



## ORIGINAL ARTICLE

# Compositional Analysis of Nipah Palm (*Nypa Fruticans*) Agricultural Wastes for Biochar Production via Self-Purging Slow Pyrolysis

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**ABSTRACT** - Agricultural wastes from Nipah palm are anticipated to rise in the future, particularly in Sarawak, Malaysia, due to its growing popularity and usage. This study utilized Nipah palm's empty fruits, peduncles, and fronds to produce biochar, which were then applied for compositional analysis for characterization. A newly developed compositional analysis by thermogravimetry employing a self-purging method was undertaken in the experiment to measure the moisture, volatile matter, ash, and fixed carbon content of the Nipah palm agricultural wastes. Biochar yield analysis was conducted for different types of Nipah palm agricultural waste. The compositional analysis showed that the agricultural waste from Nipah palm has a moisture content between 6% and 9%, a volatile matter content between 47% and 58%, a fixed carbon content between 27% and 30%, and an ash content between 7% and 16%. The ideal pyrolysis temperature for Nipah palm biochar production and carbonization was found to be 500°C for all three types of Nipah palm agricultural waste. The fronds had the highest biochar yield of 45.8% compared to empty fruit and peduncle. Therefore, compositional analysis via thermogravimetry using self-purging pyrolysis is possible. Nipah palm agricultural waste is a suitable feedstock for self-purging slow pyrolysis as it contains a good amount of volatiles and fixed carbon and can yield more than 39% of added-value biochar. This suggests that the biochar produced from self-purging slow pyrolysis of Nipah palm agricultural waste is a potential economic and environmentally sustainable waste management strategy.

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**INTRODUCTION**

*Nypa fruticans*, widely known as the Nipah palm to the natives of Sarawak, Malaysia, is used to produce Nipah palm sugar, also known as gula apong, which has lately witnessed an increasing demand and popularity in the domestic and worldwide food sectors [1]. Nipah palm could also be utilised to produce bioethanol in the bioenergy sector in addition to the food industry, with its ethanol yield per hectare being competitively comparable to that of maize and sugar cane [2]. Besides, studies show that the biomass of the Nipah palm has the potential to be used as raw material to make activated carbon, which is widely applied as an adsorbent for the removal of heavy metals, ammonia, and CO<sub>2</sub> adsorption [3–5]. Additionally, it has been reported that agricultural waste from Nipah palm may be utilised to produce bio-pellets which may be used as a renewable fuel [6]. Thus, this research employs agricultural wastes from Nipah palm to produce value-added Nipah biochar utilising pyrolysis technology as a sustainable waste management method.

Biomass pyrolysis is defined as the thermochemical degradation of biomass processes carried out at high temperatures, commonly between 300°C to 700°C, to produce biochar, bio-oil, and syngas in an oxygen-limited or inert environment [7]. The three types of pyrolysis are slow or conventional pyrolysis, fast pyrolysis, and flash pyrolysis, which are classed by the rate of heating. Slow pyrolysis is often conducted at

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low temperatures of 500°C to 600°C and at a moderate heating rate, which may improve biochar yield as solid, while fast and flash pyrolysis, on the other hand, is suggested to decrease biochar production in favour of bio-oil or syngas yield [8; 9]. Therefore, slow pyrolysis is adopted in this research to produce Nipah biochar since it enhances biochar yield as biochar is the focus of this study.

Self-purging pyrolysis is defined as pyrolysis without the need for an external purging gas [10]. The primary gases released during biomass pyrolysis are CO, CO<sub>2</sub>, H<sub>2</sub>, CH<sub>4</sub>, and light hydrocarbons [11]. These gaseous by-products from the pyrolysis of biomass are confined within the reactor and serve as the purging gas for self-purging pyrolysis [12]. Studies showed that the self-purging pyrolysis approach has successfully produced biochar, enhanced the production of biochar, and reduced the cost of building a pyrolysis reactor since it does not need an external purging gas [10; 12; 13]. The self-purging slow pyrolysis process, which has a simple design and is inexpensive, is applied in this study to convert agricultural waste from Nipah palm into value-added biochar. Biochar is a carbon-rich solid material that is produced by pyrolyzing biomass, typically at temperatures between 300°C to 700°C and in an environment with little to no oxygen [14; 15]. Besides, pyrolysis feedstock and pyrolysis conditions, such as temperature and residence time, significantly impact the biochar characteristic [16]. Therefore, it is necessary to do a preliminary study on the pyrolysis feedstock and parameters to identify whether the biochar produced is suited for a certain application. The usage of biochar is rising in popularity as an environmentally friendly material and the primary carbon sink applications, including agronomy, animal farming, anaerobic digestion, composting, environmental remediation, building materials, and energy storage, as reviewed by Osman et al. [17]. This study employs agricultural waste from Nipah palms as feedstock for self-purging slow pyrolysis to produce biochar, which will then be examined to understand more about the feedstock properties.

Thermogravimetric analysis (TGA) is the method used to analyse the weight change of a sample as a function of temperature or time in a specific environment, which may be inert or oxidative, at a regulated heating rate [18]. TGA is typically used in combination with other analysis methods such as compositional analysis for fast characterization to identify the biomass's moisture, volatile, ash, and fixed carbon contents [19; 20]. To provide an inert environment, self-purging pyrolysis was employed in this study to develop a compositional analysis by thermogravimetry of biomass without the necessity of an external purging gas.

The purpose of this research is to prepare and characterize the material properties of the Nipah palm agriculture wastes as feedstock for self-purging slow pyrolysis to produce biochar. In addition, this study attempts to provide a compositional analysis of biomass adopting the thermogravimetry method, which involves self-purging pyrolysis to provide an inert environment for the TGA experiment. Besides, this study aims to provide the ideal temperature for carbonizing Nipah biomass by examining TGA profiles and the biochar yield at the carbonizing temperature for the empty fruit, peduncle, and frond waste from the Nipah palm agricultural wastes.

## **MATERIALS AND METHODOLOGY**

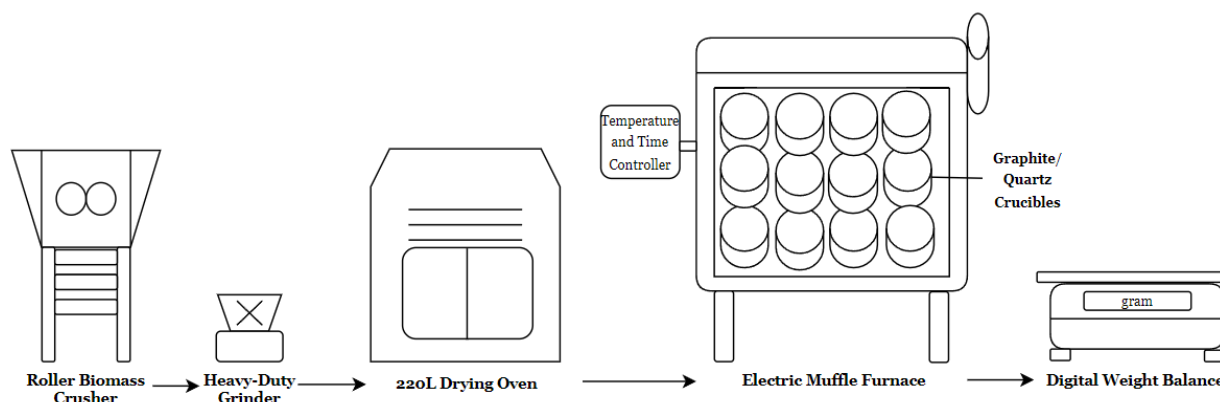
### **Materials and Equipment**

Nipah palm agricultural wastes, specifically its empty fruits, peduncles, and fronds, are obtained and collected from the mangrove forest located in Semop, Sarawak, Malaysia. Figure 1 shows the Nipah palm agricultural wastes utilized in the experiment.



**Figure 1.** Nipah agricultural wastes as feedstock were used for the experiment.

Equipment used in the experiment includes a 220L drying oven, roller biomass crusher, heavy-duty blender, control digital weight balance, graphite crucible, quartz crucible, and Nabertherm electric muffle furnace. It is equipped with a temperature and time controller to adjust the temperature, heating rate, and temperature holding duration. Both the graphite and quartz crucibles with lids were utilized as self-purging pyrolysis reactors, while the muffle furnace served as the heat source for biochar production and characterization. Figure 2 shows the schematic diagram of the equipment used in this study.



**Figure 2.** Equipment used in this study.

### Nipah Palm Agricultural Wastes Preparation

Nipah palm agricultural waste was collected, cleansed with tap water to eliminate contaminants, dried under sunlight, and crushed into smaller pieces. The crushed Nipah palm samples were then dried in an oven at 110°C for 24 hours to reduce moisture. This facilitated further grinding to turn the samples into a powder and short fibres (<2 mm) for biochar production and characterization. The sample preparation process is shown in Figure 3.



**Figure 3.** Samples preparation for characterization and biochar production.

### Compositional Analysis of Biomass by Thermogravimetry Method

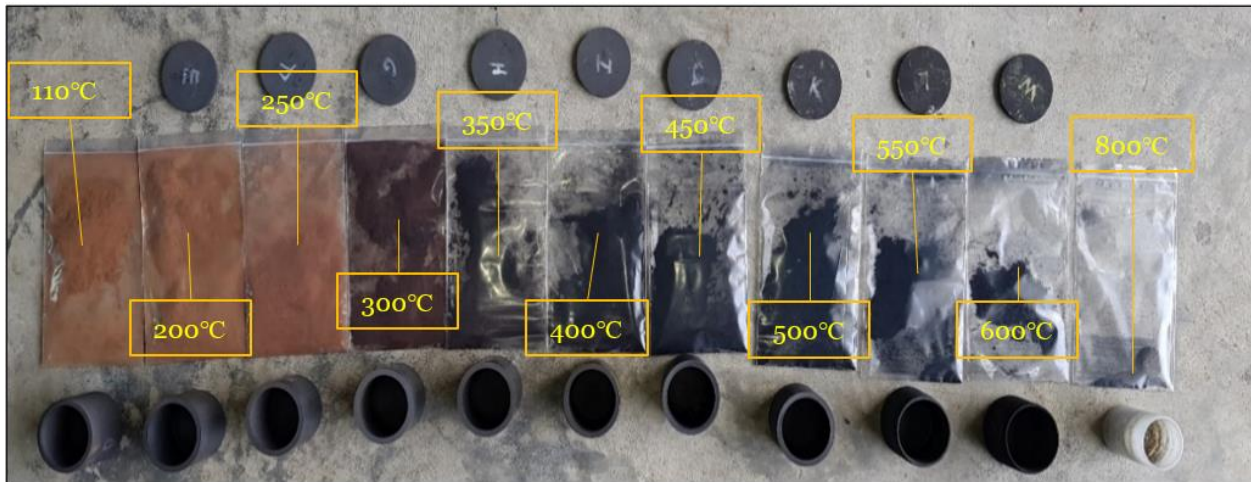
The samples were subjected to compositional analysis employing the thermogravimetry analysis (TGA) method with a regulated heating rate to characterize the Nipah palm samples. A muffle furnace served as the heat source. However, the TGA utilized a modified approach based on ASTM E1131, where self-purging is employed, and no external purging gas is required to provide an inert heating environment.

The characterization of Nipah palm agricultural waste biomasses involved weighing the samples with an initial weight of 10g to measure their weight loss while being heated at a controlled rate of 5°C/min from 30°C to 800°C. Initially, the samples were heated in the crucibles without lids to allow moisture to evaporate at temperatures of 30°C to 110°C, with a holding duration of 15 minutes at 110°C. Next, the samples were heated in the crucible with the lid on using the self-purging pyrolysis process at temperatures of 110°C to 600°C for the removal of volatile matter and carbonization. To allow combustion and produce ash, the samples were further heated in the crucibles without lids to create an oxidizing environment at temperatures of 600°C to 800°C, with a holding duration of 3 hours at 800°C.

The moisture, volatile matter, and ash contents of Nipah palm agricultural waste samples were determined by interpreting the weight losses from the thermogravimetric curve obtained following the recommended ASTM E1131 procedure. Fixed carbon content is then calculated by subtracting the sum of the percentages of volatile matter, ash, and moisture from 100% [21]. The formula in equation (1) is used to calculate the fixed carbon content of the samples.

$$\text{Fixed carbon} = 100\% - (\text{moisture content} + \text{volatile content} + \text{ash content}) \quad (1)$$

Furthermore, the obtained thermogravimetric profiles were utilized and evaluated to determine the ideal pyrolysis temperature for carbonizing the agricultural waste from the Nipah palm's empty fruit, peduncle, and frond. This involved finding the temperature at which the samples had released most of their moisture and volatiles, leaving behind a carbon-rich residue that formed biochar in a non-oxidative environment. Figure 4 shows the Nipah samples at temperatures of 110°C, 200°C, 250°C, 300°C, 350°C, 400°C, 450°C, 500°C, 550°C, 600°C, and lastly 800°C. There were two holding times: 15 minutes for drying and allowing evaporation at 110°C and 3 hours at 800°C for combustion to produce ash.



**Figure 4.** Nipah sample by products collected at different temperatures after undergoing the thermal degradation process for TGA analysis.

## Biochar Production and Yield Analysis

Nipah palm biochar was produced by carbonizing Nipah palm samples in a closed crucible. A sample mass of 35g (filling 3/4 of the reactor) was used with the self-purging slow pyrolysis technique. The heating rate was 5°C/min, with a 15-minute holding time at the ideal pyrolysis temperature (determined by referencing the TGA profile obtained). The yields of Nipah biochar from peduncles, fronds, and empty fruits were investigated and compared. Equation (2) was used to calculate the yield of biochar.

$$Y = \frac{m_f - m_i}{m_s} \times 100\% \quad (2)$$

where  $Y$  is the biochar yield [%],  $m_f$  is the mass of the crucible with the sample at the end of the pyrolysis reaction [g],  $m_i$  is the mass of the crucible without the sample after the pyrolysis reaction [g],  $m_s$  the initial mass of the sample before the pyrolysis reaction [g].

## RESULTS AND DISCUSSION

### Compositional Analysis by Thermogravimetry

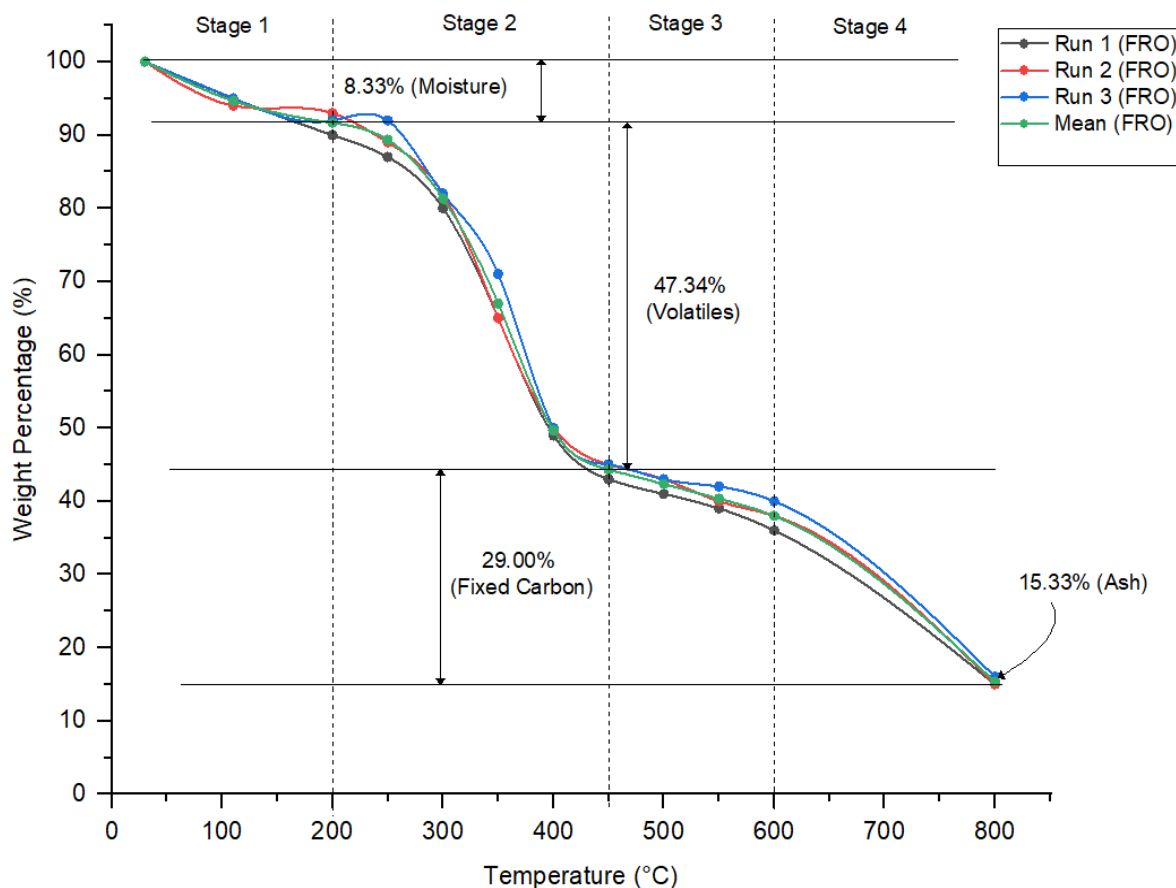
The data collected through compositional analysis by the thermogravimetry method of the Nipah palm samples were used to create graphs that visually illustrate the relationship between the weight percentage of the samples and temperature to determine the sample's moisture, volatile matters, ash, and fixed carbon content. Furthermore, the thermogravimetric profiles produced were utilized to determine the ideal temperature range for complete carbonization to produce Nipah biochar with a satisfactory yield percentage. Figures 5-7 present the TGA profiles of Nipah palm empty fruit, peduncle, and frond. To ensure data reliability, the experiment was conducted in triplicate (denoted as Run 1, Run 2, and Run 3). The "mean" data point represents the average value calculated from these three independent runs, reflecting the moisture, volatiles, fixed carbon, and ash content within the samples.

The compositional analysis for the Nipah palm frond was shown in Figure 5. The initial weight loss of the Nipah palm frond can be observed in Figure 5, occurring at temperatures ranging from 30°C to 200°C (Stage 1). The weight loss at this temperature is attributable to the removal of Nipah palm frond moisture and light volatile content since water evaporates at 100 °C and drying of biomass typically occurred at about 100 °C to 200 °C [22; 23]. The Nipah palm frond has a moisture content of 8.33%, as shown in Figure 5.

Furthermore, a major weight loss of 47.34% is observed at temperatures of 200 °C to 450 °C (Stage 2). During this stage of the pyrolysis process, highly volatile matter, such as syngas, was released from the frond of the Nipah palm. Additionally, it is shown that starting at temperatures of 450 °C to 600 °C (Stage 3), the rate of mass loss is relatively minimal, suggesting that the majority of the Nipah palm frond's volatile matter has been removed and the frond has mostly carbonized, where carbon is now its primary component.

Moreover, the carbon is burnt in an oxidizing environment at temperatures of 600 °C to 800 °C (Stage 4), leaving a 15.33% mass residue, which corresponds to the ash content of the Nipah palm frond. The Nipah palm's frond has an ash content comparable to the study by Tamunaidu and Saka (2011) and Evelyn et al. (2022), which were 11.7% and 16.5%, respectively [24; 25].

Since the contents of moisture, volatile matter and ash are known, the fixed carbon is determined using the mass difference, as shown in Figure 5, which shows that the frond of the Nipah palm contains 29% of fixed carbon content. Finally, the TGA profile results showed that the ideal temperature for producing Nipah palm frond biochar is 500 °C. This is because according to the TGA profile, Nipah palm frond loses most moisture and volatile components at 500 °C, resulting in a carbon-rich residue known as biochar.



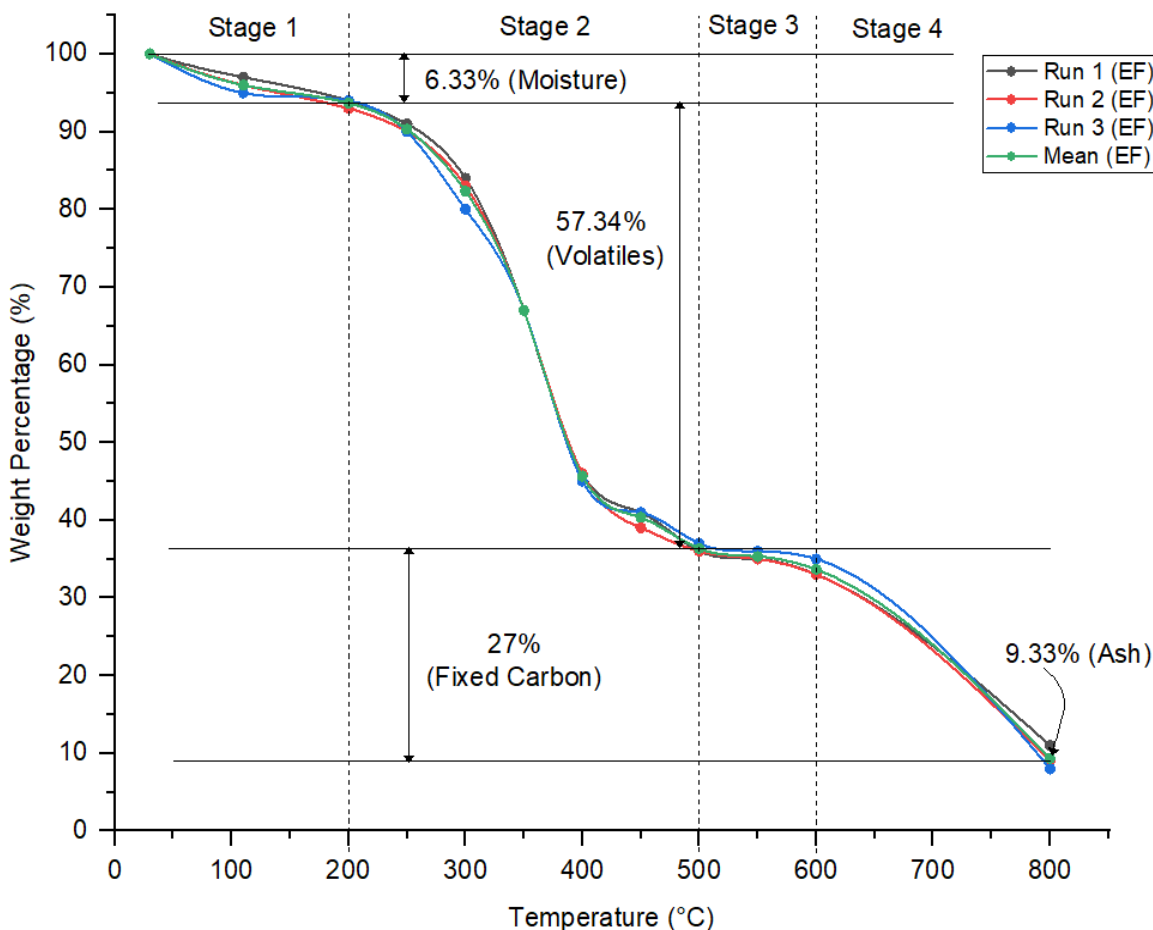
**Figure 5.** Compositional analysis by thermogravimetry analysis (TGA) of the Nipah palm's frond.

The compositional analysis for the Nipah palm's empty fruit is shown in Figure 6. Figure 6 depicts the initial weight loss of the empty fruit at temperatures ranging from 30 °C to 200 °C (Stage 1). This weight loss is attributed to the elimination of light volatile matter, such as moisture, since water evaporates at 100 °C, and drying of biomass typically occurs between 100 °C and 200 °C [22; 23]. As shown in Figure 6, the moisture content of the empty Nipah palm fruit is 6.33%.

Major weight loss is observed at temperatures ranging from 200 °C to 500 °C (Stage 2), with a loss of 57.34%. Devolatilization occurs in this stage, where volatiles like syngas are removed from the empty fruit during the pyrolysis process. It is further revealed that starting at 500 °C and reaching 600 °C (Stage 3), the rate of mass loss is very low. This suggests that most of the volatile matter has been removed, leaving the empty fruit mostly carbonized, with carbon as the main component.

Furthermore, in an oxidizing environment at temperatures ranging from 600 °C to 800 °C (Stage 4), the remaining carbon combusts, resulting in a mass residue of 9.33%. This mass residue represents the ash content of the empty Nipah palm fruit. The ash content is slightly higher than what Tamunaidu and Saka (2011) reported (8.2% for fruit shell and 8.1% for fruit husk) [25].

Since the moisture, volatile matter, and ash content are known, the fixed carbon content is determined by mass difference. As shown in Figure 6, the empty fruit of the Nipah palm contains 27% fixed carbon. Finally, the TGA profile results indicate that the ideal temperature for producing Nipah palm empty fruit biochar is 500 °C. At this temperature, the empty fruit loses most moisture and volatile components, resulting in a carbon-rich residue known as biochar.



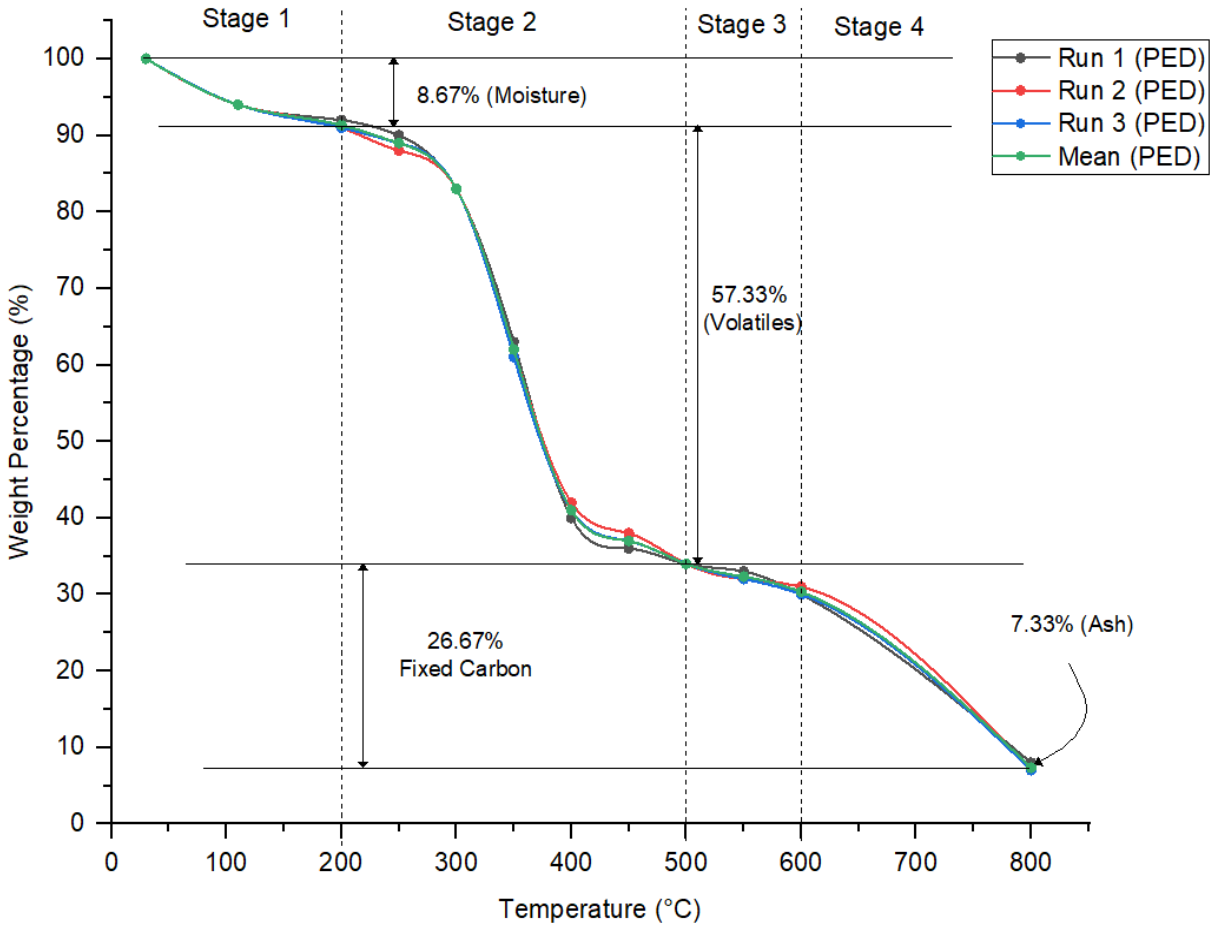
**Figure 6.** Compositional analysis by thermogravimetry analysis (TGA) of the Nipah palm's empty fruit.

The compositional analysis of the Nipah palm's peduncle is shown in Figure 7. Figure 7 shows the initial weight loss of the Nipah palm peduncle at temperatures ranging from 30 °C to 200 °C (Stage 1). This weight loss is due to the removal of the Nipah palm peduncle's light volatiles, such as moisture. Water evaporates at 100 °C, and biomass drying typically occurs at around 100 °C to 200 °C [22; 23]. As shown in Figure 7, the moisture content of the Nipah palm peduncle is 8.67%.

Further, drastic weight loss is observed at temperatures ranging from 200 °C to 500 °C (Stage 2), with a weight loss of 57.33%. This weight loss represents the volatile matter content of the Nipah palm peduncle as devolatilization occurs in this stage. Volatiles such as syngas are released from the peduncle during the pyrolysis process at this stage. Starting at 500 °C and reaching 600 °C (Stage 3), the rate of mass loss becomes very low. This signifies that most of the volatile matter has been removed from the Nipah palm peduncle, and carbon is the major remaining component, indicating the peduncle has undergone carbonization.

Furthermore, in an oxidizing environment with temperatures ranging from 600 °C to 800 °C (Stage 4), the carbon is combusted, leaving a mass residue of 7.33%. This mass residue constitutes the ash content of the Nipah palm peduncle. After determining the moisture, volatile matter, and ash content, the fixed carbon content is calculated by mass difference, as illustrated in Figure 7. The analysis shows that the Nipah peduncle has a fixed carbon content of 26.67%.

Finally, the TGA profile obtained suggests that the ideal temperature for pyrolysis to produce Nipah palm peduncle biochar is 500 °C. This is because according to the TGA profile, the Nipah peduncle loses most moisture and volatile components at 500 °C, resulting in a carbon-rich residue known as biochar.



**Figure 7.** Compositional analysis by thermogravimetry analysis (TGA) of the Nipah palm’s peduncle.

The Nipah palm frond, peduncle, and empty fruit shell exhibited similar moisture content, ranging from 6.33% to 8.67%. However, the peduncle and empty fruit shell displayed a higher volatile matter content (57.33% and 57.34%, respectively) and lower ash content (7.33% and 9.33%, respectively) compared to the frond (47.34% volatile matter and 15.33% ash content). Notably, the frond possessed the highest fixed carbon content (29%), while the peduncle and empty fruit shell exhibited marginally lower values (26.67% and 27%, respectively). Previous study shows that Nipah palm samples ( frond, shell, leaf and husk) has a cellulose and hemicellulose contents were 8.9% to 45.6% and 21.8% to 26.4%, respectively and lignin content was 19.4% to 33.8% while having ash content from 5.1% to 11.7% [25]. This supports current studies as cellulose, and hemicellulose are major contributors to volatiles due to their decomposable nature, while lignin's high fixed carbon content is associated with biochar formation, other factors like conversion process and temperature also significantly influence the final biochar yield [26; 27].

**Table 1.** Nipah palm agricultural waste composition comparison

Biomass Sample	Moisture Content (%)	Volatile Matter Content (%)	Fixed Carbon Content (%)	Ash Content (%)
Nipah Palm Frond	8.33	47.34	29.00	15.33
Nipah Palm Peduncle	8.67	57.33	26.67	7.33
Nipah Palm Empty Fruit Shell	6.33	57.34	27.00	9.33

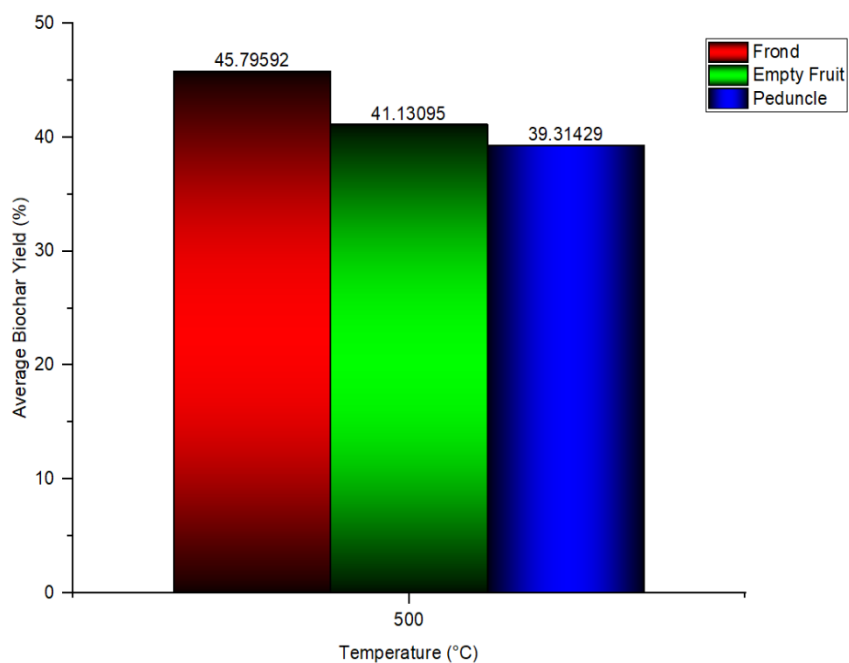


## Different Types of Nipah Palm Agriculture Waste Biochar Yield

Nipah palm agricultural wastes, including empty fruit, peduncle, and fronds, were carbonized in closed crucibles with 35 g of sample mass using the self-purging slow pyrolysis technique. The heating rate was 5°C/min, with a 15-minute holding time at the pyrolysis temperature of 500°C. The total pyrolysis process took 1 hour and 50 minutes.

This temperature (500°C) was chosen because it is ideal for completely carbonizing the three different parts of Nipah palm agricultural waste, as suggested by the TGA profile in Figures 5 to 7. The Nipah biochar yield produced from different types of Nipah palm agricultural waste is compared in Figure 8.

Figure 8 compares the biochar yields of the three Nipah palm waste types (fronds, peduncle, and empty fruit) under identical pyrolysis conditions. Among these types, the frond yielded the highest biochar (45.8%), while the peduncle yielded the lowest (39.31%) and the empty fruit resulted in an intermediate yield (41.13%). This difference is due to the fact that the frond has a higher fixed carbon content and lower volatile content compared to both the peduncle and empty fruit. The amount of biochar yield is closely dependent on the composition of the biomass, particularly the amount of fixed carbon it contains [28].



**Figure 8.** Average biochar yield of Nipah palm frond, empty fruit, and peduncle agricultural waste under the same pyrolysis conditions.

## CONCLUSION

In conclusion, the experiment successfully demonstrates that compositional analysis via thermogravimetric analysis (TGA) using self-purging pyrolysis is technically feasible. This approach successfully determined the moisture, volatile matter, ash, and fixed carbon content of biomass samples obtained from Nipah palm fronds, empty fruit, and peduncle agricultural waste.

Furthermore, the analysis using TGA employing self-purging slow pyrolysis revealed that the agricultural waste from Nipah palm has a moisture content ranging from 6% to 9%, a volatile matter content between 47% and 58%, a fixed carbon content of 27% to 29%, and an ash content of 7% to 16%. Among the samples, the Nipah palm frond has the highest ash (15.33%) and fixed carbon content (29%). Additionally, the Nipah palm peduncle has the highest moisture content (8.67%), while the empty fruit has the highest volatile matter content (57.34%).

The TGA profiles produced indicate that the ideal pyrolysis temperature for Nipah palm biochar production is 500°C for the feedstock. Nipah palm frond proves to be the most suitable feedstock, achieving a high biochar yield of 45.8% under optimal conditions: 500°C pyrolysis temperature, 15 minutes residence time, and a total process time of 1 hour and 50 minutes.

Therefore, Nipah palm biomass emerges as a promising feedstock for biochar production due to its high fixed carbon content and substantial biochar yield. The biochar can be utilized for various adsorption applications, including water treatment and carbon capture. To further enhance its performance, activation of the produced biochar through chemical or physical methods is recommended to optimize its surface area and porosity distribution for specific applications like water treatment or carbon capture.

## ACKNOWLEDGEMENT

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