

Effect of residence time on the torrefaction characteristics of Calliandra wood as solid biofuel

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Abstract

Increasing energy demand and the environmental impact of fossil fuel use have prompted the search for alternative and more sustainable energy sources. Calliandra wood (*Calliandra Calothyrsus*) as a renewable solid fuel has relatively high heating value and rapid growth. This study aims to examine the effect of residence time variation in the torrefaction process on the characteristics of Calliandra wood chips. This research was conducted experimentally using a tubular-type continuous torrefaction reactor at two temperatures, namely 275 and 300°C, with three residence time variations: 10, 20, and 30 minutes. The main parameters analyzed included calorific value, proximate and ultimate composition, mass yield, and energy yield. The results showed that the longer the residence time, the heating value and carbon content continued to increase, but there was a decrease in mass and energy yield. At 300°C with a residence time of 30 minutes, the highest heating value was obtained at 6,399 kcal/kg, but the mass yield remained at 35%. In contrast, at 275°C with a residence time of 10 minutes, the heating value increased to 4,632 kcal/kg with a mass yield of 83%. Optimal conditions were obtained at 275°C with a residence time of 20 minutes, resulting in a heating value of 5,387 kcal/kg with a mass yield of 70%, which balanced the energy efficiency and the amount of biomass retained. Although the torrefaction process was shown to improve the quality of biomass as a solid fuel and the longer residence time resulted in higher energy, there was a trade-off in the form of reduced biomass mass. Torrefaction also makes the biomass more hydrophobic and resistant to biological damage. The results of this study are expected to be a reference for the development of torrefaction technology to support the utilization of renewable energy in Indonesia.

Keywords:

Biomass, torrefaction, solid fuel, Calliandra, residence time

1 Introduction

Energy is a major necessity that continues to increase as the human population and activities grow. Global dependence on fossil fuels has led to various challenges, including resource limitations, price volatility, and significant environmental impacts such as greenhouse gas emissions. Therefore, renewable energy has become a major focus in researching and developing alternative energy resources. Biomass has been identified as one of the renewable energy sources that has great potential due to its abundant availability and lower carbon emission characteristics compared to fossil fuels [1].

Various types of biomass waste, such as agricultural residues, forest waste, and municipal waste, have been investigated to improve

the efficiency and sustainability of their use as energy sources [2]. One type of biomass that has great potential is *Calliandra calothyrsus* wood. This wood is known for its high calorific value as well as its ability to grow on marginal lands without disrupting food production [3].

Research on biomass torrefaction has been conducted in recent years to improve the quality of biomass fuels. The effects of temperature and residence time variations on the characteristics of torrefaction products from different types of biomass have been studied, and it was found that the optimal temperature ranges from 250-300°C for increased heating value and reduction of volatile content [4][5]. Thermochemical reactions during the torrefaction process have been analyzed, showing that longer residence times can increase fixed carbon and reduce oxygen content in biomass [6]. In addition, the impact of torrefaction on combustion and gasification characteristics has been investigated, and the results show that biomass torrefied at higher temperatures has better performance in combustion and energy conversion systems [7]. The change of lignocellulose structure during the torrefaction process has also been studied, and it was found that the decomposition of hemicellulose and lignin contributed to the improvement of biomass thermal stability [8]. In addition, adjustment of residence time parameters has been found to result in significant changes in biomass fuel characteristics [9]. Similar results were reported, showing that torrefaction processes that take place at high temperatures with optimal dwell time can significantly improve the quality of biomass fuel [10].

Although many studies have been conducted on biomass torrefaction, there are still some research gaps that need to be addressed. One of the main gaps is the lack of specific studies on the effect of residence time on the characteristics of Calliandra wood torrefaction. Most of the existing studies have focused on temperature variation without in-depth exploration of the interaction of residence time with changes in biomass characteristics. In addition, although some studies have addressed the aspects of thermochemical reactivity and chemical composition changes during torrefaction, further studies are still needed regarding the influence of these parameters on energy efficiency and overall process optimization. Therefore, this study was designed to fill the gap by analyzing the effect of variation in residence time at 275°C and 300°C to determine the optimal conditions for improving the calorific value and thermal stability of Calliandra Wood.

This study aims to investigate the effect of residence time variation on the torrefaction process to improve the quality of Calliandra woody biomass as fuel. This study includes the analysis of calorific value, proximate and ultimate composition, and hydrophobic properties of torrefaction products. The results of this study are expected to contribute significantly to the development of biomass conversion technology, especially in optimizing torrefaction parameters to improve the quality of biomass-based fuels. In addition, a deeper understanding of the relationship between residence time and characteristics of the biomass produced will be an important reference for the biomass and renewable energy industries. Thus, this research has the potential to support the diversification of renewable energy sources in Indonesia, reduce dependence on fossil fuels, and sustainably contribute to carbon emission mitigation.

2 Methods

2.1 Material preparation

Calliandra wood used as a torrefaction sample material is waste or residual material that is no longer used by an industry. The Calliandra wood used was chip-shaped with a non-uniform size between 2-4 cm. Calliandra wood used for torrefaction samples in dry conditions, and Calliandra wood to be used is prepared by sorting the samples to be used, the selected Calliandra wood is stored in a 1 kg ziplock bag, and a total of 6 kg is needed for all variations of temperatures and residence time. The use of zip plastic to store

Calliandra wood samples is so as not to affect the moisture content contained in Calliandra wood due to environmental air. Examples of Calliandra wood chips used are in Fig. 1.



Fig. 1. Calliandra wood chip

2.2 Torrefaction process

The torrefaction process is carried out using a tubular-type continuous reactor with an oil jacket heating system. The fuel used as a combustion source is LPG. Fig. 2 shows the arrangement of torrefaction equipment consisting of a temperature recorder and thermocouple, electromotor, gear, water pump, torrefaction sample container, water reservoir, LPG, cooling char, and combustion chamber. Previously, the temperature recorder was calibrated by inserting the sensor connected to boiling water and ice water to ensure the temperature was appropriate. The torrefaction process was carried out under very low oxygen conditions at 275°C and 300°C with residence time variations of 10, 20, and 30 minutes. Each variation used as much as 1 kg of Calliandra wood chips, with a total of 6 kg of Calliandra wood chips. A total of 1 kg of sample is slowly inserted into the reactor through the feeding hopper, and we have set the reactor temperature as desired and waited for the selected time variation. After the torrefaction process is complete, all Calliandra wood chip samples are put into plastic containers with a zip.

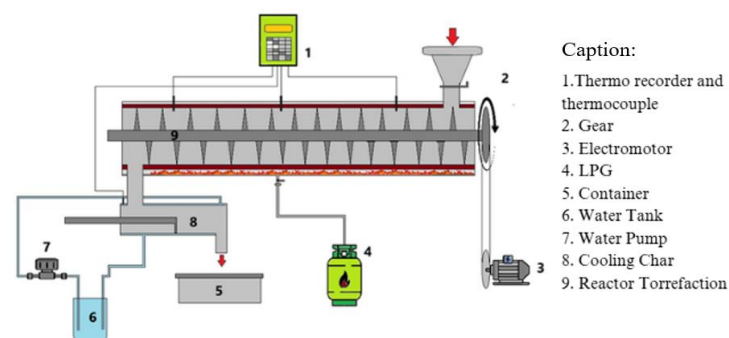


Fig. 2. Reactor setup for the torrefaction process [11]

2.3 Analysis method

2.3.1 Visual of the torrefaction product

After the torrefaction process is carried out, the first analysis carried out is to see the visual changes that occur in the torrefaction biomass samples for each variation carried out. Descriptions of the samples can be seen in Table 1.

Table 1. Sample ID

| No. | Sample ID | Description |
|-----|-----------|--|
| 1. | WCC | Calliandra Raw |
| 2. | WCC1-10 | Calliandra Torrefaction at 275°C, 10 minutes |
| 3. | WCC1-20 | Calliandra Torrefaction at 275°C, 20 minutes |
| 4. | WCC1-30 | Calliandra Torrefaction at 275°C, 30 minutes |
| 5. | WCC2-10 | Calliandra Torrefaction at 300°C, 10 minutes |
| 6. | WCC2-20 | Calliandra Torrefaction at 300°C, 20 minutes |
| 7. | WCC2-30 | Calliandra Torrefaction at 300°C, 30 minutes |

2.3.2 Calorific value

Calorific value is an important parameter to determine the combustion capability of a fuel. The calorific value of a fuel refers to the amount of heat generated from the combustion of a solid fuel [12]. Calorific value testing was conducted using the Oxygen Bomb Calorimeter BK-1A+ (Fig. 3) by ASTM D5865 standards and calibrated using benzoic acid based on ISO/TR 12148.



Fig. 3. Bomb calorimeter

2.3.3 Mass yield & energy yield

Mass yield is measured based on the amount of solids remaining after the torrefaction process, expressed as the ratio of the initial biomass mass to the final product mass. Mass yield is calculated based on Eq. (1). Meanwhile, energy yield describes the proportion of original energy that remains in the biomass after torrefaction. Energy yield can be calculated based on Eq. (2).

$$\text{Mass yield} = \frac{m_{tor}}{m_{raw}} \times 100\% \quad (1)$$

$$\text{Energy yield} = \text{Mass yield} \times \frac{HHV_{tor}}{HHV_{raw}} \quad (2)$$

Where m_{tor} is the mass of torrefied biomass, m_{raw} is the mass of original biomass, HHV_{tor} is higher heating value of torrefied biomass, and HHV_{raw} is the higher heating value of original biomass.

2.3.4 Proximate analysis

Proximate analysis was carried out on raw materials and products from the torrefaction process, carried out to determine the constituent components of solid fuels such as Moisture Content / MC (SNI 1683: 2021 point 7.1), Ash Content ASTM D3174-12 (2018) ^{el}, Volatile Matter / VM (SNI 1683: 2021 point 7.3), and Fixed Carbon / FC (By. Calculation). Proximate testing was carried out at the Bandar Lampung Industrial Standardization and Service Center.

2.3.5 Ultimate analysis

Ultimate analysis is a method used to determine the elemental content of fuels, such as carbon (C), hydrogen (H), oxygen (O), nitrogen (N), and sulfur (S). Information from this analysis is critical to understanding the characteristics of the fuel, including its heating value and impact on the environment [13]. Ultimate testing was conducted at the TekMIRA Mineral and Coal Testing Center Laboratory. Using ASTM D5373-21 (Method A) testing standards for Carbon (C), Hydrogen (H) and Nitrogen (N), ASTM D3176-24 for Oxygen (O), and ASTM D4239-18^{(e)(1)} (Method A) for Sulfur (S).

2.3.6 Hydrophobicity analysis

The hydrophobicity test is a method used to see the ability to absorb water in biomass samples after the torrefaction process. The lower the ability of the sample to absorb water, the better its hydrophobicity. The test was conducted by weighing the dry sample before immersion in water at room temperature for 30 minutes, 1 hour, and 2 hours. After immersion, the sample is drained to remove surface water, then weighed again to determine the mass change due

to water absorption. The percentage of water absorption can be calculated based on Eq. (3).

$$\text{Water absorption percentage} = \frac{\text{final mass} - \text{initial mass}}{\text{initial mass}} \quad (3)$$

3 Results and discussion

Torrefaction significantly changes the characteristics of Calliandra wood chips. After being heated in this process, the moisture content in the biomass is reduced, thus increasing its energy value and making it more resistant to moisture. In addition, the percentage of carbon content in the wood increases, while volatile substances decrease, making it more stable when burned. The color

of the wood also becomes darker, indicating that some compounds in the wood, such as hemicellulose, begin to break down. These changes indicate that torrefaction can improve the quality of Calliandra wood chips as fuel.

The quality of Calliandra wood chips after torrefaction can be seen based on the calorific value analysis, proximate analysis, and ultimate analysis shown in Table 2. Samples that were not tested for proximate and ultimate were only WCC1-10 and WCC2-10 because it was considered that the calorific value produced was still low; therefore, only samples with a high calorific value were tested for proximate and ultimate. This analysis shows the quality contained in the biomass after torrefaction. Then, based on this analysis, it can be seen whether Calliandra wood chips are equivalent to coal.

Table 2. Result analysis

| No. | Parameters | Unit | Test Method | Analysis Result | | | | |
|-----|-----------------------|---------|--------------------|-----------------|--------------------|--------------------|--------------------|--------------------|
| | | | | WCC | WCC1-20 (T. 275°C) | WCC1-30 (T. 275°C) | WCC2-20 (T. 300°C) | WCC2-30 (T. 300°C) |
| 1. | Moisture Content | % ar | SNI 1683: item 7.1 | 14.17 | 5.91 | 4.98 | 5.44 | 4.42 |
| 2. | Ash | % adb | SNI 1683: item 7.2 | 1.08 | 2.31 | 1.87 | 2.27 | 3.56 |
| 3. | Volatile Matter | % adb | SNI 1683: item 7.3 | 67.72 | 66.51 | 62.32 | 50.24 | 42.34 |
| 4. | Fixed Carbon | % adb | By. Calculation | 17.48 | 25.26 | 30.83 | 42.06 | 49.68 |
| 5. | Carbon | % adb | ASTM D5373-21 | 43.65 | 50.45 | 56.17 | 57.95 | 66.39 |
| 6. | Hydrogen | % adb | ASTM D5373-21 | 6.78 | 6.10 | 6.08 | 5.68 | 5.36 |
| 7. | Nitrogen | % adb | ASTM D5373-21 | 0.61 | 0.63 | 0.7 | 0.67 | 0.87 |
| 8. | Sulfure) | % adb | ASTM D4239-18 | 0.074 | 0.028 | 0.036 | 0.025 | 0.032 |
| 9. | Oxigen | % adb | ASTM D3176-24 | 47.81 | 41.07 | 35.14 | 33.9 | 23.79 |
| 10. | Gross Calorific Value | % adb | ASTM D5865 | 4030 | 5387 | 5304 | 5815 | 6399 |
| 11. | Mass Yield | kcal/kg | | 100% | 70% | 61% | 53% | 35% |
| 12. | Energy Yield | % | | 100% | 88% | 76% | 72% | 53% |

3.1 Visual torrefaction product

Through the torrefaction process, Calliandra wood chip biomass undergoes visual changes. Images of visual changes in Calliandra wood chips can be seen in Fig. 4. Calliandra wood chip samples after going through the torrefaction process experienced a darker color change, namely brown to black, depending on how high the

temperature is and how long the residence time is applied, which is visually similar to coal. The visual of the WCC1-10 sample still has a color like the WCC sample, but the higher the temperature and the longer the residence time the visual of the Calliandra wood chip sample will be more black like the WCC2-30 sample.



Fig. 4. Visual torrefaction result of Calliandra wood chips

3.2 Calorific value of torrefaction product

Calorific value is an important parameter to measure how good a fuel is. The torrefaction process can increase the calorific value of biomass due to the heating that occurs. The calorific value of Calliandra wood chip samples for all variations used in this study can be seen in Fig. 5. Calorific value testing of Calliandra wood chip torrefaction products with all variations has a calorific value that varies between 4632 kcal/kg to 6399 kcal/kg.

The calorific value of the raw sample is 4263 kcal/kg. When the sample went through the torrefaction process at 275°C with a residence time of 10 minutes, the heating value increased to 4632 kcal/kg, which is equivalent to sub-bituminous C coal. This increase is due to the reduction of moisture content and the beginning of hemicellulose decomposition. At a temperature of 275 °C, the

calorific value increased until 20 minutes (peak 5387 kcal/kg). After 20 minutes, there was a decrease in the calorific value to 5304 kcal/kg at 30 minutes. This indicates that over-torrefaction occurs at too long a duration because the volatile components have been decomposed significantly, while the increase in fixed carbon is not significant. Longer times cause a decrease in calorific value due to the loss of volatile substances without an equivalent increase in energy. While at a temperature of 300 °C, the calorific value continues to increase from the beginning to 30 minutes, reaching 6399 kcal/kg. The torrefaction process is more active and efficient in enriching fixed carbon content through devolatilization of lignin. There is no over-torrefaction until 30 minutes because high temperatures encourage deeper carbonization reactions and greater energy.

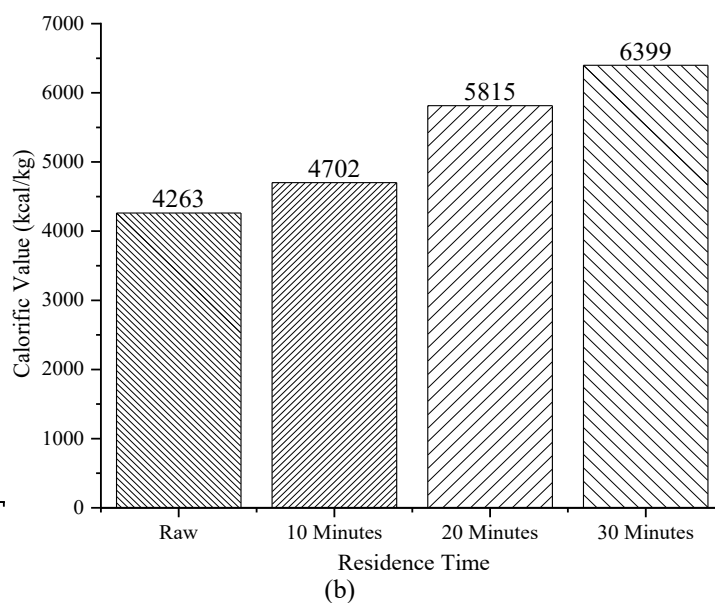
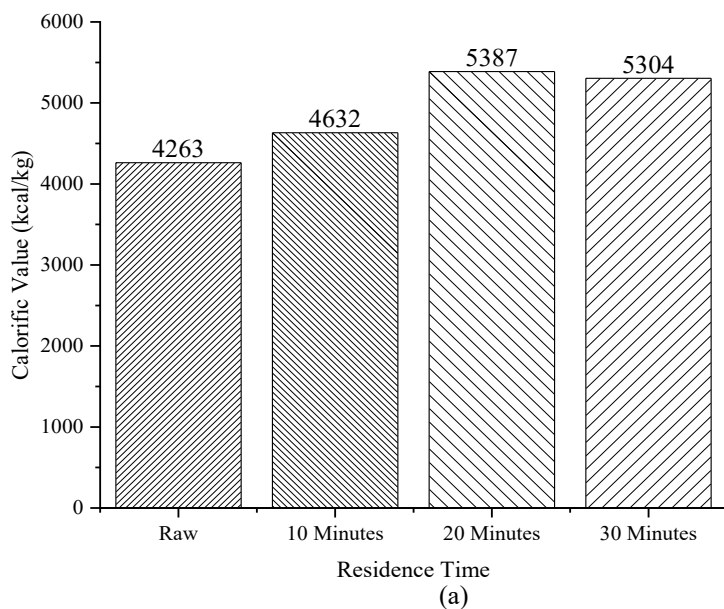


Fig. 5. Calorific value of the torrefaction product of Calliandra wood chips. (a) at 275°C; (b) at 300°C

Thus, higher temperatures (300°C) promote a more efficient and sustained torrefaction reaction for up to 30 minutes, while at 275°C, longer residence times result in energy loss due to excessive decomposition without a commensurate increase in carbon. Temperature 275°C, residence time 20 minutes, provides the best compromise between calorific value, mass yield, and energy yield, so it is ideal for industrial process efficiency. At a temperature of 300°C, a residence time of 30 minutes produces the material with the highest calorific value, suitable for applications with high energy requirements, although the total efficiency (energy yield) is low. This difference shows that high temperature (>275°C) promotes further carbonization reaction, but with the consequence of a significant decrease in mass yield.

Furthermore, the calorific value of samples WCC1-20 and WCC1-30 is equivalent to sub-bituminous B coal. Then for the calorific value in samples WCC2-10 and WCC2, 20 amounting to 4702 kcal/kg and 5815 kcal/kg, which are equivalent to sub-bituminous coal C and B. The highest calorific value is in sample WCC2-30, where the residence time is 30 minutes and the temperature is 300°C. The calorific value obtained is 6399 kcal/kg, which is equivalent to high volatile bituminous coal C. This is due to the decomposition of components such as hemicellulose, which decomposes most easily, followed by cellulose, and lignin, which decompose at higher temperatures and longer times [14].

3.3 Mass yield and energy yield

Torrefaction causes components present in the biomass, such as hemicellulose, cellulose, and lignin, to decompose. The decomposition of these chemical components causes a decrease in product mass and total energy contained. The higher the temperature and residence time in the torrefaction process, the more significant the mass decrease [6]. WCC1-10 mass yield was obtained at 83%, and energy yield at 90%. The decrease in mass yield was caused by the decomposition of hemicellulose, which began to release volatile compounds such as water and carbon dioxide. However, most of the cellulose and lignin were still intact, so the energy stored in the product remained high. This process produced a product with a fairly large mass. In WCC1-20, the mass yield decreased to 70%, and the energy yield decreased slightly to 88%. The mass yield decreased more significantly due to further decomposition of hemicellulose, while cellulose began to experience initial damage. The fixed carbon content in biomass increased, which gave it a higher calorific value. In WCC1-30, the mass yield was obtained at 61%, and the energy yield decreased to 76%, as can be seen in Fig. 6.

When the temperature was increased to 300°C with a residence time of 10 minutes (WCC2-10), the mass yield was 82% while the energy yield was 90%. At this temperature, hemicellulose was

almost completely decomposed, while cellulose began to decompose. This process produces products with high energy values despite the reduction in mass.

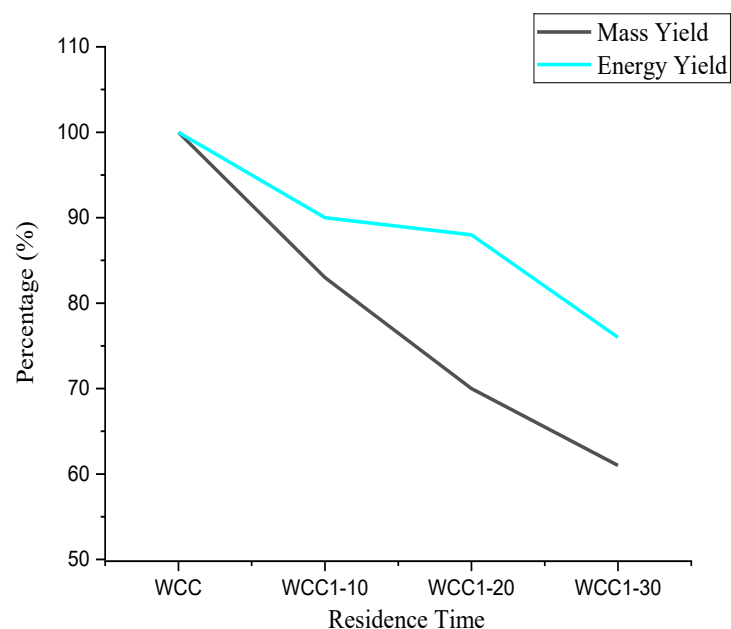


Fig. 6. Graph of mass yield and energy yield at a temperature of 275°C

When the residence time was extended for 20 minutes (WCC2-20), the mass yield was 53%, and the energy yield decreased to 72%. Under these conditions, the decomposition of hemicellulose and cellulose was more intensive, and lignin began to partially decompose. The decrease in the total energy contained in the product indicates that most of the volatile compounds have been released. Furthermore, at WCC2-30, the mass yield obtained reached its lowest point of 35%, and the energy yield only reached 53%, as can be seen in Fig. 7. At this stage, lignin decomposition became dominant, causing the release of volatile compounds in large quantities. The resulting torrefaction product has a very high fixed carbon content, but the stored energy is much smaller compared to other conditions, which makes this product less suitable if mass is prioritized.

In this study, a temperature of 275°C with a residence time of 20 minutes (WCC1-20) is the best overall operating condition, as it provides an optimal balance between calorific value enhancement, mass conservation, and energy efficiency, suitable for industrial applications such as co-firing, biomass upgrading, and large-scale biochar production.

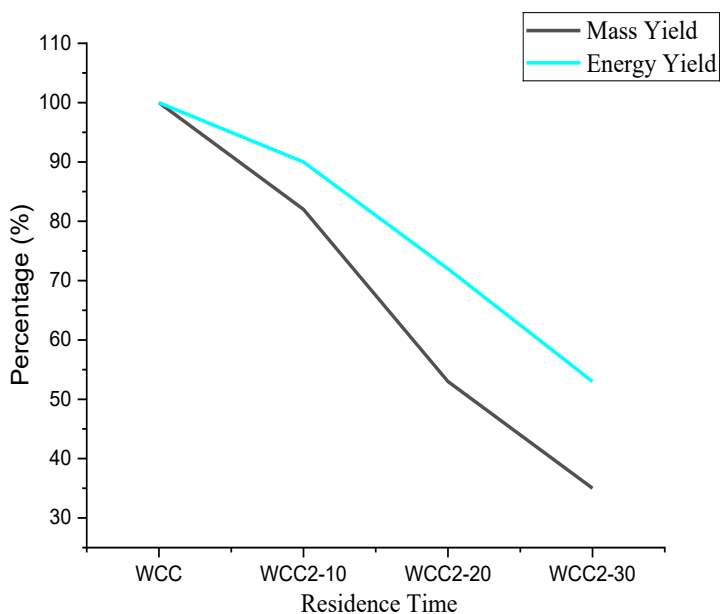


Fig. 7. Graph of mass yield and energy yield at 300°C

3.4 Proximate analysis

Proximate analysis of biomass includes several aspects, such as moisture content, volatile matter, ash, and fixed carbon, all of which affect how well biomass can be used as an energy source [15]. The proximate analysis of the Callandra wood chip sample at 275°C can be seen in Fig. 8.

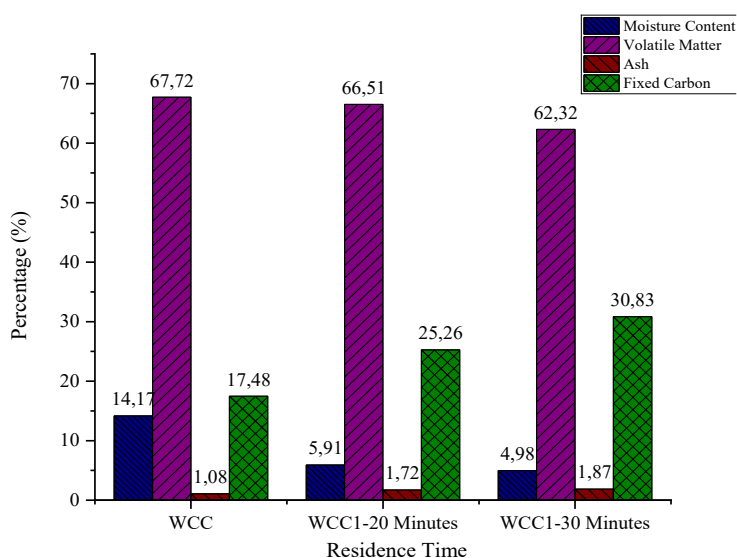


Fig. 8. Proximate graph at 275°C

In the WCC sample, the volatile matter content was still high at 67.72%, and the moisture content was 14.17%, indicating that there were still many volatile components. After torrefaction for 20 minutes (WCC1-20), the moisture content was reduced to 5.91%, while the volatile matter decreased slightly to 66.51%. The percentage of fixed carbon content increased from 17.48% to 25.26%, indicating increased carbonization. This finding is in line with the results of other studies showing that an increase in fixed carbon occurs along with a decrease in volatile content [16].

When the residence time was extended by 30 minutes (WCC1-30), the volatile matter content decreased further to 62.32%, the moisture content decreased further due to evaporation to 4.98% and the carbon percentage still increased significantly to 30.83%. The increase in carbon remains at 30 minutes, which in theory should increase the calorific value, but test data shows that this does not automatically happen. The calorific value is the result of the combined energy per unit mass plus the chemical composition, especially hydrogen, carbon, and oxygen. At 30 minutes, the torrefaction process takes longer than the optimum point for a temperature of 275°C. As a result, there is a loss of volatile compounds, which are still rich in energy. So, although carbon

increases, the net energy stored in the form of volatile compounds is lost more, causing the total calorific value to decrease slightly.

At 30 minutes, hydrogen is almost unchanged (6.10% → 6.08%), meaning that the additional energy from H remains stagnant, but the decrease in Oxygen content is significant (from 41.07% → 35.14%), and this is not completely replaced by carbon which is more efficient in increasing the calorific value. The decrease in oxygen increases the energy per mass, but it is not comparable to the loss of total mass containing light energetic compounds, which results in a slight decrease in the calorific value.

The ash content remained low with a slight increase in percentage from 1.08% (WCC) to 1.87% (WCC1-30). This increase was due to the decrease in the percentage of other contents, resulting in a more concentrated ash content. This indicates that torrefaction did not cause an increase in the content of these residues [17]. Overall, torrefaction improves fuel quality by reducing moisture and volatile matter content and increasing fixed carbon, which at 275°C with a residence time of 30 minutes produces a more energy-rich and stable biomass for bioenergy applications. Furthermore, the graph of proximate analysis on the Callandra wood chip sample with a 300°C temperature can be seen in Fig. 9.

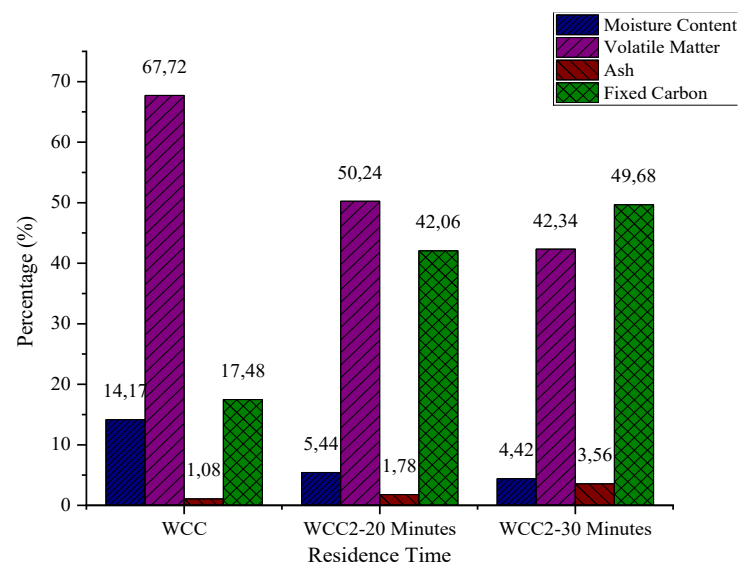


Fig. 9. Proximate graph at 300°C

Fig. 9 shows that the torrefaction process at 300°C with residence times of 20 and 30 min more significantly reduced the volatile matter content from 67.72% (WCC) to 50.24% (WCC2-20) and 42.34% in sample WCC2-30, due to the decomposition of hemicellulose and cellulose. The moisture content was also reduced further due to further evaporation at high temperature to 5.44% for WCC2-20 and 4.42% for WCC2-30 samples. This condition is consistent with the previous study, which suggested that torrefaction not only affects the volatile substances but also effectively reduces the moisture content through accelerated evaporation at high temperature [18]. Meanwhile, the percentage of fixed carbon content increased sharply from 17.48% to 42.06% at WCC2-20 and 49.68% at WCC2-30, indicating increased carbonization. The percentage ash content slightly increased to 3.56% at 30 min residence time, due to the remaining and more concentrated inorganic materials. Overall, torrefaction at higher temperatures improved the quality of biomass with longer residence time, reducing volatiles and moisture content more and increasing fixed carbon, making it more stable and energy-rich for bioenergy. These results are consistent with previous studies that show structural modification and chemical composition of biomass through torrefaction contribute positively to solid fuel characteristics [19].

3.5 Ultimate analysis

The torrefaction process decreases the percentage of oxygen in the biomass and increases the percentage of carbon. The ultimate analysis for the samples at 275°C with residence times of 20 minutes and 30 minutes can be seen in Fig.10.

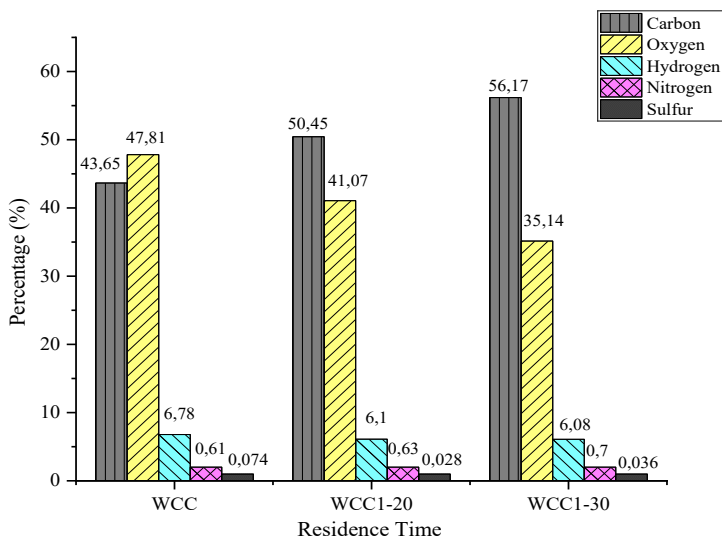


Fig. 10. Graph of ultimate at 275°C

Fig. 10 shows the ultimate analysis results of WCC, WCC1-20, and WCC1-30 samples. At the beginning (WCC), the percentage of carbon was 43.65%, hydrogen 6.78%, nitrogen 0.61%, sulfur 0.074%, and oxygen 47.81%. After being refined with a residence time of 20 minutes (WCC1-20), the percentage of oxygen and hydrogen decreased to 35.14% and 6.1%, while the percentage of carbon increased to 50.45%. The increase in carbon percentage is due to the reduction in oxygen and hydrogen percentage during torrefaction, which is caused by the decomposition of hemicellulose and cellulose and the release of volatile compounds. This increases the concentration of carbon in the sample without increasing its absolute amount. The ultimate analysis for samples at 300°C with residence times of 20 min and 30 min can be seen in Fig. 11.

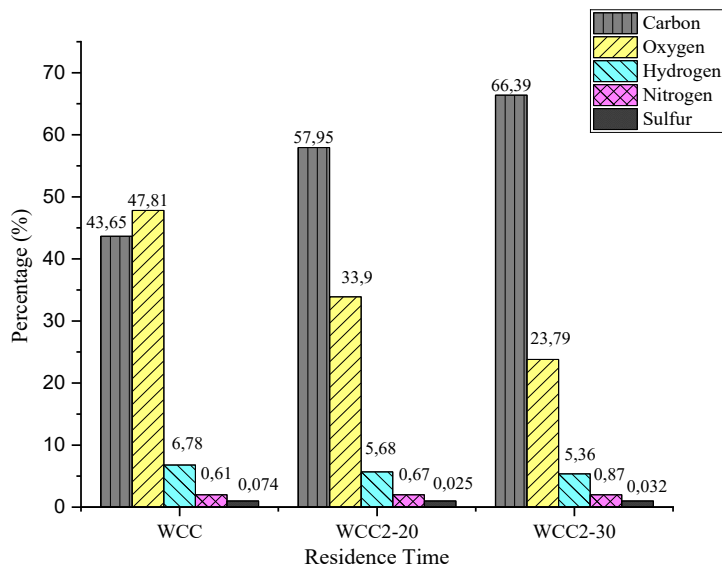


Fig. 11. Graph of ultimate at 300°C

Fig. 11 shows the results of the ultimate analysis on samples WCC, WCC2-20 and WCC2-30. Just as in the sample with a temperature of 275°C where the longer the residence time applied to the torrefaction process, the decrease in the percentage of oxygen and hydrogen will be more and vice versa to the increase in the percentage of carbon but it becomes more significant due to the higher temperature applied, namely 300°C. In the WCC2-30 sample, the increase in the percentage of carbon rose sharply to 66.39%, with the oxygen percentage dropping to 23.79%, followed by hydrogen to 5.36%. The sulfur content will remain because the torrefaction temperature still cannot vaporize the residue, but the percentage increases due to the decrease in other contents [20].

The results of the ultimate analysis show that the oxygen and hydrogen content in the Calliandra wood chip torrefaction product for all variations is less than the raw sample, so that the ratio of O / C and H / C of the torrefaction product has decreased and is close to

the same as coal. Torrefaction converts wood chips into carbon-rich solids that resemble coal. The H/C and O/C ratios are key indicators of the degree of decomposition, carbonization efficiency, and energy quality of torrefied woodchip. These ratios explain the trend in calorific value, reflect improvements in fuel quality, and are used in industrial process optimization. During the process, hydrogen and oxygen are lost faster than carbon, so H/C and O/C decrease. The lower these ratios, the higher the carbonization. Oxygen in biomass does not contribute much combustion energy and tends to reduce the calorific value. The decrease in O/C and H/C indicates that the oxygen content is reduced, the carbon content is more concentrated, which ultimately results in a higher calorific value. This relationship is clearly seen in the data Table 3: WCC2-30 (O/C 0.269, H/C 0.969) produced the highest Gross Calorific Value: 6399 kcal/kg. Fig.12 shows van Kravlen diagrams for samples WCC, WCC1-20, WCC1-30, WCC2-20, WCC2-30 and samples in other references. Where the H/C and O/C ratios are calculated using Eqs. (4) and (5) [23].

Table 3. O/C and H/C ratio of biomass

| Sample | O/C | H/C | Reference |
|-------------------|-------|-------|-----------|
| WCC | 0.821 | 1.864 | |
| WCC1-20 | 0.611 | 1.451 | |
| WCC1-30 | 0.468 | 1.298 | |
| WCC2-20 | 0.439 | 1.176 | |
| WCC2-30 | 0.269 | 0.969 | |
| SCG 300-0.5 | 0.23 | 1.13 | [21] |
| CH 300-0.5 | 0.22 | 0.99 | [21] |
| CPS 250 | 0.599 | 1.353 | [22] |
| CPS 300 | 0.388 | 1.077 | [22] |
| Wood dust biomass | 0.317 | 0.877 | [23] |
| TCS280 | 0.201 | 0.903 | [24] |

$$\frac{H}{C} = \frac{\text{Number of hydrogen (H) atoms}}{\text{Number of carbon (C) atoms}} \quad (4)$$

$$\frac{O}{C} = \frac{\text{Number of oxygen (O) atoms}}{\text{Number of carbon (C) atoms}} \quad (5)$$

The table above presents the calculated data on the ratio of oxygen to carbon (O/C) and the ratio of hydrogen to carbon (H/C) in various samples to see their classification near coal.

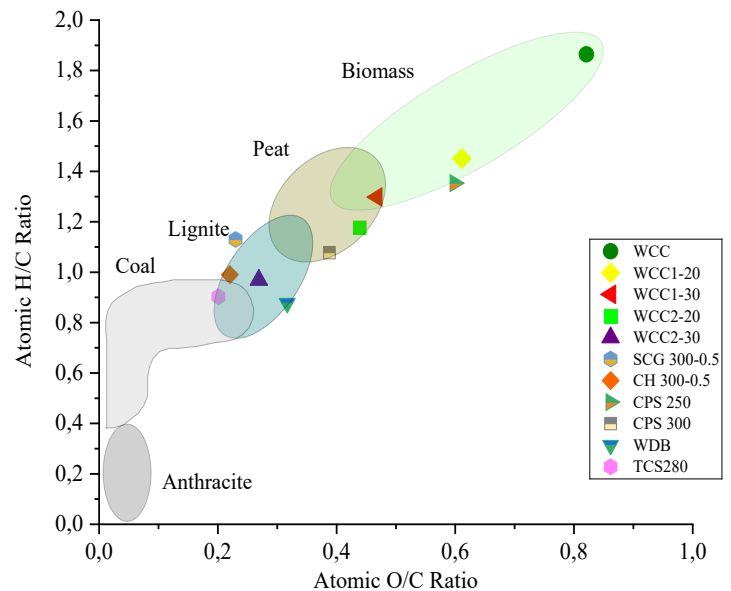


Fig. 12. Diagram van Kravlen

Fig. 12 above shows the van Kravlen diagram in displaying the classes of Calliandra wood chip biomass raw and torrefaction results based on O/C and H/C ratios. Increasing the temperature and extending the residence time during the torrefaction process had a significant impact on decreasing the atomic ratios (H/C) and (O/C) of Calliandra wood chip biomass. Before torrefaction, the H/C and O/C ratios were recorded as 1.865 and 0.821, respectively. However, after the torrefaction process, these values decreased to between 0.269-0.611 for O/C and 0.969-1.451 for H/C. This decrease is due

to the reduction of hydrogen and oxygen content during the torrefaction process, where decomposition occurs, which produces gases such as carbon monoxide (CO) and carbon dioxide (CO₂) [25]. Based on this atomic ratio, sample WWC2-30 is similar to lignite coal, and the other samples are similar to peat. According to data from various research, torrefaction biomass based on the atomic ratio is between lignite and peat.

Table 4. Hydrophobicity result

| Sample | Parameters | | | |
|---------|------------------|---------------------------|-----------------------|------------------------|
| | Initial mass (g) | Mass after 30 minutes (g) | Mass after 1 hour (g) | Mass after 2 hours (g) |
| WCC | 30 | 37 | 39 | 41 |
| WCC1-10 | 30 | 37 | 38 | 39 |
| WCC1-20 | 30 | 34 | 35 | 36 |
| WCC2-10 | 30 | 35 | 36 | 37 |
| WCC2-20 | 30 | 32 | 33 | 33 |
| WCC2-30 | 30 | 31 | 33 | 33 |

Based on Table 4, the percentage of water absorption is calculated based on formula (3), which is then presented in graph form. Fig. 13 below is a graph showing the comparison of water absorption in WCC samples. WCC1-10 and WCC1-20.

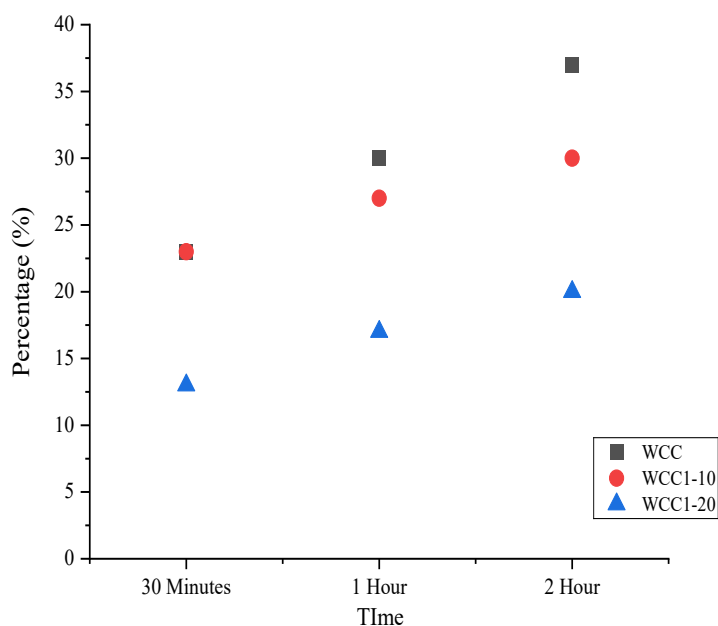


Fig. 13. Graph of hydrophobicity analysis of 275°C temperature sample

Fig. 13 above shows how much the percentage of water absorption in Calliandra wood chip biomass. In the WCC sample, the percentage of water absorption was 23% when soaked for 30 minutes, and it became 36.67% when soaked for 2 hours. After going through the torrefaction process, the WCC1-10 and WCC1-20 samples become more hydrophobic, which is lower water absorption than the WCC sample, where, after soaking for 2 hours, the two samples are only able to absorb water by 30% and 20%. Furthermore, Fig. 14 shows the hydrophobicity analysis of the 300°C sample.

Fig. 14 shows the percentage of water absorption in the Calliandra wood chip torrefaction sample. Similar to the 275°C sample, the absorption decreased due to the torrefaction process. However, the samples became more hydrophobic as the torrefaction temperature and residence time increased. Where the absorption of WCC2-20 and WCC2-30 samples stabilized when soaked for 1 hour and 2 hours, they only absorbed 10% water. This occurs due to the decomposition of hemicellulose and cellulose, while the more heat-resistant lignin remains. Lignin is naturally hydrophobic, so its dominant presence after torrefaction makes the biomass more hydrophobic [26]. Parametric sensitivity analysis showed residence time, temperature, and water content as the most influential variables, with temperature playing the most important role in the torification process of lignocellulosic biomass [27].

3.6 Hydrophobicity analysis

Hydrophobicity analysis was conducted to see the hydrophobicity of the biomass that has gone through the torrefaction process. The test was carried out by immersing the sample in a container of water for 30 minutes, 1 and 2 hours, with the initial weight of the sample of 30g. The results of water absorption in the samples can be seen in Table 4.

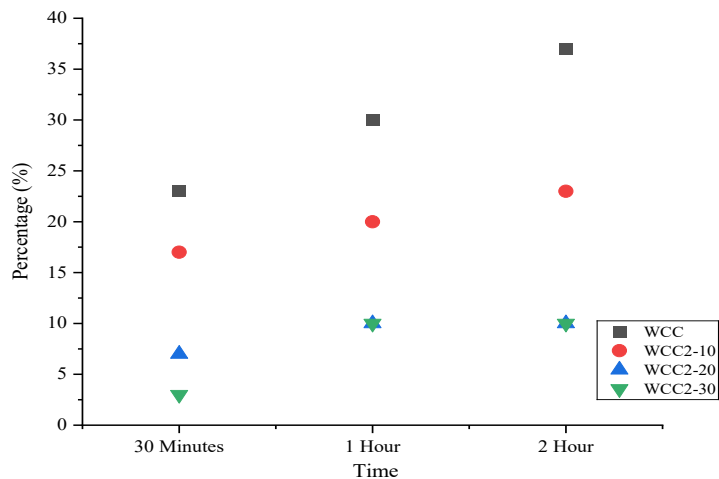


Fig. 14. Hydrophobicity analysis graph of the 300°C temperature sample

4 Conclusion

This study investigated the effect of temperature and residence time on the physical and thermal properties of Calliandra wood chips. The results show that Calliandra wood chips that have gone through the torrefaction process have better quality than the raw samples. The highest calorific value and highest fixed carbon were obtained by the sample at 300°C WCC2-30, which amounted to 6399 kcal/kg and 49.68%, which is equivalent to coal. But it has a relatively small mass yield and energy yield when weighing in terms of mass. The optimal sample of high calorific value and optimal mass yield and energy yield is in sample WCC1-20 with a temperature of 275°C, which is 5387 kcal/kg, 70%, and 88%. The longer the residence time applied, the release of volatile substances and evaporation of water will be more and more, which causes an increase in calorific value. Torrefaction also makes the product more hydrophobic, which is evident in the WCC2-30 sample, which only absorbs 10% water when soaked for 2 hours.

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