

Evaluation of Pyrolysis Temperature and Time on The Quality Improvement of Calliandra Wood Charcoal Briquettes as An Alternative Energy Source

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ABSTRACT. Calliandra (*Calliandra calothyrsus*) wood is a promising biomass feedstock due to its abundance and high calorific value. This study investigated the effects of pyrolysis temperature (400 °C and 500 °C) and residence time (30 and 60 minutes) on the quality of calliandra charcoal briquettes. Biomass was carbonised, mixed with 15% cassava starch binder, pressed, and oven dried. The briquettes were evaluated for yield, density, moisture, ash, volatile matter, fixed carbon, calorific value, combustion rate, and structural changes. Results showed that all parameters except yield were significantly influenced by temperature and time. FTIR analysis revealed increased aromatisation at higher pyrolysis conditions, while XRD indicated reduced cellulose crystallinity and the emergence of calcite and silica phases. The briquettes exhibited densities of 0.62–0.63 g/cm³, moisture contents of 2.14–2.73%, ash 3.55–4.91%, volatile matter 16.32–25.11%, fixed carbon 68.60–76.63%, calorific values 7,856–7,978 cal/g, and combustion rates 0.08–0.09 g/min. The condition of 400 °C for 30 minutes offered the most balanced performance, while 500 °C for 60 minutes yielded the highest carbonisation and energy content. These findings highlight that moderate pyrolysis conditions can produce efficient, high-quality briquettes with low energy input.

Keywords: Biomass, calliandra wood, energy, pyrolysis, wood charcoal briquettes

INTRODUCTION

One of the primary human needs is energy to carry out daily activities. New renewable energy (NRE) comes from nature and can be renewed. Renewable energy can be one of the alternative sources of energy supply because it has a low impact on environmental damage and ensures energy sustainability in the future (Setyono et al., 2019). Wind, geothermal, and biomass are examples of NRE energy with great potential in Indonesia (Adistia et al., 2020). Among the various types of new and renewable energy, biomass stands out as one of the most plentiful resources, offering versatile possibilities for conversion into different energy forms.

Biomass is organic material produced as products and waste from photosynthesis (Almu et al., 2014). It comprises cellulose and lignin with an energy content of 4,000-5,000 cal/g. Because they contains carbon

atoms (C) and consists of cellulose and lignin, biomass can contribute about 14% of global energy (Onchieku et al., 2012). Wood is one type of biomass that is very prospective (Amrullah et al., 2021) and calliandra wood is one type of wood biomass that can be used as an energy source. *Calliandra calothyrsus*, is a fast-growing, adaptive, and easy-to-develop plant. However, the leaves are only taken for livestock as they contain much protein, while the wood is discarded or burned (Daning, 2017). Previous studies report that the calorific value of calliandra wood is very high, reaching 4,600-7,200 cal/g (Pradana & Bunyamin, 2021), and contains between 20–23% lignin (Rusolono et al., 2018). Due to its high calorific value, fast growth, and resistance to heavy pruning, the Ministry of Environment and Forestry of Indonesia recommends calliandra wood as one of the firewood-type plants (Rusolono et al., 2018). Calliandra wood

produces around 65 m³/ha/year of biomass (Febijanto, 2019). Its wood density ranges from 0.50 to 0.80 g/cm³, which contributes to high energy content and combustion stability, although its relatively low moisture and anatomical structure allow it to dry rapidly and ignite efficiently (Kalina et al., 2022; Simpson & Tenwolde, 1999; Stewart et al., 2001; White & Dietenberger, 2010). While the leaves are used as animal feed, the branches and twigs are often discarded or burned on site, resulting in waste and air pollution. Converting this wood into charcoal can turn a disposal problem into a valuable energy commodity.

Charcoal briquettes are agglomerated products of carbonised biomass. They are denser than raw charcoal, burn more steadily and are easier to handle and transport. Some advantages of using wood charcoal briquettes as fuel are that they are cheaper, environmentally friendly, easy to store, and renewable (Dharma et al., 2017). However, the quality of briquettes depends strongly on the feedstock, binder, and pyrolysis conditions. Previous research on calliandra wood biomass has examined torrefied pellets and biochar. For example, there a study torrefied calliandra wood pellets at 300°C for 60 min and observed ash contents of 0.90% and calorific values of 6,303.8 cal/g (Sutapa & Hidayatullah, 2023), while biochar today reported that slow pyrolysis of calliandra wood at 500°C for 60 min produced charcoal with moisture content 6.87%, ash content 5.65%, volatile matter 10.10% and fixed carbon 77.40%, resulting in a heating value of 7,145 cal/g (Pasaribu et al., 2025). These studies highlight that calliandra wood biomass produces charcoal with moderate yield but high fixed carbon and energy density at temperatures above 300°C.

Despite these advances, few studies have optimised pyrolysis parameters for calliandra wood briquettes or analysed the effect of binder ratios. Optimal pyrolysis conditions are feedstock-specific; a general range of 350–650°C and 30–90 min is recommended for woody biomasses (Birhanu et al., 2025; Yang et al., 2016), but the optimum for calliandra wood has not been thoroughly investigated. Moreover, cassava starch is a promising binder because its gelatinised amylose and amylopectin form hydrogen-bonded networks that enhance briquette strength and calorific value. For leafy or fibrous biomass, binder concentrations of 15–40% (biomass:binder ratios around 3:5) are recommended to ensure adequate cohesion (Obi et al., 2022). However, there is limited discussion of binder selection and ratio in calliandra wood briquettes. The present study aims to fill these gaps by systematically investigating how pyrolysis temperature (400°C and 500°C) and residence time (30 and 60 min), combined with 15% cassava-starch binder, affect the physicochemical properties of calliandra wood charcoal briquettes. The objectives were to (i) determine the yield, density, moisture content, ash content, volatile matter, fixed carbon,

calorific value and combustion rate of briquettes under different pyrolysis conditions; (ii) compare the results with existing standards and previous calliandra wood studies; and (iii) identify the conditions producing briquettes with the best balance between energy content and resource use.

EXPERIMENTAL SECTION

The materials used in this study were ±6 years old calliandra wood branches and twigs obtained from the utilization block of Wan Abdul Rachman Forest Park, Bandar Lampung, cassava starch as an adhesive, a red methyl indicator, 0.0709 N Na₂CO₃, and distilled water.

Pyrolysis Process

Calliandra wood in the form of branches and twigs is chopped and air-dried in the sun for seven days until it reaches a moisture content of 12 ± 1%, measured by oven drying. The material was put into a fixed-bed type reactor without inert gas flow. The reactor was heated from room temperature to 400°C and 500°C at a heating rate of 10°C per minute. At each pyrolysis temperature, the holding time was set for 30 min and 60 min. After the pyrolysis time is complete, the heating is stopped and the reactor is allowed to cool naturally to a safe temperature. The final product produced in the form of calliandra wood charcoal was taken after the reactor cooled down.

Wood Charcoal Briquettes Manufacture

Calliandra wood is charred using the pyrolysis method. Furthermore, the charcoal is pulverized using a pestle, then sieved with a size of 40–60 mesh in order to obtain uniform charcoal particles. The refined charcoal flour is then mixed with adhesive to form a homogeneous mixture. Charcoal biobriquettes were made with 15% adhesive and 75% charcoal raw materials. The biobriquette paste was then put into a briquette mold and was pressed using hydraulic press with a pressure of 32.88 MPa. The molded briquettes were dried in an oven at 40°C for 24 hours.

Determination of Wood Charcoal Briquettes Characteristics

The wood charcoal briquette characteristics determination followed the standards of SNI 01-6235-2000 (Badan Standardisasi Nasional, 2000), EN 1860-2 (European Committee for Standardization (CEN), 2005), and GOST 7657-84 (Gosudarstvenny Standard, 1984), including density, moisture content, ash content, volatile matter, fixed carbon, calorific value, burning rate, and charcoal yield.

Fourier Transform Infrared Spectroscopy (FTIR) Analysis

5 mg of calliandra wood charcoal powders (40–60 mesh) were analyzed using an FTIR spectrometer (Bruker-Tensor II, Germany) over the range 4000–500 cm⁻¹ by the Attenuated Total Reflectance (ATR) method. Samples were scanned 45 times with a resolution of 2 cm.

X-ray diffractometer (XRD) Analysis

An X-ray diffractometer (XRD, Maxima, Shimadzu, Kyoto, Japan) was used to investigate the crystalline characteristics of bamboo strands. Segal’s method was used to calculate the relative crystallinity (Burton et al., 2009; Segal et al., 1959).

RESULTS AND DISCUSSION

Wood Charcoal Yield

Wood charcoal yield is the result of charcoal produced from the pyrolysis process to the initial weight of raw materials before the pyrolysis process. The yield is calculated to determine the efficiency of calliandra wood in turning charcoal through the pyrolysis process. The yield is expressed in percentage yield (%), and the higher the yield value, the more efficient the manufacturing process. Several factors affect charcoal yield, including pyrolysis temperature, pyrolysis time, and the type and condition of raw materials.

The results showed the highest charcoal yield in the 400°C 60 min treatment, followed by 500°C 30 min, 400°C 30 min, and 500°C 60 min treatments (**Figure 1**). Pyrolysis at high temperatures produces lower yields because some of the organic material that makes up the wood will turn into gas. According to Park, (2018), the charring yield (weight and volume) will decrease as the charring temperature increases. At 500°C, the chemical components of wood, such as hemicellulose, followed by cellulose and lignin, are decomposed to gas and other volatile matter, leaving charcoal or solid carbon and non-volatile components. In addition to the treatment, the condition of the raw materials also affects the charcoal yield produced. The raw material conditions include wood type and wood moisture content. This study’s results follow research conducted by Kalina et al., (2022), which showed that lignocellulose-based materials such as wood produce yields of around 25–37% depending on the pyrolysis temperature and time.

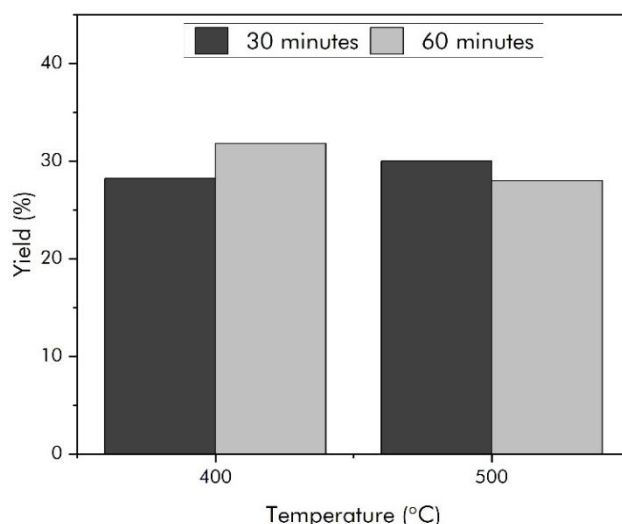


Figure 1. Wood charcoal yield of briquettes at various pyrolysis temperatures and times

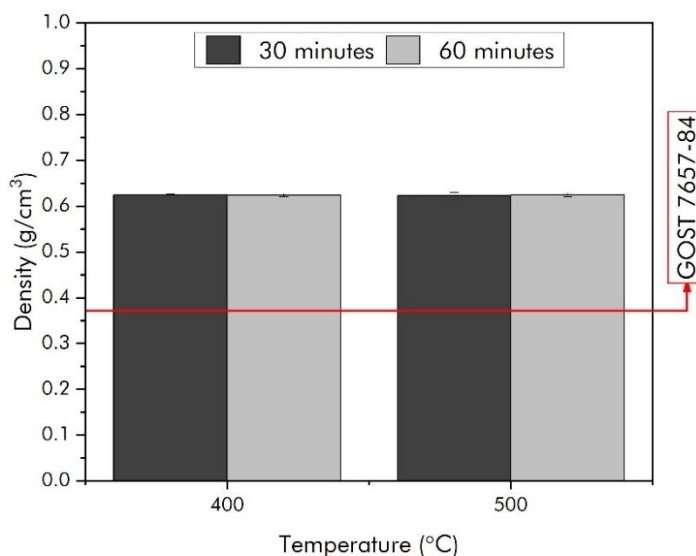


Figure 2. The density of briquettes at various pyrolysis temperatures and times

Density

Density is one of the important parameters in characterizing the wood charcoal briquettes. The wood charcoal briquettes' density affects the mechanical strength, burning time, and energy efficiency. The density of the resulting charcoal wood charcoal briquettes ranged from 0.62–0.63 g/cm³ (Figure 2). These results have met the criteria GOST 7657-84 set, with a standard density value of >0.37 g/cm³. The results of the variance analysis showed that the interaction between the pyrolysis temperature factor and the pyrolysis time factor and that each factor had no significant effect ($P \geq 0.05$) on the density value of the wood charcoal briquettes.

Several factors affect charcoal briquettes' density, including the material's specific gravity, compression pressure, moisture content of charcoal, type and proportion of adhesive material, and pyrolysis process. These results indicate that the density of calliandra charcoal briquettes (0.62–0.63 g/cm³) contributes significantly to their combustion characteristics. When compared with other woody biomass briquettes, the calliandra briquettes exhibit a relatively higher density than briquettes from Mexican hardwoods (0.37–0.50 g/cm³) (Ruiz-aquino et al., 2019) and fall within the upper range of briquettes from mangrove branch wood (0.58–0.71 g/cm³) (Tambunan et al., 2023). This is likely influenced by the inherently high basic density of calliandra wood (0.50–0.80 g/cm³) (Stewart et al., 2001) and the application of high-pressure compaction (32.88 MPa) during briquetting. According to (Huang, 2014), high-density briquettes offer better combustion efficiency because their compact structure reduces void spaces, allowing them to burn longer and release more heat. Furthermore, (Hero et al., 2024) noted that combustion rate is inversely related to density; higher-density briquettes tend to have slower, more stable burning, which is advantageous for sustained heat output. These findings underscore the superior energy-per-volume ratio and thermal stability of calliandra charcoal briquettes compared to briquettes made

from lower-density wood species. Comparable briquette densities (0.55–0.67 g/cm³) have been reported for torrefied calliandra wood pellets (Sutapa & Hidayatullah, 2023), supporting the suitability of calliandra wood for densified fuel. High briquette density also produces an enormous energy-per-volume ratio. Aljarwi et al., (2020) states that the higher the forging pressure applied, the higher the density value of the resulting charcoal briquettes. This phenomenon is due to the mechanism of binding biomass under high pressure, which causes the emergence of attractive forces between solid particles and interlocking bonds (Rahman et al., 2003).

Moisture Content

The moisture content of charcoal briquettes produced has different values. The water content value ranged from 2.14–2.73%. The analysis of variance showed that the pyrolysis temperature factor and the pyrolysis time factor had a significant effect ($P \leq 0.05$) on the moisture content of the wood charcoal briquettes. Duncan's further test showed that the moisture content value in each interaction of the pyrolysis temperature and pyrolysis time factor had a significant effect. The highest water content is owned by wood charcoal briquettes with a temperature treatment of 400°C for 30 minutes, followed by a temperature treatment of 400°C for 60 minutes. Briquettes with a temperature of 500°C for 30 minutes are not significantly different, and wood charcoal briquettes with a temperature treatment of 500°C for 60 minutes are the lowest. The moisture content of the results of this study meets the standard values required by GOST 7657-84, SNI 01-6235-2000, and EN 1860-2.

The wood charcoal briquettes produced at 400°C and 30 minutes have the highest moisture content. This result shows that at lower temperatures and pyrolysis times, the water in the wood charcoal has not entirely come out, so the water content of the wood charcoal briquettes is still high. The lower the pyrolysis temperature and time, the more difficult it is to achieve total water evaporation. This statement is in line with

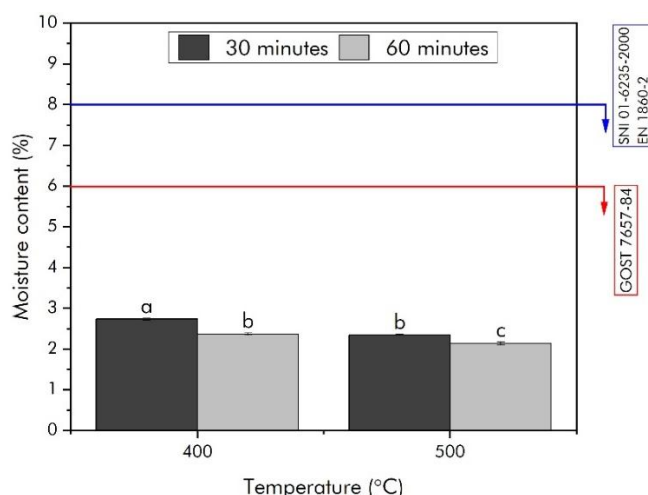


Figure 3. The moisture content of briquettes at various pyrolysis temperatures and times

Camarta et al., (2020) statement that the moisture content of wood charcoal briquettes is influenced by temperature and charring time, as well as surrounding environmental factors such as humidity. The lowest water content is owned by wood charcoal briquettes treated with a temperature of 500°C for 60 minutes. This phenomenon shows that the higher the temperature and the longer the pyrolysis time, the lower the moisture content of the resulting wood charcoal briquettes. At this temperature and time, it is assumed that the water in the charcoal has completely evaporated. Moisture values were well below the maximum limits ($\leq 8\%$) prescribed by SNI 01-6235-2000 and comparable to torrefied calliandra wood pellets, which had moisture contents around 6.40% (Suyitno et al., 2022), and pyrolysed calliandra wood at 500°C for 60 min had 6.87% moisture (Pasaribu et al., 2025). At this point, the wood charcoal briquettes in this study will have a very low moisture content, making them more efficient to use in combustion, producing less smoke, and increasing calorific value (Sutapa & Hidayatullah, 2023).

Ash content

The ash content of wood charcoal briquettes indicates the amount of inorganic material remaining unburned during the pyrolysis process. Ash content determines the quality of wood charcoal briquettes. High ash content indicates that many inorganic materials are still not burned during the pyrolysis process, which causes a decrease in combustion quality, leaves much residue, and reduces heat efficiency. On the other hand, low ash content indicates that the wood charcoal briquettes produced are of good quality because the combustion process produces little residue, more efficient heat, and less pollution.

The ash content of the results of this study ranged from 3.55–4.91%, with the highest ash content value belonging to the wood charcoal briquettes with 500°C 60 min pyrolysis treatment and the lowest ash content

belonging to the wood charcoal briquettes with 400°C 30 min pyrolysis treatment (**Figure 4**). The wood charcoal briquettes produced have ash content values following SNI 01-6235-2000 and EN 1860-2 standards but do not meet the GOST 7657-84 standard with ash content $< 6\%$. This result can be caused by several factors, including the type of raw material that has a high mineral composition, the pyrolysis process that is not optimal, and contamination of raw materials. Calliandra plants are rich in minerals such as calcium (7.10 g/kg DM), potassium (6.00 g/kg DM), magnesium (3.60 g/kg DM) and silica (Heuzé et al., 2017), which remain as ash after combustion. Raw material contamination refers to impurities in the raw material that cause the mineral content in the charcoal to be quite high and leave ash as a combustion residue. Impurities such as SiO_2 , Al_2O_3 , Fe_2O_3 , CaO , and external impurities (from the environment) can interfere with briquette combustion (Ristianingsih et al., 2015).

The results of the variance analysis showed that the interaction between the pyrolysis temperature factor and the pyrolysis time factor had no significant effect ($P \geq 0.05$) on the ash content of the wood charcoal briquettes. However, each factor had a significant effect ($P \leq 0.05$) on the ash content of the wood charcoal briquettes. Duncan's further test shows that the pyrolysis temperature factor is significantly different from other pyrolysis temperature factors, and the pyrolysis time factor is also significantly different from other pyrolysis time factors. Torrefaction studies on calliandra wood reported ash contents of 0.90%–1.68% (Sutapa & Hidayatullah, 2023), suggesting that the higher ash in this study may derive from mineral deposition during pyrolysis or contamination from the reactor. XRD analysis (**Figure 4**) confirmed the presence of calcite (CaCO_3) and traces of silica in the char, indicating that calcium oxalate crystals in calliandra wood are converted to calcite during cooling.

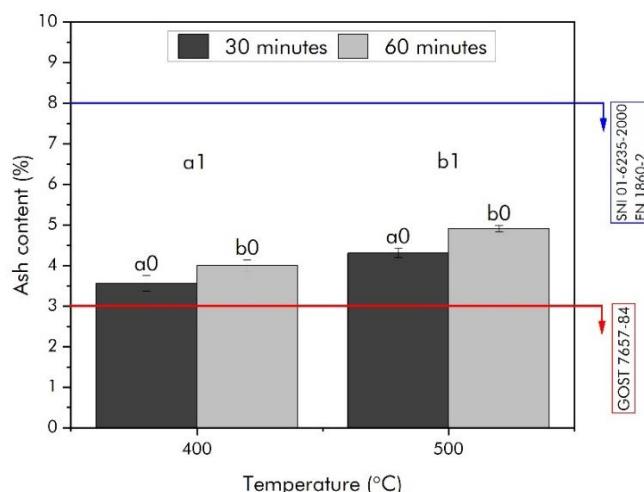


Figure 4. Ash content of briquettes at various pyrolysis temperatures and times

Volatile matter

Volatile content is the component of raw materials such as cellulose, hemicellulose, and lignin, which undergo decomposition and are released when the raw material is heated (Afrianih et al., 2022). The value of volatile matter produced ranged from 16.32–25.11%, with the highest volatile matter value in wood charcoal briquettes with pyrolysis treatment of 400°C 30 minutes and the lowest in wood charcoal briquettes with pyrolysis treatment of 500°C 60 minutes (Figure 5).

Wood charcoal briquettes in all treatments have not fulfilled the SNI 01-6235-2000 standard with a volatile matter standard of $\leq 15\%$. The high temperature and pyrolysis time resulted in more volatile matter loss. The high value of volatile matter of wood charcoal briquettes can be caused by non-carbon compounds such as H_2 , CO , CO_2 , and CH_4 as a result of incomplete decomposition (Anasthasia et al., 2020), especially hydrogen compounds that are firmly attached to carbon atoms (Alpian et al., 2020). Higher temperatures and longer residence times reduced the volatile fraction because hemicellulose and cellulose decompose and volatiles escape as gases (Yang et al., 2016). Similar challenges have been noted in other studies: torrefied calliandra wood pellets had volatile matter around 77% (Suyitno et al., 2022), and pyrolysed calliandra wood at 500°C for 60 min yielded volatile matter near 10% (Pasaribu et al., 2025). The relatively high volatile matter in our briquettes may be due to the presence of starch binder and incomplete devolatilisation at the temperatures used. Further increasing pyrolysis temperature or extending residence time could reduce volatile matter, but this would also lower yield and increase ash.

The results of the variance analysis showed that the interaction between the pyrolysis temperature factor and the pyrolysis time factor had no significant effect ($P \geq 0.05$) on the charcoal briquette volatile matter. However, each factor had a significant effect ($P \leq 0.05$) on the volatile matter value of the briquettes. Duncan's

further test shows that the pyrolysis temperature factor is significantly different from other pyrolysis temperature factors, and the pyrolysis time factor is also significantly different from other pyrolysis time factors. High temperatures during the pyrolysis process cause cellulose, hemicellulose, and lignin to degrade more easily and volatile matter to evaporate more quickly, so what remains are carbon atoms that can affect the value of fixed carbon and calorific value. The higher the value of volatile matter, the lower the fixed carbon and calorific value (Afriyenti et al., 2018). However, a high value of volatile matter can facilitate the combustion process in briquettes because some volatile matter are flammable gases (Adipratama et al., 2021).

Fixed carbon

Fixed carbon is the carbon content of briquettes resulting from the reaction of cellulose or hemicellulose during combustion (Budiawan et al., 2014). Wood charcoal briquettes with the highest carbon value were those treated with 500°C 60 min pyrolysis, and the lowest were those treated with 400°C 30 min pyrolysis, with results ranging from 68.60–76.63% (Figure 6). The overall value of fixed carbon has not been able to meet EN 1860-2 and GOST 7657-84 standards. The value of ash and volatile matter strongly influences the value of fixed carbon. The higher the value of ash and volatile matter, the smaller the value of fixed carbon, resulting in a low calorific value.

The results of the variance analysis showed that the interaction between pyrolysis temperature factors and pyrolysis time factors had no discernible effect ($P \geq 0.05$) on the carbon-bound briquettes. However, each factor had a real effect ($P \leq 0.05$) on the value of fixed carbon. Duncan's further tests showed that the pyrolysis temperature factor significantly differed from other pyrolysis temperature factors, and the pyrolysis time factor also significantly differed from other pyrolysis time factors.

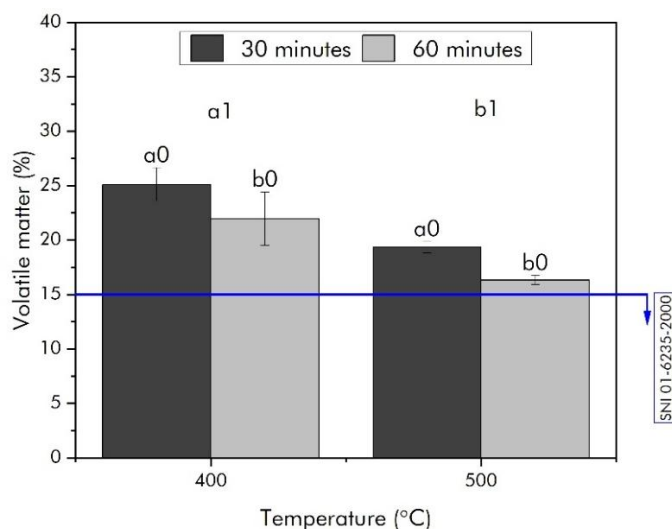


Figure 5. Volatile matter of briquettes at various pyrolysis temperatures and times

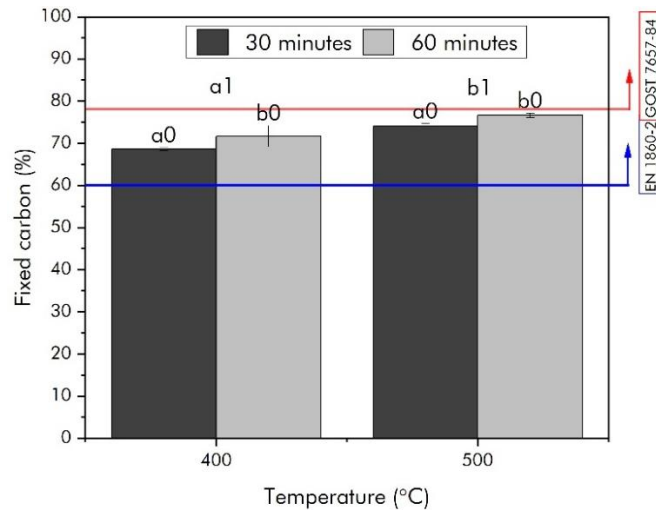


Figure 6. fixed carbon of briquettes at various pyrolysis temperatures and times

Charcoal briquettes with high fixed carbon content tend to be more porous and hygroscopic, which allows them to absorb moisture from the surrounding environment. Upon moisture absorption, the charcoal briquettes may swell, and as they dry, they can shrink and lose structural integrity, leading to slight disintegration or fragmentation. This phenomenon can cause the appearance of ‘shrinking’ or reduction in size. Furthermore, while the high density of calliandra wood (ranging from 0.50 to 0.80 g/cm³) contributes to high calorific value and combustion stability, it is not the primary reason for rapid burning. Rather, the relatively low moisture content and open pore structure facilitate oxygen diffusion, enabling efficient ignition and combustion. Thus, the physical change observed in the briquettes is a combined effect of moisture dynamics and structural response rather than density alone (Arifin & Noor, 2016; Simpson & Tenwolde, 1999; White & Diertenberger, 2010).

Calorific Value

Calorific value is an essential test parameter in determining wood charcoal briquette’s quality as a fuel. Wood charcoal briquettes with a high calorific value indicate that the briquettes are of good quality

because the higher the calorific value, the higher the heat produced (Ngene et al., 2024). The results showed that the calorific value ranged from 7,856.01–7,978.48 cal/g. The wood charcoal briquettes that had the highest calorific value were wood charcoal briquettes with pyrolysis treatment at 500°C for 60 minutes, and the lowest were wood charcoal briquettes with pyrolysis treatment at 400°C for 30 minutes (Figure 7). The study results produced wood charcoal briquettes that have met the SNI 01-6235-2000 standard, which is >5000 cal/g in all treatments. This result happens because the calorific value is greatly influenced by other parameters such as density (Figure 2), moisture content (Figure 3), ash content (Figure 4), volatile matter (Figure 5), and bonded carbon (Figure 6). The higher the temperature and pyrolysis time, the higher the calorific value. This phenomenon is suspected to happen because, with a high temperature and a long time, perfect carbonization will occur so that it can produce briquettes with lower moisture content and volatile matter (Ngene et al., 2024). However, if the temperature and time are too high, it can produce high ash levels.

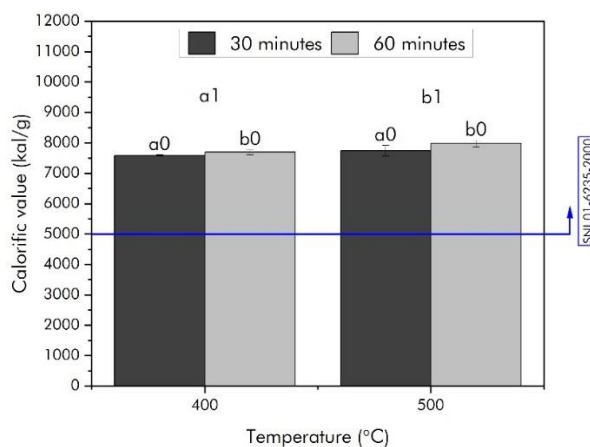


Figure 7. Calorific value of calliandra wood charcoal wood charcoal briquettes at various variations in pyrolysis temperature and time; 0:pyrolysis temperature variation factor; 1:Pyrolysis time variation factor; The difference in letters shows a real influence

The analysis of variance (ANOVA) showed that the interaction between pyrolysis temperature factors and pyrolysis time factors had no discernible effect ($P \geq 0.05$) on the calorific value of the briquettes. However, each factor had a real effect ($P \leq 0.05$) on the calorific value of wood charcoal briquettes. Duncan's further tests showed that the pyrolysis temperature factor significantly differed from other pyrolysis temperature factors, and the pyrolysis time factor also significantly differed from other pyrolysis time factors. The increase in temperature and pyrolysis time in this study was proven to increase the calorific value of wood charcoal briquettes, and this is suspected to occur along with the increase in the value of wood charcoal briquette's fixed carbon. This phenomenon can be attributed to the rise in internal reaction temperature, which lowers the volatile matter and moisture content, ultimately increasing the fixed carbon content (Hasibuan et al., 2024).

The calorific values of the calliandra briquettes in this study, which ranged from 7,856 to 7,978 cal g⁻¹, are notably higher than those previously reported for calliandra biochar produced through slow pyrolysis at 500°C (7,145 cal/g) (Pasaribu et al., 2025). Additionally, the energy values recorded here are comparable to the upper limit of those found in charcoal from Mexican hardwoods, which range from 29.38 to 32.11 MJ/kg (7,022–7,678 cal/g) (Ruiz-aquino et al., 2019). These comparisons suggest that calliandra briquettes not only meet but exceed the energy performance of many established wood-based briquettes. The improvements in calorific value at higher pyrolysis temperatures, particularly under the 500°C for 60 min condition, are consistent with the increased degree of carbonisation observed in the FTIR and XRD results. Therefore, calliandra briquettes can be considered a competitive renewable energy source in terms of thermal performance and stability. However, as pyrolysis temperature and time increase, the reduction in volatile matter outweighs the corresponding increase in ash content. Further

emphasize that ash content and its mineral composition significantly influence pyrolysis behavior (Puri et al., 2024). Excessive ash not only diminishes energy density but may also contribute to operational challenges such as slagging and fouling during combustion.

Combustion rate

The results showed that the wood charcoal briquettes that had the highest combustion rate were wood charcoal briquettes with pyrolysis treatment of 400°C for 30 minutes, and briquettes with the lowest combustion rate were wood charcoal briquettes with treatment of 500°C for 60 minutes, with results ranging from 0.08–0.09 g/min (Figure 8). A higher combustion rate indicates better quality in wood charcoal briquettes, reflecting a faster burning time and enhanced performance during combustion. This is supported by Febriani, who states that wood charcoal briquettes with a reasonable combustion rate (0.08–0.10 g/min) will burn easily with a relatively fast ignition time and quickly burn out (Febriani et al., 2022). The combustion performance of calliandra briquettes is influenced not only by their chemical composition but also by their physical structure. In particular, their relatively high density contributes to a steady and sustained burn, allowing for consistent heat release over time. This behaviour aligns with findings from previous studies, which show that denser briquettes tend to burn more slowly and efficiently due to reduced porosity and limited oxygen diffusion (Hero et al., 2024; Huang, 2014). Such combustion characteristics are advantageous for applications requiring prolonged thermal output and fuel efficiency.

The results of the statistical analysis showed that the interaction between pyrolysis temperature factors and pyrolysis time factors and each factor had no discernible effect ($P \geq 0.05$) on the combustion rate value of wood charcoal briquettes. Duncan's further test showed that the combustion rate values of all factors and all treatments had no discernible effect.

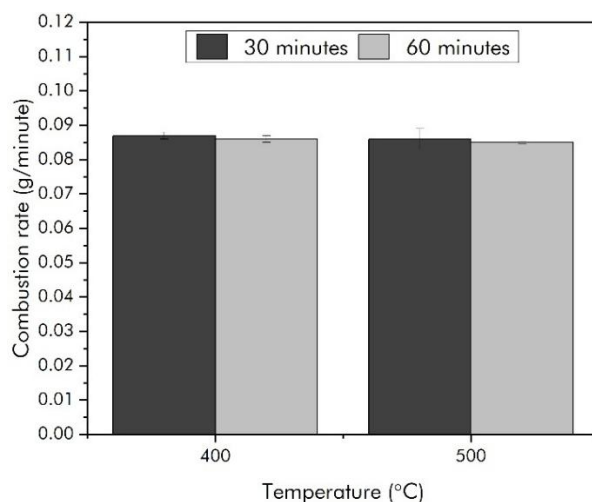


Figure 8. Calliandra wood charcoal briquettes combustion rate at various variations in pyrolysis temperature and time

Considerable parameters affecting the combustion rate are the density and compression pressure. Increasing the density and compression pressure of wood charcoal briquettes can increase the density of wood charcoal briquettes to cause a reduction in the air gap between the particles. In addition, ash content can also affect the rate of combustion. High ash content can lead to low heat transfer inside the briquettes (Aziz et al., 2019).

Fourier Transform Infrared Spectroscopy (FTIR)

FTIR spectra of calliandra wood charcoals pyrolyzed at 400°C and 500°C for 30 min and 60 min are presented in **Figure 9** and the assignment of functional groups and structural polymers identified are presented in **Table 1**. The spectrum of calliandra wood charcoals showed some characteristic absorption bands associated with functional groups derived from lignocellulosic materials. Broad bands around 3728 cm⁻¹ and 3634 cm⁻¹ were observed, which correspond to the O–H stretching vibration, which is usually associated with hydroxyl groups in cellulose, hemicellulose, or water content. CH₂ groups in wood polymers were observed by bands at 2926 cm⁻¹, 2865 cm⁻¹, 1459 cm⁻¹, and 1368 cm⁻¹. The C=O stretching vibration of the ester bond was indicated in peak at 1735 cm⁻¹. The band at 1648 cm⁻¹ assigned to the C=C and C=O stretching vibrations of the aromatic ring, while the peak at 1510 cm⁻¹ indicates the C=C ring stretching vibration of lignin. The aromatic CO and phenolic OH stretching vibrations were reflected by the band at 1243 cm⁻¹. The band at 1037 cm⁻¹ was assigned to the undecomposed cellulosic and ligneous (C–O stretching).

All wood charcoal samples have shown chemical changes. The FTIR spectrum showed a significant decrease in all bands and peaks that were previously present in calliandra wood. The bands indicating OH groups (3728 cm⁻¹ and 3634 cm⁻¹) decreased drastically but remained present until pyrolysis treatment of 600°C for 60 min. The gradual decrease

in the intensity of these bands with increasing temperature and duration of pyrolysis indicates the breakdown of polar compounds and loss of bound water. This observation is consistent with previous findings on biochar from various biomasses (Chen et al., 2008; Domingues et al., 2017; Jindo et al., 2014; Keiluweit et al., 2010). The presence of OH groups in all wood charcoals may also be influenced by the OH groups from starch adhesives (Deeyai et al., 2013). Absorption at 2926 cm⁻¹ and 2865 cm⁻¹ (C–H stretching) showed a decrease but remained present indicating a heat-resistant aliphatic structure, while the peak at 1368 cm⁻¹ began to absent in wood charcoals produced by pyrolysis temperature of 500. The band reflecting undecomposed cellulosic and ligneous showed a decreased intensity and shifted from 1037 cm⁻¹ to 1055 cm⁻¹. This decrease is due to thermal damage to cellulose, leaving the aromatic core originating from the lignin fraction exposed to the pyrolysis products. The intensity of the bands appeared lower in wood charcoal with longer pyrolysis duration indicating further cellulose degradation. The others peaks have lost intensity since the mildest pyrolysis treatment.

Besides the decreasing intensity of some bands and the disappearance of peaks from calliandra wood, the spectrum of wood charcoals showed several bands and peaks that appeared and strengthened. The presence of a prominent band at 1648 cm⁻¹ and 1576 cm⁻¹ is attributed to the C=O and C=C stretching vibrations suggesting the formation of anhydrides, ketones, esters, and carbon as well as aromatic components such as lignin. This band remained relatively stable at higher temperatures, suggesting that lignin possesses greater thermal resistance compared to polysaccharides. The persistence of aromatic signals also confirms partial aromatization or carbonization during pyrolysis, aligning with FTIR results of biocoal fuel derived from palm oil biomass (Gani et al., 2024). Confirmation of the presence of aromatic carbon is also shown by the appearance of bands at 875 cm⁻¹, 813 cm⁻¹, and 752 cm⁻¹, which

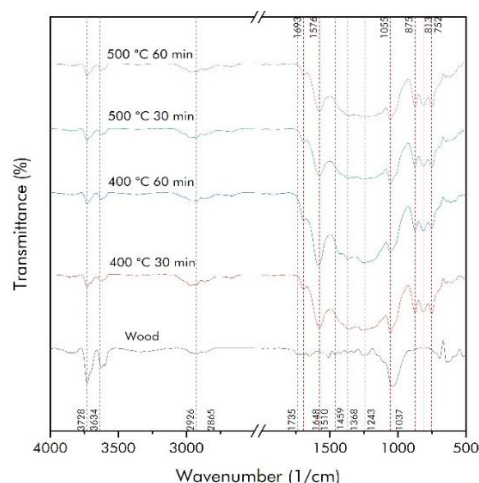


Figure 9. FTIR spectra of the bio-briquette samples

correspond to out-of-plane deformation of aromatic C–H bonds (Guo & Bustin, 1998; Lin-Vien et al., 1991). The signals detected near these bands indicate the possibility of O substitution in furan-like and quinone-like structures (van der Marel & Beutelspacher, 1976). Overall, the FTIR analysis revealed that higher pyrolysis temperatures and longer residence times resulted in reduced hydrophilic functional groups and enhanced aromatic carbon structures. These changes improve the stability and energy characteristics of the resulting wood charcoals.

X-ray diffraction (XRD)

X-ray diffraction (XRD) analysis was conducted to determine the structural changes in the raw material

of calliandra wood and the wood charcoals produced with various temperatures and durations of pyrolysis. Calliandra wood showed a shoulder at around 15.60° and broad diffraction peaks at 22.90° and 35.10° , which are characteristic of semicrystalline cellulose (Figure 10). These peaks reflect the ordered cellulose chain arrangement interspersed with amorphous regions (French, 2014). In the pyrolysis products even at the low temperature and duration, the intensity associated with cellulose decreased drastically. This indicates a decrease in cellulose crystallinity and degradation of cellulose into an amorphous material which is also related to the decrease in the band at 1037 cm^{-1} in the FTIR.

Table 1. Functional group assignments and structural polymers identified from FTIR spectra of wood charcoal samples

Wave Number (cm^{-1})	Peak interpretation	Structural polymer	References
3728, 3664	Free OH stretching	Lignocellulosic polymer (cellulose and hemicellulose), polysaccharides (starch)	(Chen et al., 2008; Deeyai et al., 2013)
2926	Asymmetric C–H stretching	Lignocellulosic polymer	(Keiluweit et al., 2010)
2865	Symmetric C–H stretching	Lignocellulosic polymer	(Keiluweit et al., 2010)
1735	C=O stretching	Lignocellulosic polymer	(Chen et al., 2008)
1693, 1648, 1576	C=C, C=O stretching	Lignocellulosic polymer Lignin aromatic structure	(Chen et al., 2008; Chun et al., 2004; Keiluweit et al., 2010)
1510	C=C stretching	Lignin aromatic structure	(Chen et al., 2008)
1459, 1368	C–H ₂ bending	Lignocellulosic polymer	(Chen et al., 2008)
1243	CO–, OH– stretching	Lignin aromatic structure	(Chun et al., 2004)
1037, 1055	C–O stretching	Lignocellulosic polymer	(Keiluweit et al., 2010)
875, 813, 752	C–H bending	Lignin aromatic structure	(Keiluweit et al., 2010)

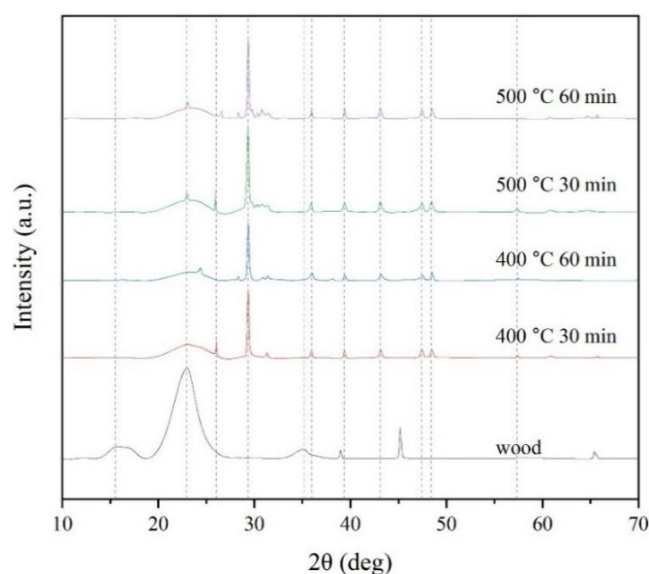


Figure 10. X-ray diffraction of the calliandra wood charcoal samples

Several peaks found in wood charcoal can be assigned to calcite such as at 35.90°, 39.40°, 43.10°, 47.40°, 48.40°, 57.40° and the main peaks at 29.40° (Xu & Poduska, 2014). At higher temperatures the similarity to calcite is stronger with the presence of a peak at 23.10° (Tintner et al., 2018). The increase in intensity at 29.40° indicates that the calcite content in wood charcoals increases with increasing pyrolysis temperature as also found in biochar in previous studies (Tintner et al., 2018; Yuan et al., 2011). The formation of calcite may be due to the reaction between CO₂ trapped in the pores for carbonation with CaO (Smidt et al., 2010) after cooling. CaO itself can be formed from calcium oxalate residues which are crystals that commonly form in various types of plants (Franceschi & Nakata, 2005).

The variation in pyrolysis duration also shows differences, especially the emergence of peak at 26.10° which is only found in wood charcoals with a pyrolysis duration of 30 minutes. The peak is likely related to SiO₂ which is also reported to be found in peanut straw biochar (Yuan et al., 2011). Further pyrolysis processes up to 60 minutes likely produce different reactions so that the peak at 26.10° were absent. The difference in crystal structure formed in this wood charcoals may also be related to differences in the characteristics of the main parameters of the briquette such as ash content, volatile matter, and calorific value due to differences in temperature and pyrolysis duration as discussed above.

CONCLUSIONS

This study confirmed that variations in pyrolysis temperature and residence time significantly influenced the quality of calliandra wood charcoal briquettes, as assessed by key parameters such as density, moisture content, ash content, volatile matter, fixed carbon, calorific value, and combustion rate. The best overall performance was obtained at 400°C for 30 minutes, which produced briquettes with high density (0.63 g/cm³), low moisture content (2.30%), moderate ash (4%), and high calorific value (7,900 cal/g), aligning closely with national (SNI 01-6235-2000) and international (EN 1860-2, GOST 7657-84) quality standards.

Although the volatile matter content (20%) slightly exceeded the standard threshold, other parameters met or exceeded the minimum requirements for high-quality fuel briquettes. Treatments at 500°C improved carbonisation and energy content but resulted in lower yield and increased ash. These trends were supported by FTIR and XRD analyses, which showed greater aromatisation and reduced cellulose crystallinity at higher temperatures.

In conclusion, the pyrolysis conditions tested in this study produced calliandra charcoal briquettes that largely conform to established quality standards. With minor adjustments, such as improved volatile matter control, these briquettes demonstrate strong potential

as efficient, standard-compliant renewable solid fuels. Further research is recommended to explore broader ranges of pyrolysis conditions, alternative binders, and the effects of pretreatment techniques on charcoal briquette quality.

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