operating temperatures and heating rates. For each heating rate, as the pyrolysis temperature increased, the biochar yield decreased, whereas the gas and liquid product yields tended to

increase. The heating rate had a more pronounced influence on the liquid product yield. At 5 °C/min, HVLP yield remained higher than that of LVLP; however, at 10 °C/min, similar yields were obtained. As the heating rate increased to 20 °C/min, the LVLP yield became higher than that of HVLP. According to Akhtar and Saidina [44], increasing the heating rate alleviates heat and mass transfer limitations while shortening the duration for secondary reactions, including tar cracking and repolymerization. Consequently, the higher LVLP yield observed at elevated heating rates is likely attributable to the suppression of LVLP repolymerization, thereby limiting HVLP formation. This finding is comparable to the results obtained in our earlier investigation of EFB pyrolysis [45]. Biochar yields tended to decrease with increasing temperature and heating rate, possibly due to the primary decomposition of the feedstock and secondary decomposition of biochar [46,47]. In addition, higher biochar yields were obtained at lower temperatures because the available thermal energy was insufficient to decompose the organic material. In contrast, higher temperatures delivered sufficient energy to enhance cleavage of chemical bonds within the material [48]. As reported by Di Blasi et al. [49], in the pyrolysis of RH and other agricultural residues, the biochar yield decreased with increasing temperature. The high biochar yield was associated with its elevated carbon content and the presence of inorganic components. The gas yield was more strongly influenced by pyrolysis temperature than by heating rate. The increase in gas yield with increasing temperature is associated with more extensive secondary decomposition of pyrolysis products [50].

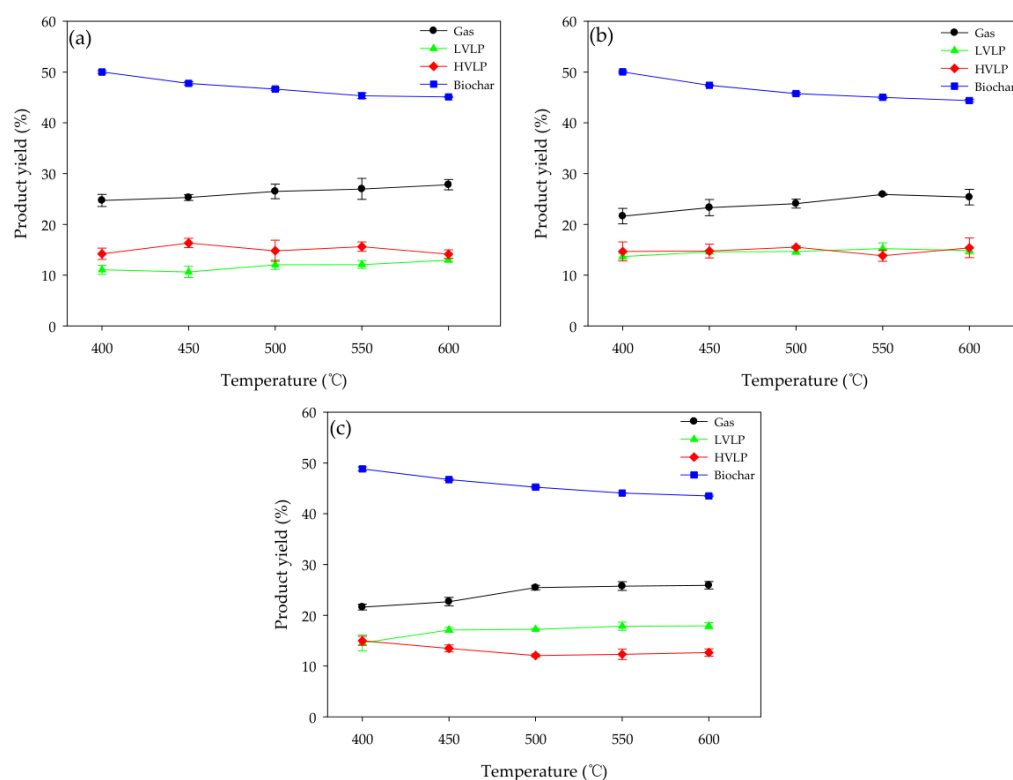


Figure 2. Product yields of RH pyrolysis at heating rates of (a) 5, (b) 10, and (c) 20 °C/min.

Table 3. Product yields from RH pyrolysis at different temperatures and heating rates.

Heating Rate (°C/min)	Temperature (°C)	Gas (%)	LVLP (%)	HVLP (%)	Biochar (%)
5	400	24.72 ± 1.19	11.07 ± 0.89	14.20 ± 1.11	50.01 ± 0.27
	450	25.29 ± 0.53	10.62 ± 1.11	16.33 ± 0.92	47.76 ± 0.30
	500	26.49 ± 1.42	12.05 ± 0.93	14.80 ± 2.08	46.66 ± 0.27

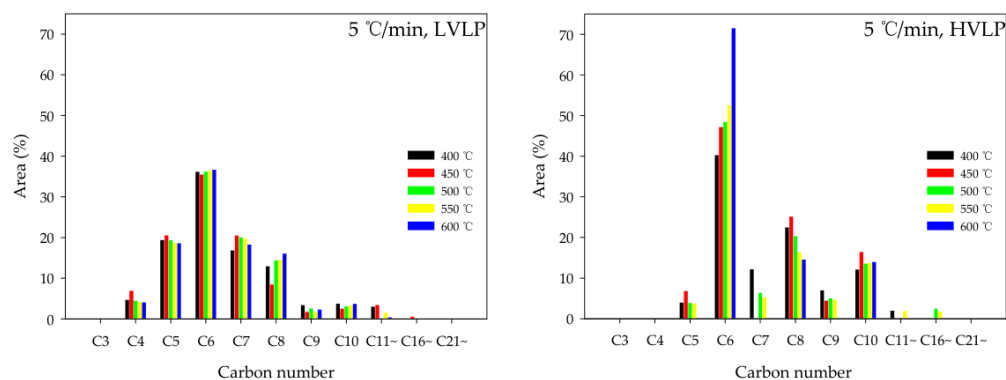
10	550	26.98 ± 2.06	12.08 ± 0.77	15.61 ± 0.93	45.34 ± 0.57
	600	27.81 ± 1.01	12.95 ± 0.33	14.13 ± 0.85	45.11 ± 0.10
	400	21.62 ± 1.52	13.67 ± 0.58	14.67 ± 1.86	50.03 ± 0.18
	450	23.31 ± 1.59	14.56 ± 0.35	14.75 ± 1.36	47.38 ± 0.11
	500	24.07 ± 0.86	14.66 ± 0.54	15.51 ± 0.21	45.76 ± 0.24
	550	25.92 ± 0.17	15.24 ± 1.12	13.85 ± 1.11	45.02 ± 0.23
20	600	25.36 ± 1.53	14.85 ± 0.67	15.38 ± 1.94	44.40 ± 0.20
	400	21.63 ± 0.57	14.56 ± 1.57	14.98 ± 0.86	48.84 ± 0.41
	450	22.68 ± 0.83	17.12 ± 0.47	13.48 ± 0.68	46.72 ± 0.19
	500	25.44 ± 0.47	17.24 ± 0.13	12.07 ± 0.29	45.25 ± 0.17
	550	25.75 ± 0.86	17.85 ± 0.79	12.32 ± 1.02	44.09 ± 0.02
	600	25.91 ± 0.73	17.91 ± 0.62	12.66 ± 0.72	43.51 ± 0.15

3.3. Product Analysis

To minimize analytical uncertainty associated with the limited quantity of the products, each product analysis was performed using combined samples obtained from three replicate experiments.

3.3.1. Characterization of Liquid Products

Figure 3 presents the carbon number distribution of the chemical compounds detected via GC–MS in liquid products from RH pyrolysis. The results showed that the liquid products contained approximately 5–50 compounds with carbon numbers ranging from C3 to C21, with the highest proportion at C6, followed by C5, C7, and C8. Based on carbon number, C6 compounds mainly consisted of anhydrosugars, including 1,4:3,6-Di-anhydro- α -D-glucopyranose and 1,6-anhydro- β -D-glucopyranose, as well as phenolic compounds such as 1,2-benzenediol. Anhydrosugars are produced from cellulose decomposition, whereas phenolic compounds are derived from the decomposition of lignin [51,52]. Therefore, the increase in C6 compounds in HVLP with increasing reaction temperature is considered to be attributable to the enhanced thermal decomposition of lignin components at higher temperatures, resulting in increased formation of phenolic compounds. The results indicated that heating rate and temperature had limited effects on the carbon number distribution of LVLP, while HVLP showed a tendency for the proportion of C6 compounds to vary with the heating rate. As mentioned in Section 3.2, this phenomenon is considered to be associated with the suppression of LVLP repolymerization at higher heating rates, which consequently limits the formation of HVLP. These observations suggest that secondary reactions, including LVLP repolymerization, may influence the formation of C6 compounds in HVLP.



(a)

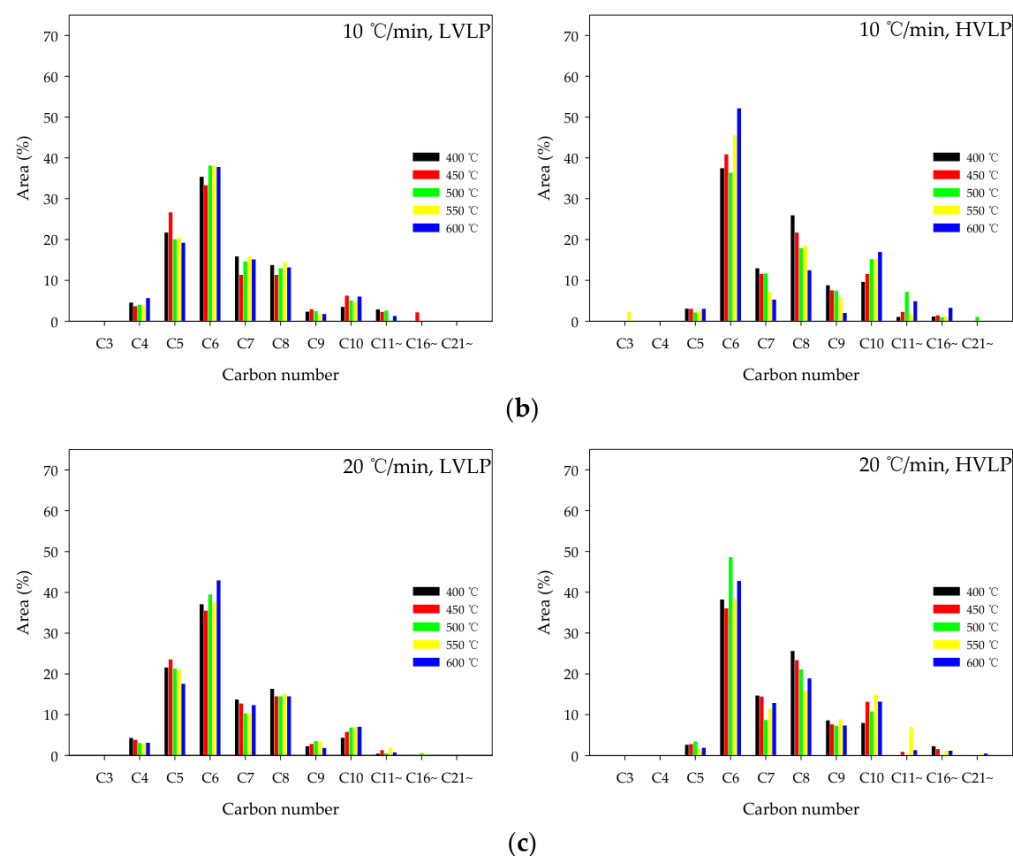


Figure 3. Carbon number distribution of compounds in liquid products from RH pyrolysis at different temperatures and heating rates: (a) 5, (b) 10, and (c) 20 °C/min.

For HVLP at 600 °C, the proportion of C6 reached 71.47% at a heating rate of 5 °C/min and decreased to 52.13% and 42.79% at heating rates of 10 and 20 °C/min, respectively, as the heating rate increased. In particular, 1,6-anhydro-beta-D-glucopyranose, the most abundant C6 compound, decreased from 37.49% to 21.15% and 12.25% as the heating rate increased from 5 to 10 and 20 °C/min.

Figure 4 shows the characteristics of liquid products analyzed into six compound classes, namely saturated, unsaturated, aliphatic, aromatic, oxygenated, and non-oxygenated compounds. The composition of compounds in the LVLP remained nearly unchanged under varying temperature and heating rate conditions. Conversely, HVLP exhibited a high proportion of aromatic and unsaturated compounds at 400 °C, which slightly decreased with increasing temperature. This behavior is attributed to the primary decomposition of lignin, producing aromatic compounds such as 2-methoxyphenol and 2,6-dimethoxyphenol with unsaturated side chains. As the pyrolysis temperature increases, these structures are known to be progressively converted into saturated forms [53]. Accordingly, the fractions of saturated and aliphatic compounds increased with increasing temperature. Moreover, the high oxygen content in the liquid product from RH pyrolysis was attributed to the oxygen naturally present in the cellulose and hemicellulose of RH [28]. The high abundance of oxygenated compounds detected in this study is consistent with previous studies on RH fast pyrolysis, which also reported that the liquid product was dominated by oxygenated compounds [26]. Therefore, upgrading the liquid product to a usable transportation fuel through deoxygenation and dehydration is widely recommended [54].

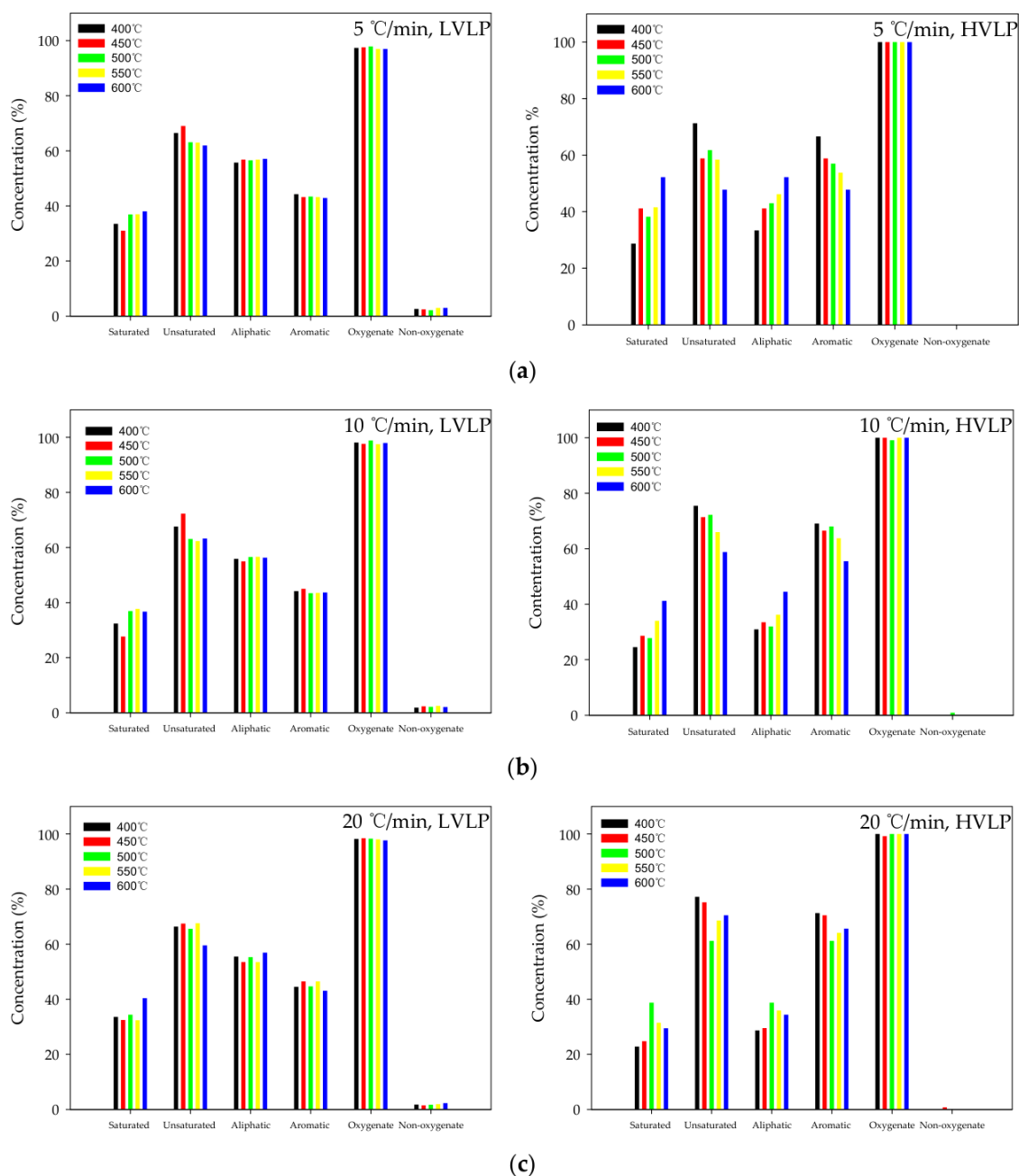


Figure 4. Distribution of compounds in liquid product from RH pyrolysis at different temperatures and heating rates: (a) 5, (b) 10, and (c) 20 °C/min.

3.3.2. Characterization of Gas Products

As presented in Figure 5, the emission characteristics of CO, CO₂, and THC generated during the heating process up to reaction temperatures of 400, 500, and 600 °C at a heating rate of 10 °C/min. The gas fraction was dominated by CO₂, followed by CO, with a minor amount of THC depending on the pyrolysis temperature. According to the literature [55–57] CO is mainly produced through decarbonylation of carbonyl-containing structures and decarboxylation of carboxyl-containing compounds, whereas CO₂ is primarily generated from decarboxylation of cellulose and hemicellulose. THC was mainly present as CH₄, formed through demethylation of lignin [58].

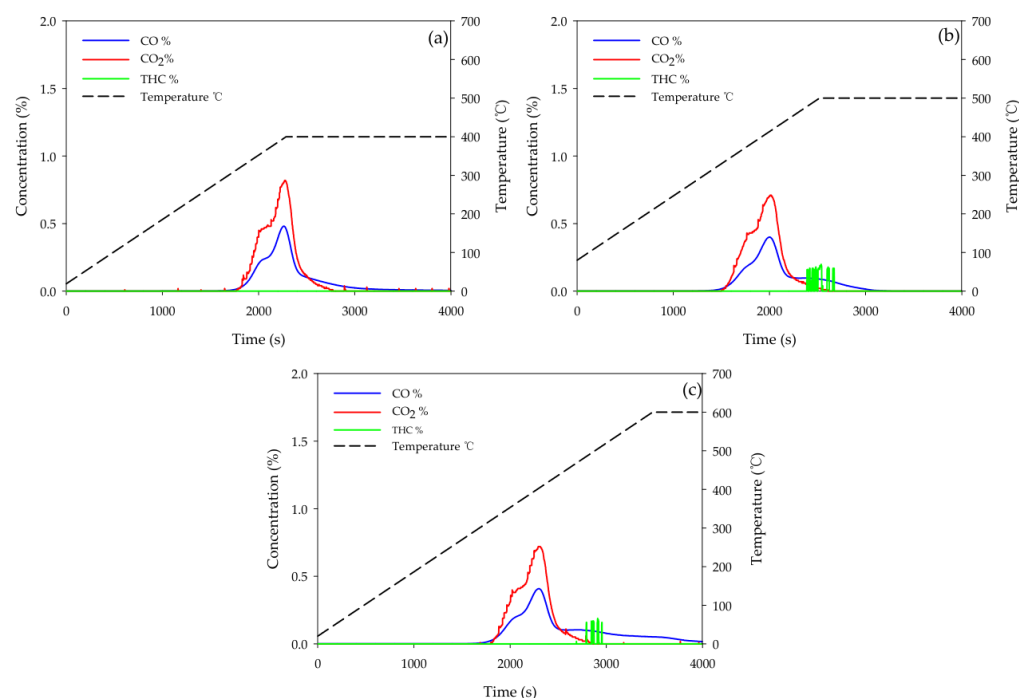
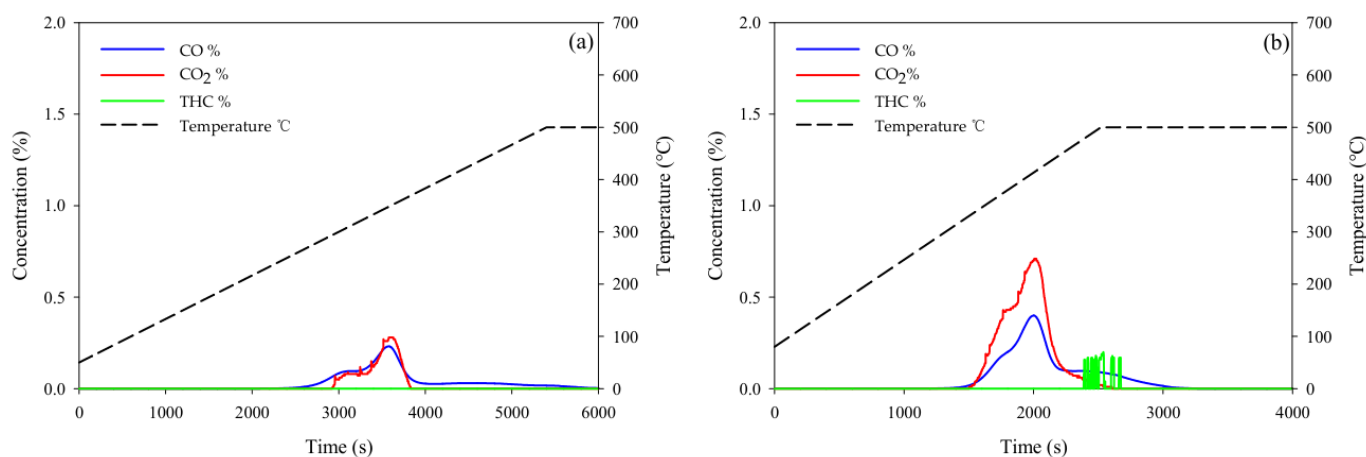


Figure 5. Gas composition from RH pyrolysis at a heating rate of 10 °C/min under pyrolysis temperatures of (a) 400, (b) 500, and (c) 600 °C.

The concentration peaks of CO and CO₂ occurred at approximately 300 °C. Compared with CH₄, CO and CO₂ are evolved at lower temperatures, mainly due to the degradation of hemicellulose and cellulose [49]. In contrast, CH₄ is released at temperatures above 400 °C as a result of the decomposition of lignin involving methyl radicals [59,60]. However, additional analyses of H₂ and various light hydrocarbons are required to achieve a more in-depth understanding of the gas formation mechanisms during RH pyrolysis.

Figure 6 shows the changes in gas products with varying heating rates at a reaction temperature of 500 °C. The results indicate that the concentration of each gas component increased with increasing heating rate because a higher temperature was reached more rapidly at higher heating rates.



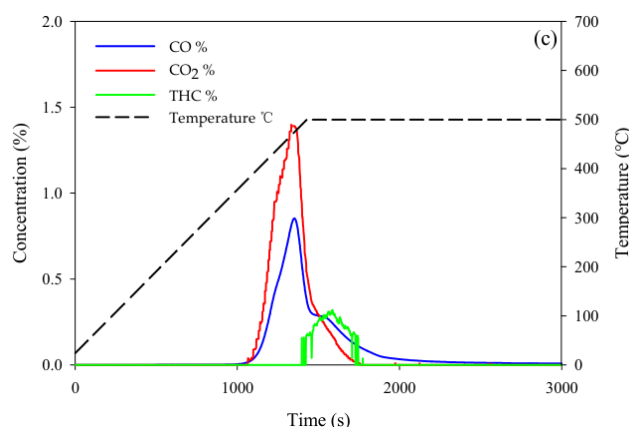


Figure 6. Gas composition from RH pyrolysis at a temperature of 500 °C under heating rates of (a) 5, (b) 10, and (c) 20 °C/min.

3.3.3. Characterization of Biochar

Table 4 presents the chemical composition of biochar at various temperatures and heating rates determined via XRF analysis. The high SiO₂ content is attributed to the inherently high Si content in the raw RH [61]. The concentration of SiO₂ at 600 °C with a heating rate of 20 °C/min reached approximately 90.97%, which is consistent with previous studies reporting that the SiO₂ content of RH biochar increases with increasing pyrolysis temperatures and reaches its maximum value at 550 °C [62]. The high SiO₂ content makes it a suitable component for building materials [63] and ceramic products used in membrane filters for water treatment [64]. Moreover, silicon-rich biochar obtained from the pyrolysis of agricultural waste can be utilized as a soil amendment due to its potential to promote plant growth, root development, and protection against pests and diseases [65]. However, minor compounds such as P₂O₅, SO₃, K₂O, and CaO decreased with increasing temperature. The K₂O content ranged from 3.39% to 4.82%, depending on the types and amounts of fertilizers used during rice cultivation [63]. Additionally, no significant changes were observed in trace amounts of Al₂O₃, MnO, NiO, and ZnO, as their concentrations were below 1%, indicating that these minerals were unaffected by the pyrolysis temperature and heating rate.

Table 4. Chemical composition of biochar at various temperatures and heating rates.

Oxide Composition (wt.%)												
	SiO ₂	Al ₂ O ₃	P ₂ O ₅	SO ₃	K ₂ O	CaO	MnO	Fe ₂ O ₃	Cr ₂ O ₃	MgO	ZnO	NiO
Heating rate of 5 °C/min												
400 °C	89.33	–	2.12	0.28	4.11	3.16	0.29	0.38	0.29	–	–	–
450 °C	89.77	0.17	1.81	0.29	3.66	2.93	0.20	0.71	0.41	–	–	–
500 °C	89.95	–	1.71	0.27	4.01	3.24	0.28	0.12	–	0.38	–	–
550 °C	90.99	–	1.73	0.25	3.57	2.86	0.26	0.08	–	0.23	–	–
600 °C	90.20	–	1.84	0.27	3.83	3.27	0.29	–	–	0.23	0.02	–
Heating rate of 10 °C/min												
400 °C	88.93	–	1.96	0.35	4.58	3.49	0.22	0.15	–	0.29	–	–
450 °C	90.27	–	1.85	0.31	4.03	2.99	0.29	–	–	0.22	–	–
500 °C	90.48	–	1.89	0.23	3.94	2.88	0.20	0.08	–	0.24	–	–
550 °C	90.76	–	1.83	0.19	3.62	2.73	0.20	–	0.31	0.26	–	0.05
600 °C	90.56	–	1.76	0.28	4.09	2.87	0.19	–	–	0.20	–	–
Heating rate of 20 °C/min												
400 °C	88.92	–	1.76	0.40	4.82	3.47	0.36	–	–	0.24	–	–

450 °C	90.36	–	1.75	0.34	4.17	2.80	0.25	0.08	–	0.22	–	–
500 °C	88.95	–	2.09	0.22	4.18	3.07	0.27	0.49	0.42	0.26	–	–
550 °C	91.48	–	1.59	0.29	3.39	2.66	0.18	0.12	–	0.25	–	–
600 °C	90.97	–	1.56	0.23	3.69	2.93	0.26	0.08	–	0.24	–	–

Table 5 presents the ultimate analysis of biochar obtained from RH pyrolysis at different temperatures and heating rates. The elemental composition of biochar was influenced by the pyrolysis temperature; carbon and other elemental contents increased, whereas the oxygen content decreased with increasing temperature. As reported in [66], increased carbonization enhances the carbon content, mainly in the form of aromatic carbon at high pyrolysis temperatures. The lower oxygen content in pyrolyzed biochar is attributed to dehydration and decarboxylation during the thermal decomposition of biomass [67].

Table 5. Ultimate analysis of biochar from RH pyrolysis at different temperatures and heating rates.

Heating Rate (°C/min)	Temperature (°C)	Elemental Composition (wt.%)				
		N	C	H	O	Others
5	400	0.37	42.49	2.20	7.54	47.38
	450	0.40	44.69	1.99	6.01	46.90
	500	0.35	43.85	1.73	4.29	49.76
	550	0.33	45.08	1.48	3.30	49.79
	600	0.36	45.95	1.32	2.98	49.37
10	400	0.36	42.49	2.41	9.02	45.70
	450	0.35	41.54	1.97	6.81	49.31
	500	0.38	45.65	1.77	4.50	47.68
	550	0.32	43.86	1.47	3.80	50.52
	600	0.34	44.47	1.22	3.02	50.92
20	400	0.36	43.87	2.37	9.45	43.93
	450	0.35	42.28	2.05	6.70	48.59
	500	0.34	41.76	1.67	4.46	51.74
	550	0.31	43.46	1.47	3.33	51.41
	600	0.34	45.01	1.25	2.89	50.48

Figure 7 shows the variations in the H/C and O/C ratios of biochar with pyrolysis temperature and heating rate. The results indicate that the H/C and O/C ratios decreased significantly with increasing pyrolysis temperature at all heating rates, primarily due to the reduction in hydrogen and oxygen contents and the enrichment of carbon. In contrast, the influence of heating rate on the H/C and O/C ratios was comparatively insignificant.

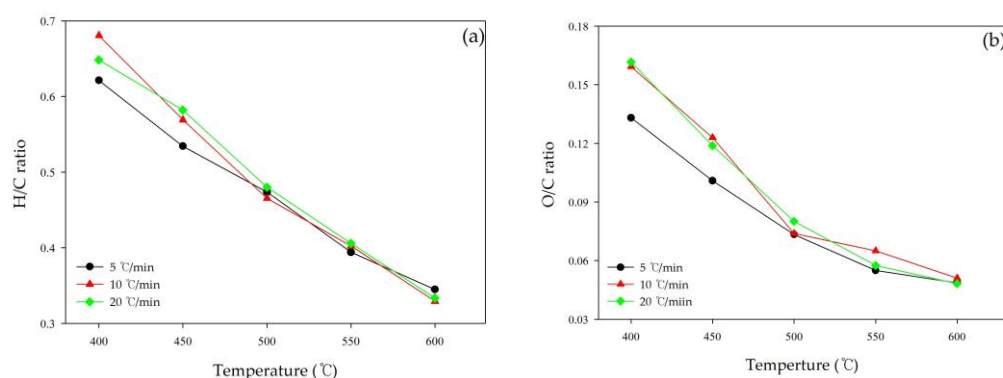


Figure 7. (a) H/C and (b) O/C ratios of biochar at various temperatures and heating rates.

Figure 8a illustrates the FTIR spectra of the raw RH and biochar under different pyrolysis temperatures with a heating rate of 10 °C/min. The results indicate that the peaks in the range of 3600–3200 cm⁻¹ in the raw material correspond to the O–H stretching of alcohol groups, and their intensity decreased with increasing pyrolysis temperature due to the loss of phenolic or alcoholic groups [68–70]. The band in the range of 2950–2850 cm⁻¹ is attributed to C–H stretching vibrations of aliphatic CH₂ and CH₃ groups, which are more pronounced in the raw material but decrease with increasing pyrolysis severity [28,68,71]. The C–H functional groups diminished with increasing temperature due to demethylation and dehydration during biochar formation [72]. The weak peaks in the range of 1700–1500 cm⁻¹ might be attributed to C=O stretching vibrations [57,71,73]. Therefore, as shown in Figure 8a, with increasing temperature above 400 °C, peaks assigned to hydroxyl, carboxyl, carbonyl, and methoxyl gradually decrease due to thermal cracking, indicating that these groups are transformed into gaseous components (CO, CO₂, and CH₄) and liquid products [69]. Also, the broad peaks observed at approximately 1100–1000 cm⁻¹ and 802–780 cm⁻¹ are attributed to SiO₂ structures and are present in all samples [74,75]. Figure 8b shows the XRD patterns of raw RH and biochar. XRD results showed the broad peaks at approximately 22° (2θ) in all samples (raw RH and biochar), which are attributed to the structure of cellulose [74,76]. These cellulose-derived peaks gradually decrease in intensity and become broader as the pyrolysis temperature increases, indicating a reduction in cellulose crystallinity [76].

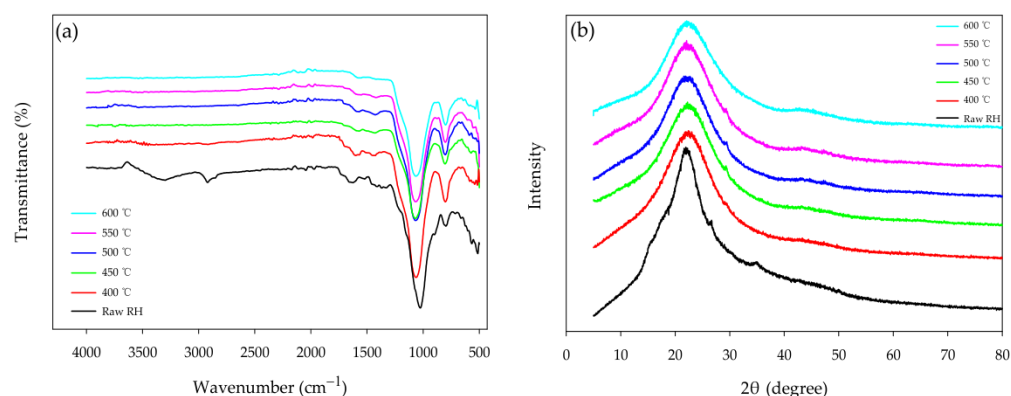


Figure 8. (a) FTIR spectra and (b) XRD patterns of raw RH and biochar obtained at various temperatures at a heating rate of 10 °C/min.

3.4. Kinetic Analysis of RH

Figure 9a shows the thermogravimetric analysis (TGA) curves used for the kinetic analysis of RH pyrolysis. The TGA results indicated that the thermal decomposition of RH occurs in two distinct stages. The weight loss below 200 °C (<4%) reflected moisture removal and was excluded from the kinetic analysis. Significant mass loss began at approximately 225 °C, with rapid decomposition occurring in the 350–390 °C range depending on the heating rate. This first stage was attributed to the thermal degradation of hemicellulose and cellulose, where hemicellulose begins to decompose at 225–300 °C [77], while cellulose rapidly decomposes at 300–400 °C [78]. The second stage, occurring above 400 °C, is attributed to lignin decomposition [79,80], although Kumar et al. [81] reported that lignin degradation extends over a wider temperature range of 500–800 °C. It has been reported that hemicellulose is prone to decomposing at low temperatures due to its low degree of polymerization, whereas cellulose undergoes depolymerization, leading to the formation of volatile products such as CO and CO₂, as well as liquid products [82]. The decomposition of lignin requires high temperatures owing to its aromatic rings, which have diverse side chains and chemical bonds [83,84]. There was no further weight reduc-

tion at the final temperature of 800 °C, and the solid residue remained at approximately 36–39%, depending on the heating rate. Figure 9b presents the derivative thermogravimetry (DTG) data. Overlapping DTG peaks were observed over the temperature range of 220–400 °C, depending on the heating rate, which are attributed to the decomposition of hemicellulose and cellulose. This overlap is attributed to the partial overlap in the decomposition temperature ranges of hemicellulose and cellulose.

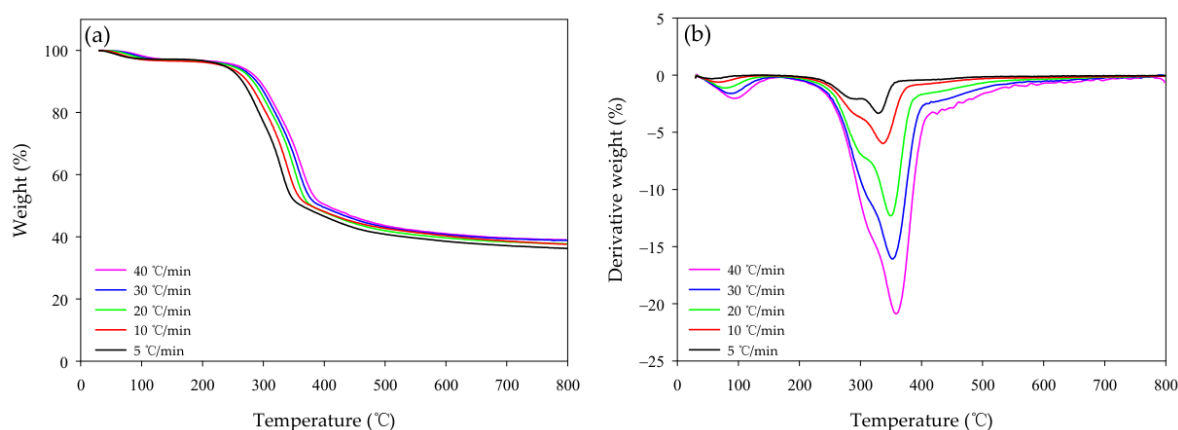


Figure 9. (a) Thermogravimetric analysis (TGA) and (b) derivative thermogravimetry (DTG) of RH at different heating rates.

Various methods have been employed for the kinetic analysis of thermal decomposition using TGA, including the Flynn–Wall–Ozawa, Kissinger, Horowitz–Metzger, Friedman, and Coats–Redfern methods. The Kissinger and Friedman methods are differential methods based on multiple heating rates, whereas the Horowitz–Metzger and Coats–Redfern methods are integral methods that utilize a single heating rate. In addition, the Flynn–Wall–Ozawa method is an integral method based on multiple heating rates. However, the Kissinger and Horowitz–Metzger methods assume first-order reaction kinetics and are applicable only over a limited degree of conversion, which restricts their ability to evaluate activation energies across the entire conversion range. Therefore, in this study, the Coats–Redfern method, which utilizes a single heating rate, and the Friedman method, which utilizes multiple heating rates, were employed as representative integral and differential methods, respectively, to investigate the pyrolysis kinetics of rice husk. This approach also enabled an assessment of the effects of both the mathematical methodology (integral versus differential methods).

3.4.1. Estimation of Activation Energy Using the Integral Method

The activation energy (E) at each heating rate was calculated using Equations (9) and (10), as shown in Figure 10. Because the change in conversion with temperature is small at conversion levels above 0.70 or below 0.10, resulting in increased uncertainty, the kinetic analysis was conducted over the conversion range of $0.10 < \alpha < 0.70$. As shown in Figure 10, the activation energy was determined from the slope of the line with the highest R^2 value for each assumed reaction order (n), and the results are summarized in Table 6. Table 6 shows that the activation energy of RH pyrolysis ranged from 52.23 to 60.90 kJ/mol with slight variation depending on the heating rate. This is comparable to values reported in [85], which ranged from 58.02 to 65.16 kJ/mol, but are lower than those reported in [86], which ranged from 72.7 to 97.5 kJ/mol.

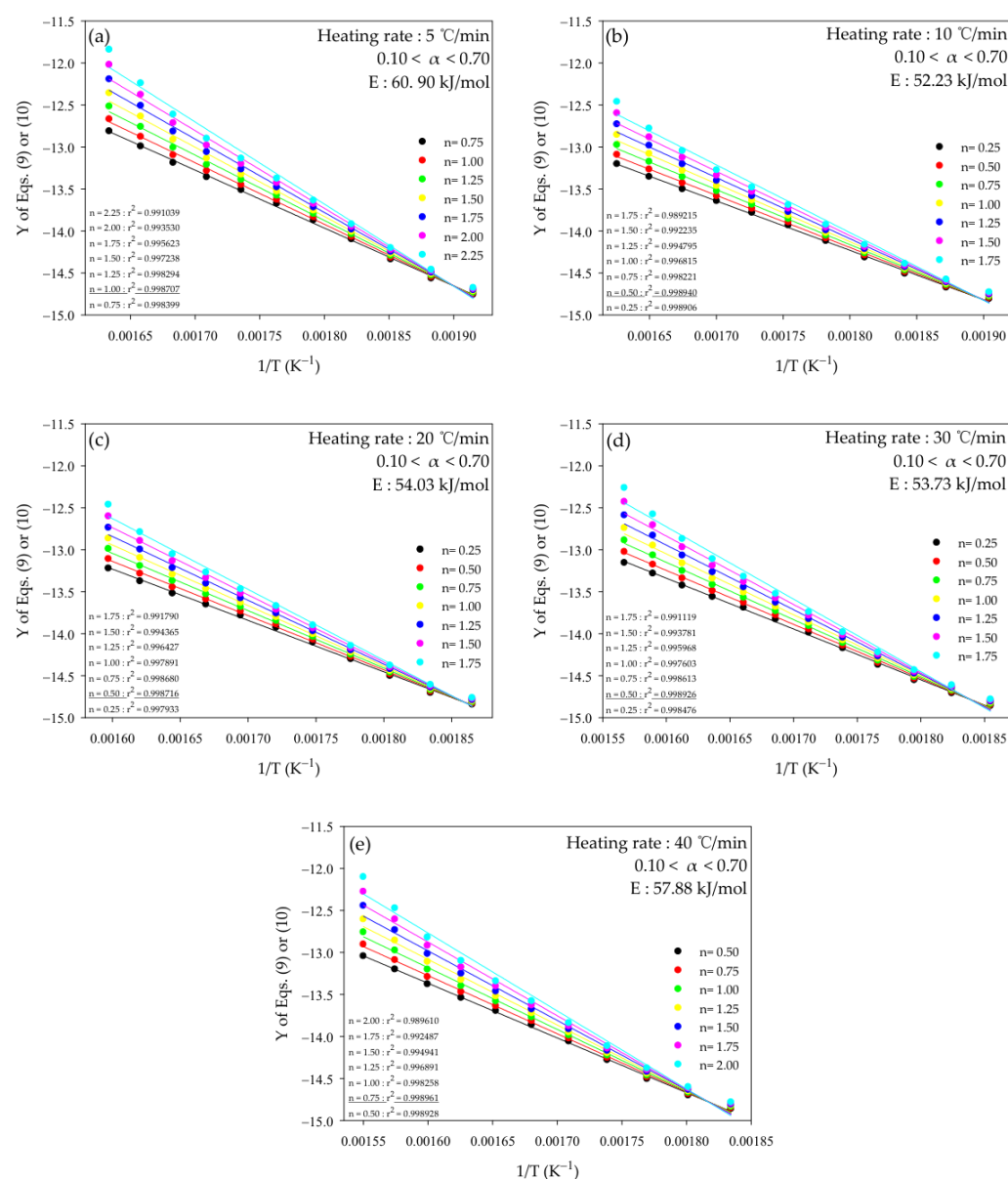


Figure 10. Application of the integral method at heating rates of (a) 5, (b) 10, (c) 20, (d) 30, and (e) 40 °C/min.

Table 6. Activation energies derived from the integral method at different heating rates.

Conversion (α)	Heating Rate (°C/min)	Activation Energy (kJ/mol)
0.10 < α < 0.70	5	60.90
	10	52.23
	20	54.03
	30	53.73
	40	57.88

3.4.2. Estimation of Activation Energy Using the Differential Method

Figure 11 and Table 7 show the activation energy results at various degrees of conversion (α) obtained from the differential method known as the Friedman method. Since this method uses multiple heating rates, it cannot evaluate the variation of activation energy with heating rate; however, it has the advantage of allowing the analysis of activation energy variation with conversion. The activation energy increased with increasing con-

version in the initial conversion range of $0.10 < \alpha < 0.45$. It reached a maximum of 214.18 kJ/mol at $\alpha = 0.40$, then slightly decreased up to $\alpha = 0.60$, and subsequently increased again with further conversion. Overall, the activation energies derived from the differential method were higher than those obtained from the integral method. This observed distinction may result from the complex multi-step decomposition characteristics of RH, whereas the integral method provides an apparent activation energy based on an assumed reaction model. As a result, the Friedman method is generally considered more suitable for describing the kinetic analysis of RH pyrolysis. The activation energies derived from the differential method were higher than those obtained using the integral methods. This discrepancy is likely attributable to differences in the mathematical formulations and assumptions underlying the two approaches. As mentioned above, a comparison between the activation energies reported in the literature and those calculated in the present study showed that the activation energies derived from the integral methods were generally in better agreement with the literature values. Although the activation energies obtained from the differential method were substantially higher than those derived from the integral methods, the differential method provides additional insight into the variation of activation energy as a function of conversion. Therefore, combining both methods is considered desirable to provide a more reliable kinetic interpretation of RH pyrolysis.

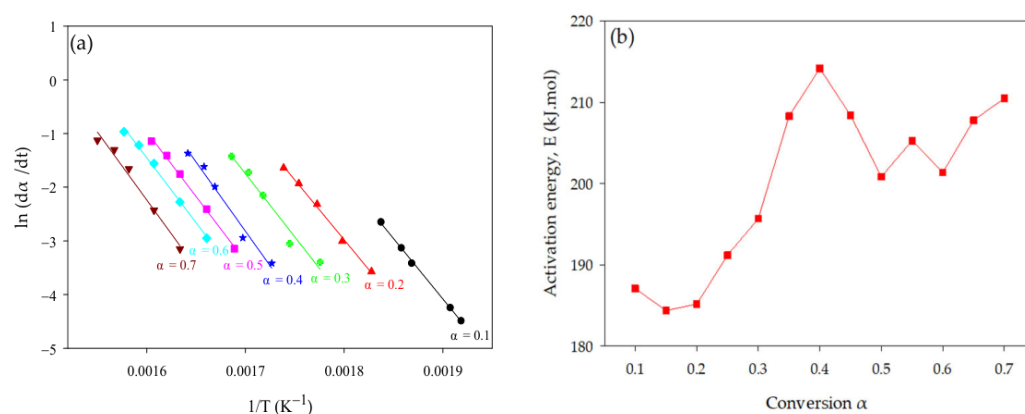


Figure 11. (a) Application of the differential method and (b) activation energies versus conversion.

Table 7. Apparent activation energies obtained from different degrees of conversion.

Conversion (α)	Temperature and Heating Rate	Activation Energy (kJ/mol)
0.10	248 °C (5 °C/min)–271 °C (40 °C/min)	187.10
0.15	237 °C (5 °C/min)–263 °C (40 °C/min)	184.39
0.20	247 °C (5 °C/min)–275 °C (40 °C/min)	185.18
0.25	255 °C (5 °C/min)–284 °C (40 °C/min)	191.21
0.30	263 °C (5 °C/min)–293 °C (40 °C/min)	195.69
0.35	271 °C (5 °C/min)–301 °C (40 °C/min)	208.30
0.40	279 °C (5 °C/min)–309 °C (40 °C/min)	214.18
0.45	286 °C (5 °C/min)–316 °C (40 °C/min)	208.40
0.50	292 °C (5 °C/min)–323 °C (40 °C/min)	200.85
0.55	297 °C (5 °C/min)–328 °C (40 °C/min)	205.28
0.60	302 °C (5 °C/min)–334 °C (40 °C/min)	201.37
0.65	307 °C (5 °C/min)–339 °C (40 °C/min)	207.80
0.70	312 °C (5 °C/min)–345 °C (40 °C/min)	210.49

4. Conclusions

The present study illustrated that conducting pyrolysis of rice husk (RH) at temperatures above 500 °C and a heating rate greater than 10 °C/min proved optimal for maximizing LVLP yield, whereas HVLP was produced mainly at lower heating rates. As the pyrolysis temperature rose, the biochar yield decreased, while the gas yields increased.

GC-MS analysis showed that heating rates significantly influenced the chemical composition of HVLP compared to LVLP. The liquid product derived from RH pyrolysis was mainly dominated by C6 compounds, followed by C8 and C5 compounds. In addition, the chemical composition of biochar was primarily affected by pyrolysis temperature rather than heating rate. XRD analysis revealed that the diffraction peak at approximately 22° corresponds to cellulose-related structures. Gas evolution analysis indicated that the concentrations of CO₂, CO, and CH₄ increased with increasing temperature, with a maximum CH₄ production observed at 500 °C. Furthermore, low nitrogen and sulfur elemental contents in both raw RH and biochar suggest their potential as cleaner and more environmentally friendly energy resources.

TGA demonstrated that RH decomposed in two main stages, associated with hemicellulose and cellulose, followed by lignin decomposition along with residual biochar. Within the conversion range of $0.10 < \alpha < 0.70$, the apparent activation energy determined by the integral method varied from 52.23 to 60.90 kJ/mol and was lower than that from the differential method. In contrast, the differential method illustrated higher activation values ranging from 184.39 to 210.49 kJ/mol, depending on the degree of conversion. Overall, applying both integral and differential methods to evaluate the activation energy of RH is recommended to provide a more reliable kinetic interpretation of RH pyrolysis.

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