Wood Pellet Driven-Biochar Characterization Produced at Different Targeted Pyrolysis Temperatures

Irna Thya Rani1,2, Jiho Yoo2,*, Byung Bae Park1,**, Wahyu Hidayat3, Bangun Adi Wijaya1,2, Sihyun Lee2, Sangdo Kim2, Hokyung Choi2, Donghyuk Chun2, Hyuk Im2, Soohyun Kim2

1 Department of Environment and Forest Resources, College of Agriculture and Life Science, Chungnam National University, Daejeon, Republic of Korea
2 Clean Air Research Laboratory, Korea Institute of Energy Research (KIER), Daejeon, Republic of Korea
3 Department of Forestry, Faculty of Agriculture, University of Lampung, Bandar Lampung, Indonesia

* Corresponding Author. E-mail address: jyoo@kier.re.kr
** Corresponding Author. E-mail address: bbpark@cnu.ac.kr

ABSTRACT

The imperative transition to renewable energy drives the need for innovation. Biomass, particularly wood pellets, has demonstrated poor performance in co-firing scenarios. This study employed pyrolysis to convert wood pellets into biochar with improved fuel quality. The biochar production and characterization were investigated at pyrolysis temperatures of 400°C to 500°C. The findings revealed significant improvements: the observed fixed carbon content increased from 67.2% to 78.8%, and the calorific value increased 1.2 times higher within the pyrolysis temperature increased. On the other hand, as the pyrolysis temperature increased from 400°C to 500°C, biochar yields decreased from 49% to 37%. Thermogravimetric (TG) analysis revealed distinct weight loss during heating, illuminating component volatilization and residue accumulation. Furthermore, Fourier transform infrared (FTIR) spectroscopy elucidated structural changes, demonstrating the evolution of cellulose and hemicellulose into aromatic structures. Ultimately, these insights into biochar characteristics informed the optimization of pyrolysis processes, contributing to the production of superior biochar for renewable energy applications.

1. Introduction

The world’s population has grown significantly in recent decades, leading to an ever-increasing need for goods, chemicals, and energy sources (Chen et al. 2018). The substantial population growth is accompanied by a simultaneous surge in worldwide emissions of greenhouse gases (GHGs) (Mishra and Mohanty 2021); this surge poses a significant obstacle to achieving the objective of Negative Emission Technology (NET) zero emissions by 2050 (Simmons et al. 2021; Muslih et al. 2022). In addition to climate goals, the urgency of achieving carbon neutrality is motivated by the need to limit the average global temperature rise to well below the critical two-degree threshold (Qi et al. 2020). Among the top 10 countries with the highest carbon dioxide emissions, Indonesia ranks 12th (Pambudi et al. 2023). In 2025, Indonesia is expected to achieve a 23% renewable energy mix (Yudiartono et al. 2023); 3.59% will be co-fired (Reyseliani and Purwanto 2021). Unfortunately, the government has to tackle many challenges to achieve the
target, such as technological transformation (Raihan et al. 2023), sustainable resources, and public policy (Pambudi et al. 2023). This tendency suggests that Indonesia needs to allocate resources towards the development of renewable energy options such as solar, wind, and bioenergy. Additionally, it should focus on enhancing regulations and technological advancements to boost the efficiency and cost-effectiveness of these sources. To reduce emissions, there is a critical need to promote sustainable resource management and conservation practices. Furthermore, Indonesia should prioritize improving its energy infrastructure and increasing its investments in the renewable energy sector.

Co-firing involves coal combustion in coal-fired power plants while incorporating biomass, reducing coal consumption (Sami et al. 2001). This approach offers a means for Indonesia to diminish its dependence on coal and transition toward cleaner energy sources, consequently curbing emissions and enhancing sustainability within its energy sector. Nevertheless, co-firing presents challenges as biomass performs less efficiently than coal, as highlighted in recent studies (Chen and Liu 2023; Hariana et al. 2023; Shobar et al. 2020; Sugiyono et al. 2023). Additionally, biomass combustion is challenging to control due to its high moisture content and lower energy density (San Juan et al. 2021), resulting in lower energy output than coal. Therefore, to make biomass suitable for co-firing it necessitates pretreatment. One of the pretreatment methods that is commonly used is pyrolysis (Antonangelo et al. 2019). Pyrolysis is a thermochemical treatment that converts biomass into carbon-rich products (Haryanto et al. 2021; Yaashikaa et al. 2020). When pyrolysis occurs in an oxygen-free environment (inert atmospheric conditions), biomass is heated to temperatures ranging from 400°C to 900°C (Hadiya et al. 2022). Within this process, biomass is transformed into various components, including bio-oil (liquid), biochar (solid), and syngas (gaseous). Various factors, including the process conditions, influence the relative proportions of these products (Vuppaladadiyam et al. 2022). The char produced through biomass pyrolysis is known as biochar, which stands out as the predominant product of the pyrolysis process, possessing a high energy density, rendering it a viable fuel to blend with coal for boiler applications (Wang et al. 2020).

A range of biomass sources can be utilized to produce biochar. For instance, Pipiška et al. (2022) and Rubiyanti et al. (2019) employed wood pellets, which are commonly used for biochar production in co-firing applications. Additionally, various other biomass sources such as agricultural wastes (Nabila et al. 2023; Yrjälä et al. 2022), food wastes, sludge (Kumar et al. 2021), and manure (Wani et al. 2021) can also be harnessed to generate biochar. The unique characteristics of these biomass types significantly influence the resulting biochar properties. Furthermore, the attributes of biochar can be influenced by the specific pyrolysis conditions applied, including factors such as temperature and residence time, which can impact biochar yield and chemical composition (Makavana et al. 2020; Mohd et al. 2019; Yang et al. 2020). By controlling the temperature and residence time, the pyrolysis process can produce a biochar product with specific properties. Thus, it is imperative to consider these factors when setting up a pyrolysis process to generate the desired biochar product.

Pyrolysis temperature in biochar production is vital because it influences biochar’s yield, properties, and quality. Different temperatures can result in biochar with varying characteristics, impacting its suitability for applications such as soil improvement, contaminant removal, and carbon sequestration, which is still subject to research. This paper aimed to analyze the characteristics of biochar-driven wood pellets at specifically targeted temperatures ranging from 400°C to 500°C. The characteristic encompasses the physicochemical of biochar for fuel. Thus,
studying these characteristics can guide the design of pyrolysis processes, allowing for the best production of biochar products.

2. Materials and Methods

2.1. Feedstocks Preparation

The selection of commercially procured biomass was Idemitsu Wood Pellets obtained from Yeoju, South Korea, with a diameter of 0.8 cm. At the stage for the investigation, a carefully batched of approximately 2 kg of wood pellets was used as the raw material for subsequent biochar production. The physicochemical properties of wood pellets are detailed in Table 1.

2.2. Pyrolysis Process

Biochar was produced via pyrolysis using a custom counter flow multi baffle (COMB) reactor (Hidayat et al. 2024). First, wood pellets were fed into a feeder connected to a container. The setup was then purged with a nitrogen flow rate of 5.3 m³/min, creating an oxygen-free environment needed for pyrolysis. Subsequently, three peaks charring temperatures were applied at 400°C, 450°C, and 500°C with a heating rate of 10°C/min. The process was continued pyrolyzed for up to 15 minutes, following a cooling phase at room temperature, and the resulting biochar was carefully weighed.

2.3. Biochar Characterization

In preparation for feedstock and biochar characterization, a grinding process was employed to obtain a consistent particle size before conducting several detailed analyses.

2.3.1. Proximate analysis

Proximate analysis was conducted using a thermogravimetric analyzer (TGA801, LECO Korea Co., Ltd., Seoul, Korea) to determine volatile matter (VM), ash, and fixed carbon (FC). Empty crucibles were loaded into the sample carousel, and the weights were automatically recorded. Subsequently, approximately 0.5 g samples were added, and the weights were recorded before the method-specified furnace-temperature program commenced. The mass change of each sample was continually monitored throughout the analysis, and typically, the mass change percentage was reported after each step.

2.3.2. Ultimate analysis

Ultimate analysis was performed using an elemental analyzer (Vario EL Cube, Elementar, EA Korea Co., Ltd., Seoul, Korea). Samples of approximately 2 mg were meticulously prepared to ensure representativeness. The sample combustion process was initiated within a furnace set at 1,150°C, followed by an afterburner at 850°C. The elemental composition, encompassing carbon (C), hydrogen (H), sulfur (S), nitrogen (N), and oxygen (O), was determined at once through the instrument’s integrated software. The software facilitated accurate calculations and analysis of the resulting data, i.e., all elemental composition was obtained and analyzed automatically within the system.
2.3.3. Calorific value analysis

Calorific value (CV) was evaluated using an oxygen bomb calorimeter (6400 Automatic Isoperibol Calorimeter, Parr Instrument Company, MK Science Co., Ltd., Seoul, Korea). The calorimeter was set up following the manufacturer’s instructions and ensured proper calibration. The oxygen bomb was assembled by placing approximately 0.5 g sample into a heat-resistant crucible and securely sealing it with oxygen. The sample within the bomb was ignited, allowing heat release during combustion. The calorimeter then accurately measured this heat change, providing data on the CV of the sample.

2.3.4. Thermogravimetric and derivative thermogravimetry analysis

Thermogravimetric (TG) and derivative thermogravimetry (DTG) were analyzed using a thermogravimetric analyzer (SDT-Q600, TA Instruments-Waters Korea Ltd., Seoul, Korea) to investigate the thermal attributes of materials. TG and DTG analysis was conducted using 10 mg sample mass, a heating rate of 10°C/minute from room temperature to 900°C, and an inert nitrogen atmosphere maintained at a 100 mL/min flow rate. Before initiating these analyses, detailed calibration of the TG and DTG systems was undertaken, while experimental repetition was embraced to confirm the reliability of the result.

2.3.5. Fourier transform infrared (FTIR) spectroscopy analysis

The functional group determination of the biochar samples was inspected using an FTIR-ATR spectrometer (Perkin Elmer Spectrum 100 FTIR/ATR, Perkin Elmer Korea, Seoul, Korea). The FTIR provided precise information, enhancing comprehension of material structural characteristics. Approximately 1 g of the sample was manually ground with a pestle and mortar to achieve a particle size of < 0.2 mm. The milled samples were then placed directly on the ATR sampling accessory window crystal, and the spectra were obtained over the wavenumber range from 4000 cm\(^{-1}\) to 800 cm\(^{-1}\), respectively.

3. Results and Discussion

3.1. Proximate, Ultimate, and Calorific Value

The analysis results presented in Table 1 provide crucial insights into the transformations that occur during the pyrolysis process, highlighting variations in proximate and ultimate for both raw feedstock and the resulting biochar samples.

<table>
<thead>
<tr>
<th>Treatment</th>
<th>Proximate (wt.%) db</th>
<th>Ultimate (wt.%) daf</th>
</tr>
</thead>
<tbody>
<tr>
<td>Raw</td>
<td>18.9 80.9 0.2 48.5 3.8 44.6 2.5 0.6</td>
<td></td>
</tr>
<tr>
<td>400°C</td>
<td>67.2 28.7 4.1 68.8 2.8 27.3 0.8 0.3</td>
<td></td>
</tr>
<tr>
<td>450°C</td>
<td>73.7 21.7 4.5 74.5 1.8 22.8 0.7 0.3</td>
<td></td>
</tr>
<tr>
<td>500°C</td>
<td>78.8 16.5 4.8 80.4 1.1 17.5 0.7 0.2</td>
<td></td>
</tr>
</tbody>
</table>

Notes: FC = fixed carbon, VM = volatile matter, CV = calorific value, db = dry base, daf = dry ash free.
Regarding proximate analysis, the raw feedstock exhibits a substantial volatile matter (VM) content of 80.9%, indicating its high susceptibility to thermal degradation. This result aligns with the typical behavior of biomass, known for its abundant volatile components (Muhammad et al. 2021). However, pyrolysis significantly alters this composition, especially as the charring temperature increases. In the case of biochar at 400°C, the VM content drops remarkably to 28.7%, accompanied by a substantial increase in fixed carbon (FC) to 67.2%. This shift signifies the progressive breakdown of volatile components into more stable carbon-rich structures, resulting in the development of a charred product (Lapczyńska-Kordon et al. 2022). As the temperature further elevates to produce biochar at 450°C and 500°C, the FC content continues to increase while volatile matter further diminishes (Kwon et al. 2020). This trend underscores the increasingly pronounced influence of temperature on driving volatile components off the matrix, leading to higher carbon concentration within the biochar (Yang et al. 2018).

Furthermore, the ultimate analysis provides insights into the elemental composition changes induced by pyrolysis. With increasing pyrolysis temperature, there is a general reduction in all elemental components except carbon (Wakamiya et al. 2022). This is a direct consequence of the volatilization of hydrogen, oxygen, nitrogen, and sulfur-rich compounds during pyrolysis, leaving behind a more carbon-enriched residue (Zhou et al. 2021). Notably, the carbon content in the biochar samples is substantially higher than in the raw feedstock, corroborating the carbon enrichment achieved through pyrolysis. Additionally, Fig. 1 illustrates the relationship between feedstocks (raw biomass and different biochar samples) and their corresponding biochar yield and CV.

![Graph showing the relationship between yield and calorific value (CV) vs. temperature]

**Fig. 1.** The dynamic trade-off between yield and calorific value (CV) underscores the impact of pyrolysis temperature on biochar production characteristics.

As depicted in Fig. 1, biochar yield decreases with increasing pyrolysis temperature. The raw biomass yields 100% of biochar, but this yield diminishes as the temperature rises. For instance, biochar at 400°C yields around 49%, while biochar at 450°C and 500°C yields even less at approximately 39% and 37%, respectively. This decreasing trend highlights how higher
pyrolysis temperatures result in lower biochar production \((\text{Cha et al. 2016})\). Conversely, the CV, which signifies the energy content, exhibits a contrasting pattern. The raw biomass has a CV of 4,480 kcal/g. As the pyrolysis temperature increases, the CV of the produced biochar also rises. For instance, biochar at 400°C has a higher CV of 6,332 kcal/g, biochar at 450°C further elevates to 6,496 kcal/g, and biochar at 500°C reaches the highest CV of 7,633 kcal/g. This upward trend underscores that higher pyrolysis temperatures yield biochar with enhanced energy content \((\text{Zhang et al. 2017})\). In essence, the graph illuminates the trade-off between biochar yield and energy content. While higher pyrolysis temperatures lead to lower biochar yields, they concurrently result in biochar with greater energy potential, reinforcing the complex dynamics of biomass conversion through pyrolysis \((\text{Tripathi et al. 2016})\).

In summary, the proximate, ultimate, and calorific value analysis illustrates the significant transformations that take place during biomass pyrolysis. The process enhances the carbon content, energy density, and stability of the resulting biochar, rendering it a potentially valuable resource for energy generation and environmental applications \((\text{Hu et al. 2016})\). These findings underline the critical role of pyrolysis temperature in modulating the physicochemical properties of biochar, thereby guiding future endeavors in biomass conversion and sustainable energy solutions \((\text{García et al. 2021})\). Fig. 2 illustrates the changing hydrogen-to-carbon ratio (H/C) and oxygen-to-carbon ratio (O/C) among different feedstocks: the initial raw feedstock and three biochar samples produced at varying pyrolysis temperatures.

The diagram depicts the changes in H/C and O/C for different feedstocks, including raw biomass and biochar samples generated at various pyrolysis temperatures. As the pyrolysis temperature increases, the H/C and O/C ratios decrease. This tendency leads to the enrichment of carbon content in the resulting biochar \((\text{Kim et al. 2012})\). The descending trend in H/C and O/C ratios is visually evident, highlighting the influence of pyrolysis temperature on the elemental composition of biochar. The data points for raw feedstock biochar at 400°C, 450°C, and 500°C are
connected, illustrating the systematic alteration in composition through the pyrolysis process. This trend underscores the significance of temperature modulation in shaping biochar properties, particularly its carbon-centric disposition and energy density enhancement (Reza et al. 2020).

3.2. TG and DTG Analysis

The TG and DTG analysis results for the raw sample unveil a comprehensive depiction of its thermal degradation behavior across different heating stages. These analyses are fundamental in understanding how the sample evolves under increasing temperatures, as shown in Fig. 3 and Table 2.

![Fig. 3. Thermal degradation behavior across different heating stages for raw biomass feedstock.](image)

The TG and DTG analysis of the raw sample reveals distinct weight loss stages. In the initial stage, there is a weight loss of approximately 9.25%. This initial weight loss is attributed to the release of volatile matter and moisture content in the sample as the temperature begins to rise. As the temperature advances, the sample enters the second stage, characterized by a significant weight loss of about 68.55%. This stage corresponds to the breakdown of complex organic compounds within the sample. The third stage indicates an additional weight loss of approximately 3.8%, possibly signifying the further decomposition of residual organic matter. These successive stages of weight loss culminate in a residue of 18.4%, representing the stable material left behind after the pyrolysis process (Cisse et al. 2022).

<table>
<thead>
<tr>
<th>Samples</th>
<th>Weight loss (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Raw</td>
<td>Stage 1</td>
</tr>
<tr>
<td></td>
<td>9.25</td>
</tr>
</tbody>
</table>

Table 2. The thermal dynamics and compositional changes within biochar responding to the increase in temperature
The DTG analysis provides a derivative curve corresponding to the weight loss rate at different heating stages. This curve offers a complementary perspective, pinpointing the peaks in the weight loss rate associated with each degradation stage. The combined information from TG and DTG analyses affords a detailed understanding of the dynamic changes that occur during the thermal degradation of the raw sample (Ahmed et al. 2020). In summary, the TG and DTG analysis of the raw sample offers valuable insights into its thermal behavior, unveiling the distinct weight loss stages and shedding light on the evolving composition as the temperature increases. This in-depth analysis forms the basis for understanding the intricate transformation mechanisms that biomass undergoes during the pyrolysis process (Arous et al. 2021).

3.3. FTIR Analysis

The FTIR analysis shows the intricate molecular vibrations and functional groups within the biochar samples, shedding light on their structural composition and chemical characteristics (Fig. 4 and Table 3).

![FTIR analysis peaks and characteristic vibrations for biochar samples at different pyrolysis temperatures.](image)

In the FTIR analysis results, distinct peaks and characteristic vibrations emerge at specific wavenumbers, revealing the presence of various functional groups and molecular bonds. It was found that the transmittance percentages were higher in raw and decreased as the temperature increased to 500°C biochar. The detected peaks were found at 3393 cm⁻¹ for the O-H stretching peak, which appears consistently in all samples. It signifies the stretching vibrations of alcoholic and phenolic hydroxyl (Zhang et al. 2018). Hydroxyl functionalities are crucial due to their versatile reactivity and ability to form hydrogen bonds, which play vital roles in chemical synthesis and biological processes (He et al. 2018). The wavelength for the peaks at 2926 cm⁻¹ was for C-H stretching, which corresponds to the stretching vibrations of aliphatic carbon-hydrogen bonds (Fu et al. 2019), commonly found in various organic compounds, which is an indication of the presence
of cellulose, hemicellulose, and lignin (Figueiredo et al. 2017). Additionally, peaks at 1601 cm\(^{-1}\) for aromatic C=C and C=O were observed in raw and biochar at 400\(^\circ\)C, and it weakened in biochar at 450\(^\circ\)C and 500\(^\circ\)C. These aromatic C=C bonds, commonly present in benzene rings, and C=O bonds, characteristic of carbonyl compounds, are notable for their resonance-stabilized structures in organic chemistry (Santos et al. 2015). Furthermore, aromatic C-O-C stretching was found at peaks 1035 cm\(^{-1}\), and this peak was observed in all samples. Aromatic C-O-C stretching vibrations are essential for identifying aromatic ethers in organic compounds, aiding in structural analysis and quality control in fields such as pharmaceuticals and materials science (Cantrell et al. 2012). Additionally, it provides valuable insights into molecule reactivity and functional group composition in cellulose and hemicellulose, components of plant biomass (Rani et al. 2020). The presence of this peak suggests the persistence of these structural components even after pyrolysis.

### Table 3. FTIR analysis peaks and characteristic vibrations for biochar samples at different pyrolysis temperatures

<table>
<thead>
<tr>
<th>FTIR peak (cm(^{-1}))</th>
<th>Treatment</th>
<th>Characteristic vibrations</th>
<th>Functionality</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Raw 400(^\circ)C 450(^\circ)C 500(^\circ)C</td>
<td></td>
<td></td>
</tr>
<tr>
<td>3393 S</td>
<td>N N N</td>
<td>O-H stretching</td>
<td>Alcohol, and phenolic -OH</td>
</tr>
<tr>
<td>2926 S</td>
<td>N N N</td>
<td>C-H stretching</td>
<td>Aliphatic CH(_x)</td>
</tr>
<tr>
<td>1601 S</td>
<td>S S W</td>
<td>Aromatic C=C, and C=O</td>
<td>Aromatic components and C=O of conjugated ketones and quinones</td>
</tr>
<tr>
<td>1035 S</td>
<td>S S S</td>
<td>C-O-C stretching</td>
<td>C-O-C ester groups in cellulose, and hemicellulose</td>
</tr>
</tbody>
</table>

Notes: S= Strong; W= Weak; N= Negligible.

In summary, the FTIR analysis highlights the presence of alcoholic and phenolic hydroxyl groups, aliphatic carbon-hydrogen bonds, aromatic compounds, and cellulose/hemicellulose components. This information is a valuable tool for understanding the molecular makeup and functional groups within the biochar samples, further enriching our comprehension of their structural properties and potential applications (Wakamiya et al. 2022).

### 4. Conclusions

This study investigated the dynamic relationship between pyrolysis temperature and resulting biochar properties. As pyrolysis temperature increased, volatile matter decreased while fixed carbon content and energy density substantially rose in the produced biochar. This escalating temperature also led to lowered hydrogen-to-carbon and oxygen-to-carbon ratios, indicative of a shift towards a carbon-enriched structure. Thermogravimetric analysis revealed distinct weight loss stages during heating, uncovering the volatilization of different components and residue accumulation. Fourier transform infrared analysis depicted shifts in characteristic vibrations, underlining the transformation of cellulose and hemicellulose into aromatic structures. Collectively, these findings emphasize the important role of pyrolysis temperature in molding biochar attributes, offering insights for sustainable energy and environmental applications. The
complex interactions discovered by our research help to develop biomass pyrolysis technologies, which have the potential to provide a more ecologically friendly and efficient energy future.

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References


