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Biomass Fuel Characteristics of Malaysian *Khaya senegalensis* Wood-Derived Energy Pellets: Effects of Densification at Varied Processing Temperatures

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Abstract: This study addresses the effects of densification at varied pelletization temperatures on the novel Malaysian *Khaya senegalensis* wood-derived pellets biomass fuel characteristics. The lack of comprehensive understanding regarding the biomass fuel characteristics of this species prompted the research. By addressing this knowledge gap, this study explores the impact of temperature variations on key fuel properties, contributing to the optimization of sustainable biomass fuel production in manufacturing and materials processing. *Khaya senegalensis* wood, grown and harvested in Malaysia, was pelletized at different temperatures to analyze the calorific value, volatile matter content, ash content, fixed carbon, bulk density, and moisture contents of the pellets. The experimental data revealed a significant relationship between temperature and these fuel properties. Pelletizing at 75 °C produced the highest calorific value of 19.47 MJ/kg and the maximum fixed carbon content of 10.04%. A low ash level of 4.26% was achieved via pelletizing at 75 °C. According to the results, 75 °C produced the best thermophysical properties. These findings provide valuable understanding of how pelletization temperature influences fuel pellet thermophysical properties, a critical aspect in optimizing fuel pellet production, storage, advancing renewable energy resource utilization, and, finally, promoting a cleaner and more sustainable energy future.



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Keywords: biomass; pelletization; biomass pellet; sustainable energy resources; material processing; temperature; volatile matter; ash content; fixed carbon; bulk density; moisture content; calorific value; fuel properties

1. Introduction

Using fossil fuels for energy has led to a heightened awareness of global climate change, resulting in a surge of scholarly investigations and advancements in sustainable renewable energy production [1,2]. The utilization of renewable energy sources has the potential to mitigate the dependence on fossil fuels, hence leading to a reduction in greenhouse gas emissions [3,4]. Fuel pellet combustion is essential for a number of energy-related processes, including power generation, industrial processes, and home heating. Utilizing fuel pellets effectively and sustainably is crucial since it directly impacts emissions, overall environmental impact, and energy efficiency. The temperature to which they are subjected during the pelletization process is one of the major elements affecting the fuel characteristics of fuel pellets [5,6]. To maximize pellet production, storage, and use, it is crucial to comprehend how the temperature of pelletization affects the fuel characteristics of fuel pellets.

This study attempts to investigate the relationship between energy pellet fuel characteristics and processing temperature. Aspects like heating value, volatile matter content, ash content, fixed carbon, bulk density, and moisture contents are among the fuel properties

taken into account. The collected experimental data are analyzed to establish trends and correlations between temperature and fuel properties. In addition, the study investigates the mechanisms and interactions that govern the fuel combustion behavior of fuel particles under varying thermal conditions.

Previous studies have extensively investigated the complex correlation between densification temperature and the fuel characteristics of energy pellets, thereby enhancing our comprehension of their performance in diverse energy-related contexts. The research conducted by [7] examined the impact of pelletization temperature on the heating value, uncovering a positive relationship between elevated pelletization temperatures and increased energy generation. In a study conducted by [8], the researchers examined the relationship between pelletization temperature and the concentration of fixed carbon. The results of their study demonstrated that an increase in carbonization temperature leads to a corresponding rise in fixed carbon content. This increase in fixed carbon content is desirable, as it results in greater energy content and, thus, a better combustion process. These literary works jointly highlight the significance of temperature regulation in maximizing the efficiency of energy generation systems.

The significance of the pelletization temperature in relation to the fuel qualities is a crucial factor in the process of enhancing the efficiency and effectiveness of biomass pellets as a sustainable energy alternative. The temperature at which pelletization occurs has a substantial impact on the physical and chemical properties of the resultant pellets, which, in turn, affects their combustion behavior, energy efficiency, and environmental implications. The process of pelletization entails subjecting biomass resources to elevated temperatures and pressures, resulting in the formation of compact and homogeneous pellets. The temperature at which this process takes place exerts a significant influence on certain crucial fuel characteristics. In a study conducted by [9], it was observed that a higher pelletization temperature makes the material more flexible and simpler to press. Also, at high temperatures, an increase in the interfacial area between adjacent particles is anticipated, resulting in increased adhesion. In addition, ref. [10] noted that gaining a comprehensive understanding of the actual impacts of temperature on the fuel properties of biomass at a small scale is crucial for facilitating the clean and efficient utilization of biomass on a larger scale. The complex nature of this relationship highlights the necessity for a more thorough examination in order to comprehend the subtle dynamics between temperature and pellet characteristics.

The novelty of this research lies in its comprehensive exploration of the intricate relationship between pelletization temperature and fuel properties of *Khaya senegalensis* biofuel pellets. While the impact of temperature on pellet properties has been studied to some extent, this study delves deeper, unearthing a unique perspective on how varying temperatures affect pellet composition and fuel behavior, but it also offers a pioneering exploration of the potential of *Khaya senegalensis* as an alternative feedstock. These distinctive insights contribute to a deeper understanding of biofuel pelletization processes, ultimately advancing the field of renewable energy. For the preliminary stages, the use of branches trimmings waste from *Khaya senegalensis* is projected to address challenges associated with rapid growth. It is noteworthy that *Khaya senegalensis* has been intentionally planted along Malaysian roadsides to enhance road strength. The challenge lies in fast growth, leading to frequent trimming, potentially affecting traffic and posing a disposal issue for the trimmings. However, it is important to highlight that the current approach aims to minimize any adverse impacts on local ecosystems.

A considerable body of literature exists regarding the combustion of biomass derived from various sources, with particular emphasis on well-established dedicated energy crops (DEC), including poplar, willow, and miscanthus. However, there is a paucity of data regarding the fuel characteristics of pellets formed from *Khaya senegalensis* wood biomass. In addition, to date, there is currently no existing comprehensive study that provides a summary of the impact of pelletization temperature on the fuel properties of khaya wood biomass. Therefore, our paper aims to contribute to the existing body of knowledge in

this area. The findings of this study will have implications for optimizing fuel pellets' production, storage, and transportation. In addition, a thorough understanding of the effect of temperature on the fuel properties of fuel pellets will contribute to the improvement of the efficiency, environmental sustainability, and performance of pellet-based energy systems. Studying the impact of temperature on the fuel properties of fuel pellets is essential for advancing the use of biomass and other renewable energy sources in a greener and more sustainable manner. This research will pave the way for informed decision making in the design and operation of biomass-based energy systems and contribute to a sustainable and more energy-efficient future.

2. Materials and Methods

The study was conducted at the Faculty of Mechanical Engineering & Technology at Universiti Malaysia Perlis (UniMAP), Malaysia. The study focuses on the use of fully grown *Khaya senegalensis* tree branches and trunks, which were harvested at the Institute of Sustainable Agro-Technology (INSAT) Research Station, Universiti Malaysia Perlis (UniMAP) in Sungai Chuchuh, Padang Besar. The harvesting location's coordinates are 6°39'09.6" N latitude and 100°15'40.3" E longitude. The branches were dried in a natural air setting before a sample was randomly chosen for pelleting. Maintaining consistent pellet quality across diverse production is vital for the reliability and effectiveness of biofuel production. The study comprehensively incorporates rigorous quality control measures at various stages of the pelletization process. These measures involve meticulous monitoring of raw material characteristics, precise control of processing parameters, and inspection of pellet attributes. Additionally, standardized testing protocols, specifically those defined by the American Society for Testing and Materials (ASTM), were integrated into this study to assess key quality indicators.

The major goal of this study is to ascertain how differences in the temperature of the pelletization process impact the quality of wood pellets made from *Khaya senegalensis*, drawing attention to the precedent set by other papers, such as in the works of [2,3,8], where three repetitions were considered standard for biomass materials characterization. In this study, the parametric experiment was conducted with five levels of biomass temperature (25, 50, 75, 100, and 125 °C). It is important to note that throughout the experiment, each temperature condition was replicated three times. This repetition helped ensure the reproducibility and robustness of the findings.

The entire tree branch and trunk khaya wood was then chipped using a woodchipper (brand: CIMA, model: FC 6100 S) without selective separation. This method aimed to provide a comprehensive view of the pelletization process and its impact on energy pellet properties without differentiating between bark and other components. The unprocessed material was then ground using a microfine grinder (brand: IKA, model: MF 10 BASIC), resulting in smaller particle sizes. Following that, the produced particles were collected and passed through a screening procedure in accordance with the ASTM standard to obtain the necessary particle size. Particle size distribution for khaya samples before pelleting ranged from 0.015–4 mm. For this experiment, a particle size of 0.5 mm was chosen as the target size for the biomass particles before pelleting.

The biomass was transformed into pellets using a manual single-press hydraulic machine (brand: SPECAC, model: GS1501) with a temperature controller. In the experiment, a precise and controlled temperature manipulation process was employed to investigate the effects of various temperatures on the biomass samples. The temperature control was crucial to ensure the reliability and accuracy of the results. The temperature controller was then programmed to follow a specific temperature profile (25, 50, 75, 100, and 125 °C). One gram of khaya samples was inserted into a mold while waiting for the temperature controller to attain the desired temperature. After the temperature control system had attained the desired temperature, a brief period was allowed for stabilization before the khaya biomass samples were inserted into the pelletization chamber. The pellets that were produced had a 10 mm diameter.

The fuel characteristics of the pellets, including their ash content, volatile matter, fixed carbon, and calorific value, were evaluated using a muffle furnace (brand: DAEYANG, model: MF-2511). *Khaya senegalensis* fuel pellets were subjected to proximate analysis using the American Standard Testing Material (ASTM) method to determine a number of crucial properties for burning and determining energy density.

The volatile matter test was initiated by placing the sample within a muffle furnace set at a temperature of 950 ± 0.5 °C for 7 min. After a precise duration of 7 min, the sample was permitted to undergo a cooling process at room temperature. After the sample's temperature had decreased, the sample was weighed again to determine its weight loss. The determination of volatile matter was conducted using Equations (1) and (2), whereas the calculation of ash content, as indicated in Equation (3), was derived from ASTM E872-82 and ASTM D1102-84, respectively. Before determining the percentage of volatile matter, it is necessary to account for the moisture content in the pellet sample. This is performed by measuring the mass loss (B) of the sample when subjected to heating or drying. Moisture loss (A) represents the portion of mass loss attributed to moisture evaporating from the sample during heating. Then, the percentage of volatile matter can be calculated by subtracting the mass loss due to moisture (A) from the total mass loss (B). *Khaya senegalensis*' moisture content or mass loss (A) was measured using a moisture analyzer (brand: AND, model: MX-50).

$$\text{Weight loss, } B(\%) = \frac{W_i - W_f}{W_i - W_c} \times 100\% \quad (1)$$

$$\text{Volatile Matter } (\%) = B - A \quad (2)$$

where W_i = initial weight of the pellet, crucible, and cover (g), W_f = final weight of pellet sample (g), and W_c = weight of crucible and cover (g).

The ash content was calculated based on the mass of the residual remaining after the sample was heated in the air under stringently controlled time, sample weight, and equipment parameters. The sample was deposited inside the furnace and heated for 60 min at an approximate temperature of 700 ± 0.5 °C. The initial mass before heating (mass initial) and the final mass after heating (mass final) were obtained, and the standard following Equation (3) was used to calculate the ash content percentage in biomass pellets.

$$\text{Ash Content } (\%) = \frac{W_3 - W_1}{W_2} \times 100\% \quad (3)$$

where W_1 = weight of empty crucible (g), W_2 = weight of pellet sample (g), and W_3 = weight of crucible containing the ash (g).

Furthermore, this study also determined the calorific value and fixed carbon content. Fixed carbon content was determined by the difference between 100 and the total of the experimentally measured percentages of ash content, moisture content, and volatile matter in the proximate analysis [11].

The predicted high heating value (HHV), also known as the calorific value, was determined based on the correlation in Equation (4) [11]. With an R^2 value of 0.827 and a standard deviation of 1.483 MJ/kg, this equation is, thus, trustworthy. The results of the experimental proximate analysis carried out in this study were used to determine the amounts of calorific value.

$$\text{Calorific Value} = 167.2 - 1.449 \text{ VM} - 1.562 \text{ FC} - 1.846 \text{ ASH} \quad (4)$$

where FC = fixed carbon, VM = volatile matter, and ASH = ash.

Bulk density is determined by the ratio of material mass to container volume. The pellets were filled to the top surface of the 50 mL beaker and weighed using a digital scale. To account for settling, the beaker was tapped five times.

The moisture content of the manufactured khaya pellets was tested using moisture analyzer (brand: AND, model: MX-50).

Before attempting to fit an equation, it is essential to thoroughly understand the characteristics and nature of the data. During this stage, trends, patterns, and any anomalies are identified. Based on the understanding of the data and the purpose of the equation, a mathematical model or equation form that is likely to capture the underlying relationship is chosen. This process may involve selecting a linear model, exponential model, polynomial model, etc. Subsequently, statistical methods are employed to fit the chosen model to the data, and the goodness of fit is assessed by analyzing statistical metrics such as R-squared or others, depending on the nature of the data. A higher R-squared value generally indicates a better fit. If the initial fit is unsatisfactory, the model is refined, or alternative equations are considered. The process may require several iterations to identify the most suitable equation. Once a candidate equation is available, validation using additional data is performed if available. This ensures that the equation does not overfit the existing data but can generalize to new observations.

3. Results and Discussion

In this study, we focused on analyzing the impact of pelletization temperature on fuel characteristics from multiple perspectives. We experimentally investigated how different pelletization temperatures influenced essential parameters such as ash content, volatile matter, fixed carbon, and calorific value. In addition, the analysis of variance (ANOVA) was employed to examine the correlation between the temperature and the essential parameters. The mathematical equations were developed according to the experimental results. These equations provide valuable insights into how pelletization temperature changes impact the pellet's fuel properties. In addition, these equations can be used to predict the responses for given temperature limits.

The incorporation of *Khaya senegalensis* for biofuel production introduces a range of environmental implications that warrant careful consideration. This particular tree species, given its nature, has the potential for carbon sequestration, offering a positive environmental impact as it absorbs and stores carbon dioxide during its growth. However, attention must be directed towards potential emissions and energy consumption during cultivation and processing, necessitating efficient and sustainable practices to reduce the overall environmental footprint. In essence, while *Khaya senegalensis* presents opportunities for sustainable biofuel production, a balanced and inclusive approach is imperative to address potential environmental challenges and promote overall sustainability.

3.1. Ash Content

The results obtained from the parametric analysis of temperature on khaya energy pellets ash content are set out in Table 1. The ash content of a substance holds significant importance as it represents the residual matter that remains after the completion of combustion. A lower ash residue content is suggestive of an efficient and clean combustion process [9]. The ash content of the *Khaya senegalensis* biofuel pellets varied at different temperatures. At 25 °C, the ash content was measured to be 5.56%. As the temperature increased to 50 °C, there was a slight increase in the ash content, which reached 5.98%. However, at 75 °C, the ash content decreased significantly to 4.26%. Subsequently, at 100 °C, the ash content increased to 5.59%. Finally, the highest ash content of 6.88% was observed at 125 °C. These results indicate fluctuations in the ash content with varying temperatures.

Table 1. The effects of pelletization temperature on khaya energy pellets ash content.

Pelletization Temperature	Ash Content (%)
25	5.56
50	5.98
75	4.26
100	5.59
125	6.88

One potential explanation for these fluctuations could be related to the complex and dynamic nature of biomass combustion. When *Khaya senegalensis* pellets are burned, a combustion process occurs, involving the oxidation of organic compounds, resulting in inorganic ash residues. The composition and quantity of this ash can be influenced by a range of factors, including the specific properties of the biomass, combustion conditions, and temperature variations. It is possible that within the temperature range studied, certain temperature thresholds or ranges exist at which combustion behavior or ash production undergoes shifts or variations. Additionally, the presence of specific compounds or impurities in the biomass could contribute to the observed fluctuations. The composition and amount of ash produced depend on the properties of the biomass and the combustion conditions. This phenomenon is supported by Mock (2020) [12], who studied single pine wood and empty fruit bunch pellets biomass pelletization and found that the process increases the pellet bulk energy density and uniformity and can be burned in industrial furnaces. In addition, their experimental study examined the combustion behavior of single pine wood and empty fruit bunch pellets in a laboratory-scale entrained-flow reactor. It was also attributed to the low ash level in high-temperature hydrochar because the inorganic components in the feedstock dissolved in the liquid phase [13].

Another researcher [14] investigated wheat pellets and found that as the temperature decreases, the wheat ash concentration decreases. This is intuitively anticipated; as the sample loses more moisture, the ash concentration should increase because water has been removed from the sample. Apart from that, it seems possible that these results are due to the fact that, as the temperature increases, organic matter within the biomass undergoes thermal degradation [15]. This process involves the breakdown of complex organic compounds into simpler molecules, releasing volatile gases and leaving behind a solid ash residue. The reduction in ash content observed at 75 °C could be due to more efficient degradation of organic matter and subsequent release of volatile components. In addition, the ash components may undergo fusion and sintering at higher temperatures, such as 100 °C and 125 °C. Fusion refers to the melting of specific ash components, while sintering involves the development of solid structures due to the joining of melted or softened ash particles. These methods can increase ash content, as the molten or sintered particles contribute to the overall ash residue.

The R^2 value of 0.72 indicates a moderate correlation between temperature and ash content. This value means that approximately 72% of the variation in the ash content can be explained by changes in temperature. However, it also implies a significant amount of variability not accounted for by temperature alone. Other factors, such as biomass composition or combustion process, may contribute to the remaining variability in ash content.

$$\text{Ash content (\%)} = 0.0065 T^3 - 0.0984 T^2 + 0.3002 T + 5.468 \quad (5)$$

The overlap in the error bars indicates that the difference in ash content between 25 °C and 100 °C may not be statistically significant. This means that the ash content values at these two temperatures could be within the margin of error, suggesting that the observed difference may be due to random variation rather than a true effect of temperature.

The ANOVA test conducted on the data (shown in Table 2) indicates statistically significant differences in ash content among the tested temperatures. The reported p -value of 0.015 (less than 0.05) suggests that the observed differences are unlikely to occur by chance alone. This situation indicates that temperature influences the ash content, even though the overall trend is not apparent. The SS (sum of squares) column calculates the total squared deviations of each data point from the overall mean, while df (degrees of freedom) refers to the freedom of variation associated with each source. The MS column (middle square) calculates the variation within each category and provides information about the variability within specific categories. The F (F-statistic) is a ratio that compares mean squares between groups to mean squares within groups, helping to identify significant differences. The p -value represents the probability that the reported results were obtained

by chance, with lower values indicating statistical significance. Finally, the F-crit (critical F value) is used as a threshold to determine significance.

Table 2. The ANOVA results of pelletization temperature on energy pellets ash content.

Source of Variation	SS	df	MS	F	p-Value	F-Crit
Between groups	4467.646	1	4467.646	6.671533	0.015	4.196
Within groups	18,750.43	28	669.6581			
Total	23,218.07	29				

In summary, the analysis of the experimental data reveals fluctuating ash content values without a clear trend as the temperature changes. The moderate correlation coefficient (R^2) suggests that temperature explains a portion of the variation in ash content. However, other factors may also be at play. The overlap in error bars for 25 °C and 100 °C implies that the difference in ash content between these two temperatures may not be statistically significant. However, the ANOVA test confirms that there are statistically significant differences in ash content among the tested temperatures, indicating that temperature does have some impact on ash content.

3.2. Volatile Matter

The effect of temperature on the volatile matter content of *Khaya senegalensis* biofuel pellets was analyzed. Figure 1 reveals that there was a gradual rise in the volatile matter content with the increment of pelletization temperature levels. At 25 °C, the volatile matter content was 84.56%, which slightly rose to 85.42% at 50 °C. Further increasing the temperature to 75 °C yielded a volatile matter content of 85.70%. This result indicates that a higher temperature facilitated the release of more volatile components from the biofuel pellets. This suggests that the trend of increasing volatile matter content with temperature persisted. At 100 °C, the volatile matter content reached 86%, peaking at 86.40% at 125 °C. This indicates that a higher temperature allowed the release of even more volatile components from the biofuel pellets.

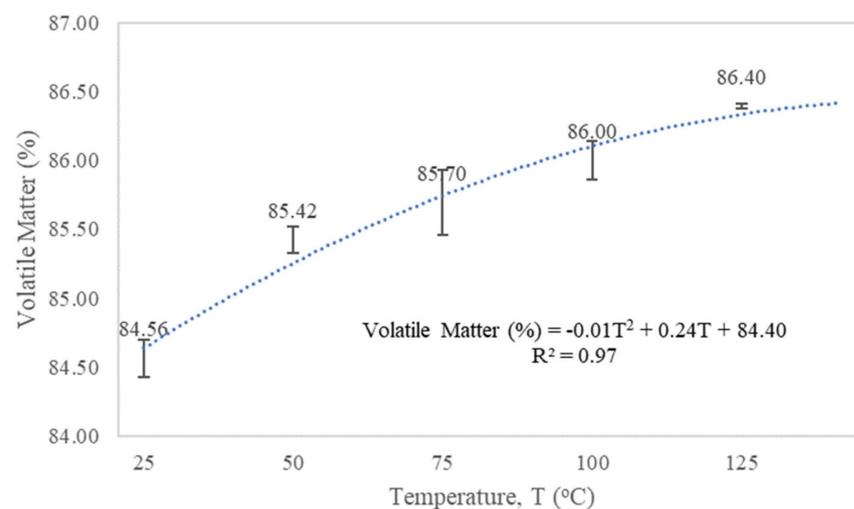


Figure 1. Evaluation of temperature on the *Khaya senegalensis* biofuel pellets’ volatile matter.

Several factors can explain the increasing trend in volatile matter content with increasing temperature. For example, as the temperature rises, the thermal decomposition of organic compounds in the biomass occurs more rapidly. This process involves the breaking down of complex organic molecules into simpler volatile compounds, such as gases and vapors. Volatile matter refers to the portion of fuel, exclusive of moisture, that is liberated when the organic or inorganic constituents of any fuel undergo thermal decomposition

at elevated temperatures [16]. Accordingly, high volatile levels make fuel more reactive during combustion, making it easier for material to ignite at low temperatures. The higher temperatures provide the necessary energy for these reactions to take place, leading to an increase in volatile matter content. On another note, ref. [17] affirmed that elevated levels of volatile matter in biomass frequently result in challenging and rapid combustion. A larger reactor volume is typically necessary to mitigate the occurrence of elevated levels of pollutant emissions, uncombusted byproducts, and polyaromatic hydrocarbons that may arise during the combustion of high-volatile-matter biomass. Therefore, decreasing the volatile matter composition is a favorable advancement for utilizing these solid fuels within a pre-existing coal-fired facility. In addition, according to [12], the volatile matter content or their physical and chemical differences may be the reason for the quicker ignition. Thus, it is a crucial fuel pellet quality to look into.

In addition, higher temperatures promote the vaporization and release of volatile components in the biomass. These volatile components, such as organic compounds, water, and other volatile gases, contribute to the volatile matter content. The increased thermal energy enables the liberation of these volatile components, leading to an overall increase in volatile matter content.

The quadratic relationship between the volatile matter and the pelletization temperature is shown in Equation (6). The data exhibited a high correlation coefficient ($R^2 = 0.97$). The R^2 value of 0.97 indicates a strong correlation between temperature and volatile matter. This implies that approximately 97% of the variation in volatile matter can be explained by changes in temperature. The high R^2 value indicates a robust relationship between the two variables.

$$\text{Volatile Matter (\%)} = -0.01 T^2 + 0.24 T + 84.40 \quad (6)$$

The single-factor ANOVA test conducted on the data as depicted in Table 3 shows that the results were not statistically significant. The p -value reported as 0.27 (greater than 0.05) suggests that the observed differences in volatile matter content among the tested temperatures may have occurred by chance. While the R^2 value of 0.97 indicates a strong correlation between temperature and volatile matter content, it is important to note that the results of the single-factor ANOVA test, with a reported p -value of 0.27 (greater than 0.05), do not reach statistical significance. This suggests that the observed differences in volatile matter content among the tested temperatures may not be statistically significant according to the data provided. While there is a robust correlation between temperature and volatile matter content, further investigation may be needed to determine the practical significance of this relationship.

Table 3. ANOVA analysis of pelletization temperature on the *Khaya senegalensis* biofuel pellets' volatile matter.

Source of Variation	SS	df	MS	F	p -Value	F-Crit
Between groups	845.25	1	845.25	1.26	0.27	4.20
Within groups	18,756.36	28	669.87			
Total	19,601.61	29				

3.3. Fixed Carbon

The experimental data on the fixed carbon content of khaya pellets, which were pelletized at different temperatures, are illustrated in Figure 2. The data analysis reveals a clear and consistent decreasing trend in fixed carbon content as the temperature increases. This implies that higher temperatures during the pelletization process led to a reduction in the fixed carbon content of the khaya pellets. Fixed carbon represents the portion of biomass that remains after volatile components are released during combustion. Understanding the relationship between temperature and fixed carbon content is crucial for optimizing the khaya pellets' combustion characteristics and energy efficiency as biofuel.

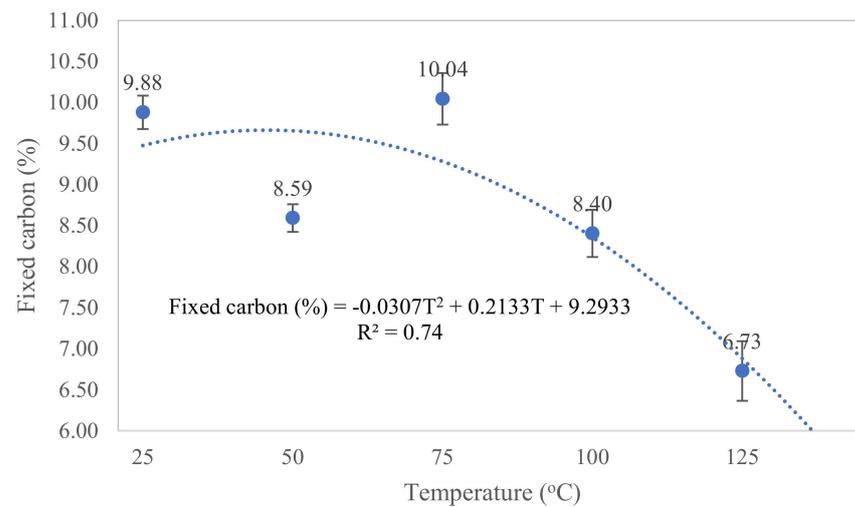


Figure 2. Fixed carbon of khaya pellets pelletized from various temperatures.

At 25 °C, the fixed carbon content was 9.88%. Increasing the temperature to 50 °C resulted in a decrease in fixed carbon content to 8.59%. This finding indicates that the higher temperature during pelletization led to a reduction in the amount of fixed carbon in the pellets. Surprisingly, at 75 °C, the fixed carbon content increased to 10.04%. This could be due to specific reactions or conditions at this temperature that promote the retention of fixed carbon in the pellets. However, as the temperature was further increased to 100 °C, the fixed carbon content decreased to 8.4%. This suggests that the higher temperature caused more fixed carbon to be lost from the pellets. Finally, at the highest temperature tested, 125 °C, the fixed carbon content reached its lowest point of 6.73%. This indicates that the highest temperature resulted in the most significant reduction in fixed carbon content.

Similar to this experiment, Aziz et al. (2011) [18] also computed the fixed carbon content of oil palm biomass through the method of difference. The experiment was replicated three times to ensure the reliability of the outcomes. In addition, Aulia et al. (2019) [8] also opted for the same method for rubber seed shell fixed carbon determination. In the same paper, the author deduced that the fixed carbon content in activated carbon samples increases with higher carbonization temperatures. This implies that the same applies to biomass densification via pelletization, where temperature significantly affects the fixed carbon of the pelletized material.

Following thermal decomposition, the remaining substance consists of both fixed carbon and ash. The predominant source of fixed carbon is derived from the lignocellulosic material. It is imperative to consider this component of the specimen when calculating the overall percentage of hemicellulose, cellulose, and lignin [19]. Following that, in another journal, ref. [12] stated that after the biomass was combusted, all that was left was ash, where the fixed carbon was gone, and there had been no reaction.

The fixed carbon (%) showed a quadratic correlation with the pelletization temperatures, as listed in Equation (7). The moderate correlation coefficient ($R^2 = 0.74$) indicates that approximately 74% of the variation in fixed carbon content can be attributed to changes in temperature. This suggests that temperature is a significant factor influencing the fixed carbon content of the khaya pellets. The statistical analysis using ANOVA shown in Table 4 confirmed the significance of the observed differences in fixed carbon content among the tested temperatures. The low p -value of 0.015 (less than 0.05) indicates that the observed variations in fixed carbon content are unlikely to have occurred by chance alone. Therefore, the temperature during pelletization has a significant impact on the fixed carbon content of khaya pellets.

$$\text{Fixed carbon (\%)} = -0.0307 T^2 + 0.2133 T + 9.2933 \quad (7)$$

Table 4. ANOVA for fixed carbon content of khaya pellets pelletized at varied temperatures.

Source of Variation	SS	df	MS	F	p-Value	F-Crit
Between groups	4467.646	1	4467.646	6.672	0.015	4.196
Within groups	18,750.430	28	669.6581			
Total	23,218.07	29				

3.4. Calorific Value

The observed range of calorific values for the biofuel pellets is shown in Figure 3, ranging from 18.81 to 19.47 MJ/kg. This indicates variations in the energy content of the pellets. The calorific value represents the amount of heat energy released during the combustion of the pellets. It is an essential indicator of their energy content and efficiency. Calorific value is primarily influenced by the chemical composition and structure of the biomass, including its moisture content, volatile matter, and fixed carbon content.

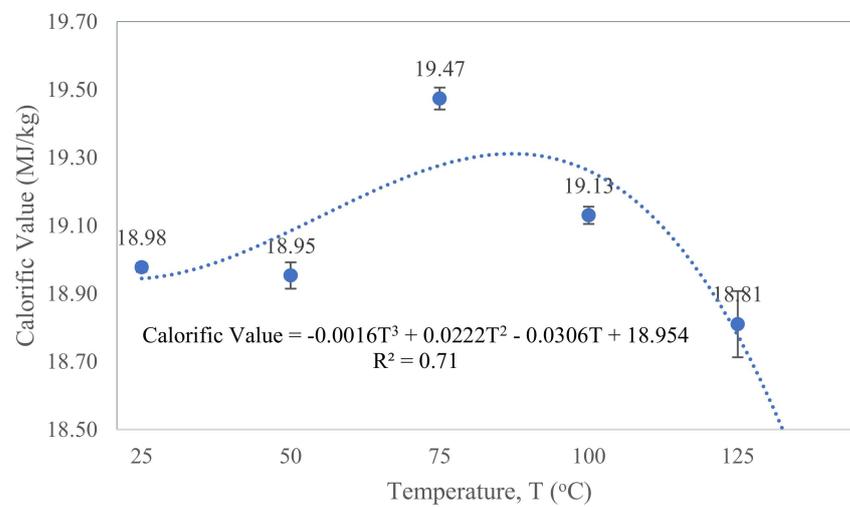


Figure 3. The calorific value of khaya pellets pelletized at different temperatures.

The correlation coefficient ($R^2 = 0.71$) indicates a moderate correlation between temperature and calorific value. This suggests that around 70.64% of the variability in calorific value can be attributed to changes in temperature. The correlation between the calorific value and the pelletization temperature is in polynomial behavior, as shown in Equation (8). The slight variations in calorific value as the temperature changes can be attributed to the thermal decomposition and chemical reactions that occur during the pelletization process. As the temperature increases, several processes come into play, which include moisture evaporation, volatile matter release, fixed carbon transformation, etc. At lower temperatures, such as 25 °C, the moisture content in the pellets is higher. As the temperature rises, the heat energy applied to the pellets facilitates the evaporation of moisture, leading to a decrease in the overall water content [20]. This reduction in moisture content can contribute to a higher calorific value, as the energy is focused more on the combustible components.

$$\text{Calorific Value} = -0.0016 T^3 + 0.0222 T^2 - 0.0306 T + 18.954 \quad (8)$$

Furthermore, as the temperature continues to increase, the volatile matter in the biomass undergoes thermal decomposition and vaporization. Volatile matter consists of volatile organic compounds, such as hemicellulose and lignin, that can quickly vaporize when exposed to heat. The release of volatile matter contributes to the combustible gases and increases the energy content of the pellets. The observed phenomenon may be attributed to the liberation of lignin, given that the aforementioned temperatures (75 °C, 100 °C, and 125 °C) surpass the average transition temperature of lignin (75 °C) [21].

In addition, at higher temperatures, the fixed carbon content of the pellets undergoes thermal degradation. Fixed carbon refers to the nonvolatile carbonaceous material remaining after releasing volatile matter. As the fixed carbon undergoes further pyrolysis and combustion reactions, its energy content may decrease, reducing the calorific value [11]. The observed increase in calorific value at 75 °C may be attributed to an optimal balance between moisture evaporation, volatile matter release, and fixed carbon transformation. The conditions at this temperature may favor the release of combustible components while minimizing the loss of fixed carbon. The subsequent decrease in calorific value at 100 °C and 125 °C suggests that the excessive heat may lead to a more significant loss of fixed carbon and potential degradation of other energy-rich components, resulting in a lower calorific value.

The statistical analysis using ANOVA confirms the significant differences in calorific value among the tested temperatures. The low *p*-value of 0.015 (less than 0.05) indicates that these variations are unlikely to be due to random chance.

In contrast to the calorific value of various woody biomass sources compiled by Osman et al. (2021) [22], the *Khaya senegalensis* calorific value, densified at varied temperature ranges from 18.81 to 19.47 MJ/kg wood, stands out for its high potential in biofuel pellet production. The calorific value of *Khaya senegalensis* wood is comparable to, or even surpasses that of, other biomass types, as demonstrated in the following comparison: greenwood: 8 MJ/kg, fuelwood: 16.10 MJ/kg, ailanthus wood: 18.93 MJ/kg, poplar: 19.38 MJ/kg, spruce wood: 19.45 MJ/kg, black locust: 19.71 MJ/kg.

In conclusion, the analysis reveals slight variations in the calorific value of *Khaya senegalensis* biofuel pellets as the temperature changes. The data suggest that moderate temperature variations during pelletization can impact the pellets' energy content.

3.5. Bulk Density

Figure 4 depicts the observed range of bulk density for khaya biofuel, which ranges from 434.65 kg/m³ to 458.70 kg/m³. In the temperature-dependent study of bulk density, several distinct observations come to light. The analysis reveals a quadratic trend. At 25 °C, the recorded bulk density stands at 451.89 kg/m³. A shift to 50 °C brings about an increase in bulk density, with the value rising to 458.70 kg/m³. However, as the temperature progresses to 75 °C, a subtle decline in bulk density is noted, registering at 447.72 kg/m³. The trend continues as the temperature rises to 100 °C, where the bulk density remains relatively stable at 447.63 kg/m³. Nevertheless, the most striking change occurs when the temperature reaches 125 °C, causing the bulk density to decrease significantly to 434.65 kg/m³.

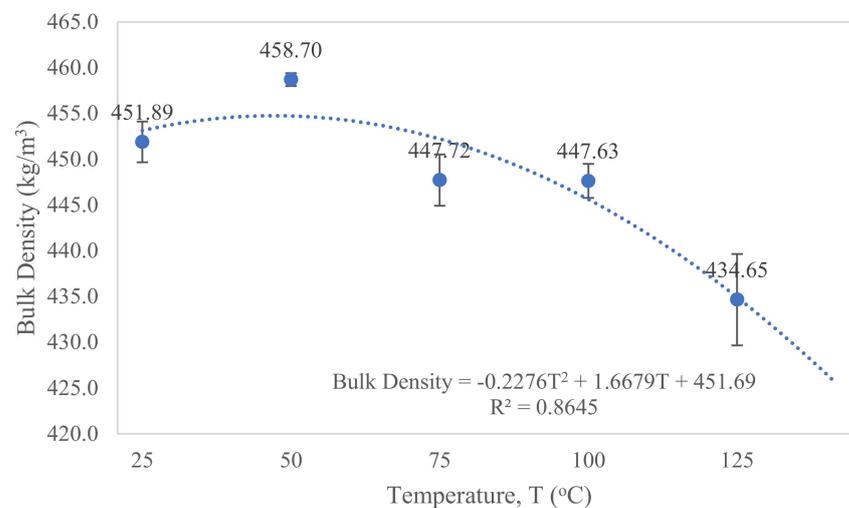


Figure 4. The bulk density of khaya pellets pelletized at different temperatures.

This temperature-dependent variance in bulk density hints at a complex interplay between the material's response to thermal energy and phase transitions. During the course of the research, a formula was discovered that encompasses the complex link between temperature and bulk density, offering insights into the underlying principles regulating these observed fluctuations.

$$\text{Bulk Density} = -0.2276 T^2 + 1.6679 T + 451.69 \quad (9)$$

The initial increase from 25 °C to 50 °C may signify a transition to a denser phase or a more efficient packing of molecules, resulting in elevated bulk density. The slight dip at 75 °C could be linked to further phase transitions or minor structural changes. The persistence of bulk density at 100 °C could suggest a state of equilibrium, potentially involving counterbalancing phase changes. The substantial drop at 125 °C is particularly intriguing and may denote a shift to a less dense phase or even the initiation of vaporization processes. The high R^2 value of 0.86 indicates a strong linear correlation between temperature and bulk density. This implies that variations in temperature are accompanied by corresponding fluctuations in bulk density. Such a relationship is often observed in materials, especially those that can undergo phase changes, chemical reactions, or thermal expansion with changing temperature. As temperature increases, molecules within the material may gain kinetic energy and move more freely, causing a decrease in the intermolecular spacing and a change in bulk density. This could be related to phase transitions or changes in the state of matter (solid to liquid, liquid to gas) occurring within the material. In their study, Cui et al. (2019) [23] examined the impact of temperature on the bulk density of fuel pellets made from wood residues with microalgae as a binder. Their findings indicated that elevating the temperature within the range of 80–160 °C, along with increasing the pressure from 120–200 MPa, resulted in a significant enhancement in the bulk density of the pellets.

The absence of overlap in error bars at most temperature points indicates that the differences in bulk density are statistically significant. This means that the variations in bulk density measurements are likely due to real differences in the material's properties at different temperatures, rather than being mere measurement errors. Furthermore, the extremely low p -value (9.81941×10^{-26}) from the ANOVA test suggests that the differences in bulk density between temperature groups are highly statistically significant. This outcome confirms that temperature is a key factor influencing the bulk density of the material.

In conclusion, the data and analysis suggest that temperature exerts a substantial impact on the bulk density of the material. The trend in the data, the absence of overlapping error bars, and the extremely low p -value from the ANOVA all indicate that these variations are not random but result from temperature-induced changes in the material's structure or state.

3.6. Pellet Moisture Content

Table 5 presents the influence of various pelletization temperatures on the resulting moisture content of Khaya energy pellets. Investigation of moisture content is essential, especially when it comes to pelletization temperature, as moisture content will be impacted by variations in temperature between pelleting and combustion. The \pm shown in Table 5 signifies the standard error of the data.

The table provides a concise overview of the moisture content in Khaya energy pellets at varying pelletization temperatures. At 25 °C, the pellets contain 11.47% moisture, with a small standard deviation of ± 0.05 , indicating a degree of consistency. As the pelletization temperature increases to 50 °C, there is a noticeable reduction in moisture content to 10.68% with a standard deviation of ± 0.11 . This trend continues as the temperature rises to 75 °C, where the moisture content further decreases to 10.19% (± 0.04). Remarkably, at 100 °C, the moisture content remains relatively stable at 10.01%, with minimal variability (± 0.003). The most substantial reduction is observed at 125 °C, where the moisture content drops significantly to 9.23% with a standard deviation of ± 0.06 . These findings underscore the inverse relationship between pelletization temperature and pellet moisture content,

suggesting that higher temperatures are more effective in reducing moisture levels within the pellets. The precision of these measurements, as indicated by the standard deviations, reinforces the reliability of the data, making it valuable for optimizing the production of Khaya energy pellets. This temperature-dependent variation in the moisture content of the pellets suggests a formula that incorporates the intricate relationship between temperature and moisture content, which was found throughout the investigation.

$$\text{Pellet Moisture Content} = 0.0027 T^2 - 0.2093 T + 11.603 \tag{10}$$

Table 5. The influence of different pelletization temperature on the resultant moisture content of khaya energy pellets.

Temperature, T (°C)	Pellet Moisture Content (%) *
25	11.47 ± 0.05
50	10.68 ± 0.11
75	10.19 ± 0.04
100	10.01 ± 0.003
125	9.23 ± 0.06

* The values are the means of triplicate measurements followed by the mean standard error (n = 3).

As pelletization temperature increases, it imparts more thermal energy to the biomass material. This elevated thermal energy accelerates the kinetic energy of water molecules within the biomass. This enables a greater number of water molecules to overcome the binding forces that hold them within the biomass. This facilitates the transition of water from a liquid to a gaseous state, leading to its release in the form of water vapor. Higher temperatures result in a more effective process. In addition, the increase in temperature influences the rate of vaporization. This process is driven by the principles of mass transfer, as water molecules move from regions of higher concentration within the biomass to regions of lower concentration in the surrounding environment. Higher temperatures enhance the rate of vaporization due to the heightened kinetic energy of water molecules, resulting in efficient moisture removal from the pellets. As a result, the moisture level in the biofuel pellets diminishes as water transforms into vapor [9,24].

The structure of the pellets is also a contributing factor. Pellets formed at higher temperatures tend to have a more compact and dense structure, which hinders moisture retention. There is less space for water to be held within the pellet matrix, resulting in decreased moisture content. This relationship underscores the importance of controlling pelletization temperature to achieve the desired moisture content. The data presented in the table demonstrate that by carefully adjusting the temperature during pelletization, it is possible to produce pellets with specific moisture content levels. This is significant for ensuring the quality of energy pellets, as lower moisture content generally leads to more efficient combustion and higher energy yield. While higher temperatures are effective in reducing moisture content, there must be a careful balance. Excessively high temperatures can lead to material degradation or undesirable changes in pellet properties. Therefore, the pelletization process must be carefully optimized to achieve the desired moisture content without compromising pellet quality.

The value of R² is 0.97. This high R-squared value indicates a strong linear correlation between the variables under consideration. In statistical analysis, R² is a measure of how well the pelletization temperature (independent variable, in this context) explains the variation in the pellet moisture content (dependent variable). An R² of 0.97 implies that approximately 97% of the variance in the dependent variable can be accounted for by the independent variable, suggesting a robust and well-fitted relationship between the two. This high level of correlation indicates that the independent variable is a strong predictor of the dependent variable, reinforcing the reliability and significance of the relationship.

To evaluate the statistical significance of the results, the researchers conducted an analysis of variance (ANOVA) test. The study's results indicated that there were statistically significant differences in moisture content seen under varying temperature settings. The obtained p -value of 2.07×10^{-7} is below the conventional significance level of 0.05, suggesting that the likelihood of obtaining these findings just by chance is quite limited. The identification of statistically significant disparities and the observation of the minimum moisture content at a temperature of 125 °C offer additional substantiation for the hypothesis that temperature exerts an influence on the moisture content of *Khaya senegalensis* biofuel pellets.

3.7. Comparison with Raw Biomass

The characteristics responses of the pellets, as well as the raw biomass, were thoroughly characterized and are presented in tabulated form in the Table 6 below. The results indicate that pelletizing at different temperatures has a significant impact on key parameters, such as bulk density, moisture content, and calorific value. This observation suggests that the pelletization process plays a crucial role in altering the physical and chemical properties of the biomass.

Table 6. The fuel characteristics comparison between the raw khaya biomass and pellets densified at varied temperature.

	Wood Chips	Pellets (°C)				
		25	50	75	100	125
Bulk density (kg/m ³)	258	451.89	458.70	447.72	447.63	434.65
Moisture content (%)	26.06	11.47	10.68	10.19	10.01	9.23
Ash content (%)	5.38	5.56	5.98	4.26	5.59	6.88
Volatile matter (%)	83.07	84.56	85.42	85.70	86.00	86.40
Fixed carbon (%)	15.46	9.88	8.59	10.04	8.40	6.73
Calorific values (MJ/kg)	16.11	18.98	18.95	19.47	19.13	18.81

Indeed, this discovery marks a significant advancement in the field. With that, the increased interest in biomass for various purposes and the circular economy has resulted in a greater emphasis on reusing, recycling, and upcycling materials. The study by Osman et al. (2019) [25] examined alternative biomass and kinetic modeling methodologies for recycling, reuse, and upcycling processes, motivated by societal desires for sustainable energy and a lower carbon footprint. The circular economy initiatives outlined in the referenced review paper provide valuable insights into the environmental and economic benefits associated with biomass reuse and recycling. Therefore, it is recommended that more research be conducted on this particular matter.

4. Conclusions

In conclusion, this study has yielded crucial insights into the impact of pelletization temperature on the combustion properties of *Khaya senegalensis*-derived fuel pellets. *Khaya senegalensis*, a readily available biomass resource in Malaysia, holds immense promise for renewable energy production. This study aimed to examine the influence of pelletization temperature on the combustion properties of fuel pellets derived from *Khaya senegalensis*. The pelletizing process was carried out at a temperature of 75 °C, resulting in the highest calorific value of 19.47 MJ/kg and the greatest fixed carbon content of 10.04%. Additionally, an evident upward trend in volatile matter was seen with increasing temperature. The pelletization process conducted at a temperature of 75 °C resulted in the attainment of a

significantly desirable low ash content of 4.26%. The fuel qualities of *Khaya senegalensis*-wood-derived fuel pellets were seen to be influenced by temperature. Through meticulous experimentation, we identified that a pelletization temperature of 75 °C stands out as the optimal choice, resulting in several noteworthy thermophysical outcomes. The current findings are expected to provide insight into the *Khaya senegalensis* pelletization process that transforms raw biomass into a highly efficient and convenient form of fuel and enables its widespread use for renewable energy generation. These enhanced qualities make *Khaya senegalensis* an even more attractive candidate for renewable energy generation, furthering its potential to contribute significantly to the sustainable energy landscape. In summary, this research provides valuable guidance for the *Khaya senegalensis* pelletization process, effectively transforming raw biomass into a highly efficient and environmentally friendly fuel source. The results are poised to advance the adoption of *Khaya senegalensis* in renewable energy production, facilitating its widespread use and contributing to the global transition toward cleaner and more sustainable energy sources.

5. Patents

Pelletization Temperature and Pressure Effects on the Mechanical Properties of *Khaya senegalensis* Biomass Energy Pellets—The copyright is held by the Intellectual Property Corporation of Malaysia, with registration number LY2023P04012.

Author Contributions: The methodology was conducted by R.I.I., while the validation process was carried out by C.Y.K. The formal analysis was also performed by R.I.I. The investigation and allocation of resources were conducted by R.I.I. The original draft of the writing was prepared by R.I.I., and the review and editing were performed by C.Y.K. The supervision of the project was provided by C.Y.K. and A.R.M. The project administration was handled by C.Y.K. and A.R.M., and the funding was acquired by R.I.I. All authors have read and agreed to the published version of the manuscript.

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