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ORIGINAL ARTICLE

Effect of Different Parameters of Modified Microwave-assisted Hydrothermal Liquefaction Process for Conversion of Palm Kernel Shells to Bio-oil

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ABSTRACT Microwave-assisted hydrothermal liquefaction (HTL) is a thermochemical process that uses microwaves to transform biomass into bio-oil, charcoal, and biogas. It is an alternate technique to conventional heating. The utilization of microwave-assisted HTL exhibits superior speed and efficiency in manufacturing products as compared to traditional heating methods. Palm kernel shells (PKS) are a type of biomass that is effectively transformed by HTL using a modified microwave oven into bio-oil, biochar, and biogas. A domestic microwave oven with a power output of 800 W was utilized to facilitate the conversion of PKS into bio-oil, charcoal, and biogas. This study was replicated using several parameters, including duration, temperature, and power, in order to achieve a maximal bio-oil yield. The study reveals that the bio-oil production obtained using microwave-assisted pyrolysis at temperatures of 350°C and 400°C ranges from 10.70 wt% to 25.60 wt% across pyrolysis durations of 6, 9, and 12 minutes. These findings demonstrate the potential of microwave-assisted HTL as an efficient method for bio-oil production from PKS.

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INTRODUCTION

Currently, the globe is experiencing an energy crisis due to the depletion of non-renewable resources including coal, natural gas, and petroleum fuel. Nations are now prioritizing renewable energy and formulating strategies to cultivate and harness its attributes to ultimately substitute non-renewable energy sources. Examples of renewable energy sources include biomass, solar, wind, hydro, and geothermal energy.

The palm kernel shell (PKS) is an endocarp that serves as a protective covering for the palm kernel, which is also referred to as the oil palm shell. The product incorporates waste management by utilizing palm nut crushing in palm oil mills to extract palm oil. The product is a rather plentiful waste material in Southeast Asia, particularly in Malaysia and Africa [1]. The kernel shell can have several shapes depending on the method of extraction or nut shattering, including irregular, angular, circular, or polygonal. PKS features a face for the concave and convex parts of the shell, and its thickness ranges from 0.15 mm to 8 mm depending on the tree species.

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Malaysia is well known for being one of the world's leading producers of oil palm. The development of the oil palm trade in the country may be traced back to 1871, when British Malaysia commenced the importation of seedlings intended for ornamental plants. In 1971, the oil-palm business saw its commercialization launch at Tenamaran Estate in Selangor, leading the way in Malaysia. The plant's original species, *Elaeis guineensis*, or African Oil-Palm, is the source of the seeds and has historically formed the backbone of the country's agroindustry [2]. The country's governing bodies launched the agricultural diversification programme in the early 1960s to boost economic growth, as the oil and palm plantation industry had experienced significant expansion following Malaya's independence. Reducing the nation's overall reliance on the two industries that propel the country tin and rubber is the initiative's primary goal [2].

Generally, hydrothermal liquefaction (HTL) is the process of converting biomass into liquid fuel by thermochemical means from waste agricultural materials, such as wood pellets from crops or manure from livestock, into liquid fuels by processing the material at high temperatures and pressurized liquid environments of between 4 MPa and 22 MPa, respectively. This results in liquid bio-crude that is viscous, black in colour, and smells like smoke. HTL breaks down biomass molecules by using its superheated characteristics to lower mass transfer resistances and increase solvent penetration of the biomass structure [3]. For feedstock processing, HTL is preferred over pyrolysis because it uses less energy than pre-dry or in-process evaporation for water removal. Water is also the primary catalyst, which helps in the process of separating the more polar by-product stream from the oil product stream [3; 4].

Bio-oil is a liquid that is produced by either HTL or pyrolyzing biomass. Compared to oil produced by pyrolysis, the oil produced by the HTL method has a good quantity of heating value but low oxygen and humidity levels. Because raw bio-oil is of lower quality and has a complex mixture of oxygenated molecules, it must be upgraded or refined further to produce chemicals or fuels with higher added values [5]. One of the three principal outputs of the HTL process is biogas, also known as non-condensable gas. Upon cooling to room temperature, certain involatile hydrocarbons obtained from the HTL technique reverted to their liquid condition. The oil residue contains traces of nitrogen, oxygen, hydrogen, and a small number of carbon dioxide particles that are released in their gaseous form. Nitrogen gas is utilized prior to liquefaction to draw out oxygen in the reactor and prevent a rise in the product's oxygen content. In the same way that the liquid percentage increases with temperature, so does the char production gradually rise while the gas production rises [6]. Often referred to as char, biochar is a solid byproduct of the HTL process that is created at lengthy residence times and extended polymerization reactions of the oil fraction. Because biochar retains soil moisture and nutrient levels, it is frequently employed in soil rehabilitation, leading to high-quality soil. Farmers use biochar to lessen the problem of heavy metal traces in their polluted soils [7].

The process of converting biomass from its natural state to a liquid four state takes the same amount of time as fossil fuels, which took a century to transform biomass into oil and natural gas. Without going through a conversion process, biomass has a low density, a non-homogenous form, and high moisture content. The conversion of biomass into liquid was made possible by a number of thermochemical processes, which also make the material easier to handle, distribute, and contain. Pyrolysis is a process that was originally created to turn biomass into oils and make methanol, acetic acid, and acetone from wood. HTL is an additional option that has been selected for this study. Because it uses less energy and doesn't require drying the feedstock, this technique is preferable. The primary objective of this study is to analyse the impact of various parameters on the extraction of bio-oils from palm kernel shell. Additionally, the study aims to evaluate the yield of bio-oil produced and characterize the bio-oil production utilizing microwave-assisted heat transfer liquefaction (HTL) and conventional heating methods.

MATERIALS AND METHODOLOGY

Palm Kernel Shell

Palm kernel shells (Figure 1) were used as the waste sample in this study. When considering other biomass residues, PKS low-moisture content makes them extremely commendable. When compared side by side, the sample's increased heating value results from the presence of traces of palm oil, which sets it apart from another conventional lignocellulosic biomass. Palm kernel shells produce high-quality biomass fuel because of their homogeneous size distribution, ease of handling, ease of crushing, and low moisture content, which limits biological activity [8].

Figure 1. The Palm Kernel Shells Sample

Microwave Assisted Rapid Hydrothermal Liquefaction Process

Figure 2 displays the diagram of the experimental setup for HTL which utilized microwave assistance. A Panasonic NN-ST342M microwave oven, with a maximum power output of 800W and operating at a frequency of 2450MHz, was adapted to facilitate the drying and pyrolysis of agricultural waste. A hole with a diameter of 40 mm was drilled at the top center of the microwave oven. A quartz reactor, spanning 40 mm in diameter and 250 mm in height, with a C24/29 socket size, was affixed to the microwave oven and supported by a retort stand. The sample temperature was measured using a thermocouple from EXTECH, USA. A T-shaped connector equipped with a C24/29 socket was utilized to connect the gas outlet of the quartz reactor to the Liebig condenser, which was installed with a water-cooling system. The Liebig condenser was linked to the three-necked collecting flask using an L-shaped connector with a C24/29 joint. The experiment involved introducing minute particle samples into the quartz reactor and subsequently placing them in the microwave oven. Stringent protocols and safety measures were implemented to avert the escape of microwave radiation into the environment. Figure 3 depicts the modified microwave-assisted experimental set up for HTL process conducted in the laboratory.

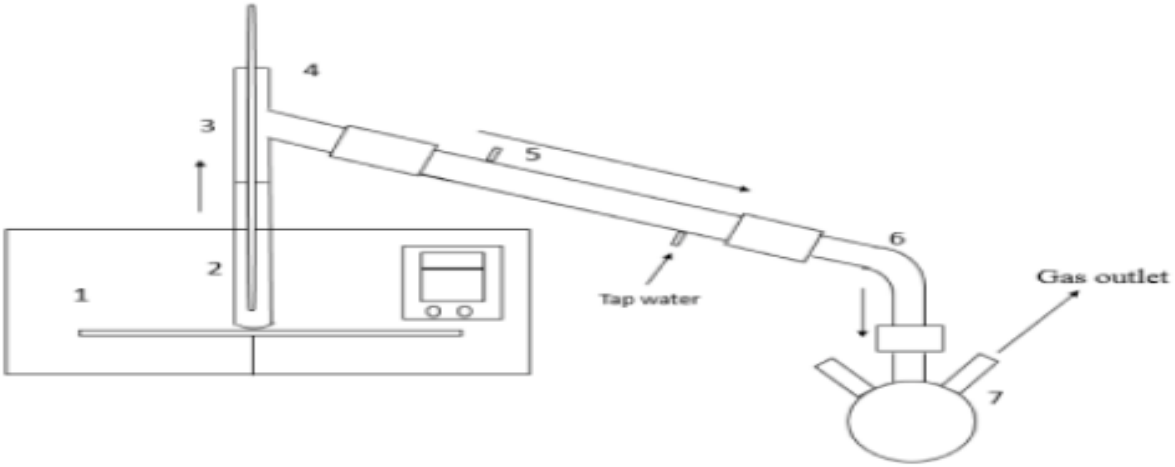

Figure 2. Schematic of HTL experiment of Palm Kernel Shell supported by microwave irradiation (1-Microwave oven, 2-Quartz Reactor, 3-Thermocouple, 4-T-shaped connector, 5-Liebig Condenser, 6-Lshaped connector, 7-3 necks collecting flask).

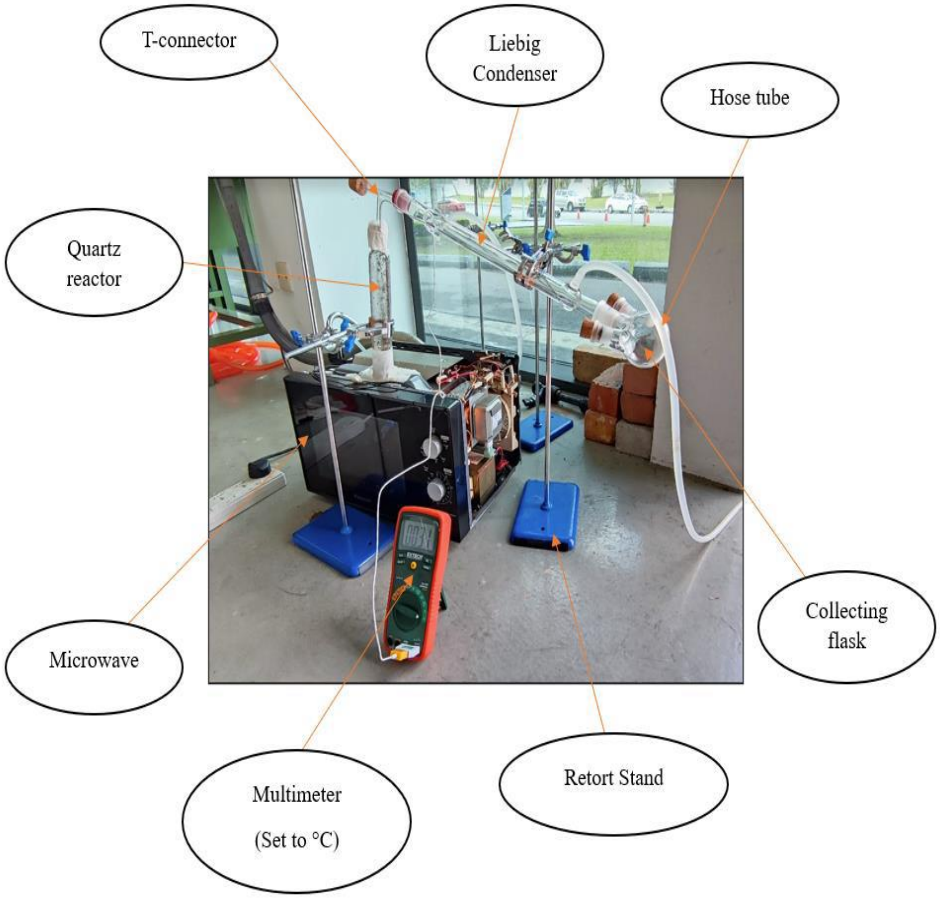

Figure 3. Modified Microwave-assisted Experimental set-up for HTL Process

The PKS sample was fragmented into small particles and introduced into the quartz reactor. In order to prevent gas leakage during the pyrolysis process, aluminum foil was used to cover all the holes of the apparatus. The experiment involved introducing 20 grams of PKS samples into a quartz reactor and placing them inside a modified microwave oven. The ideal power level for this experiment is 800 W. The experiment was replicated three times, with each pyrolysis procedure lasting 6 minutes, 9 minutes, and 12 minutes respectively. The volatile substance produced was collected in a three-neck collecting flask to obtain the bio-oil, and the equipment was rinsed with dichloromethane. The carbonaceous residue, sometimes known as char, was recovered directly from the quartz reactor. The biochar and bio-oil yields were determined based on their respective weights, whereas the gas yield was computed by measuring the weight difference.

Conventional Heating Pyrolysis

Figure 4 and Figure 5 represents the diagram and arrangement for traditional heating pyrolysis respectively. A 250 ml flask made of borosilicate glass with a round bottom was connected to a T-shaped connector and placed on a 360 W heating mantle. In order to measure the temperature within the flask, a thermocouple connected to a digital multimeter (EXTECH, USA) was put into the top of the T-shaped connector. In addition, a Liebig condenser was attached to a T-shaped connector. The outlet of the Liebig condenser is attached to both an L-shaped connector and a collecting flask. Similarly, the gas vapor that moves from the flask to the Liebig condenser is condensed using a water-cooling system, just like in the modified microwave. The collecting flask accumulates oil that has condensed, and its weight is measured and documented.

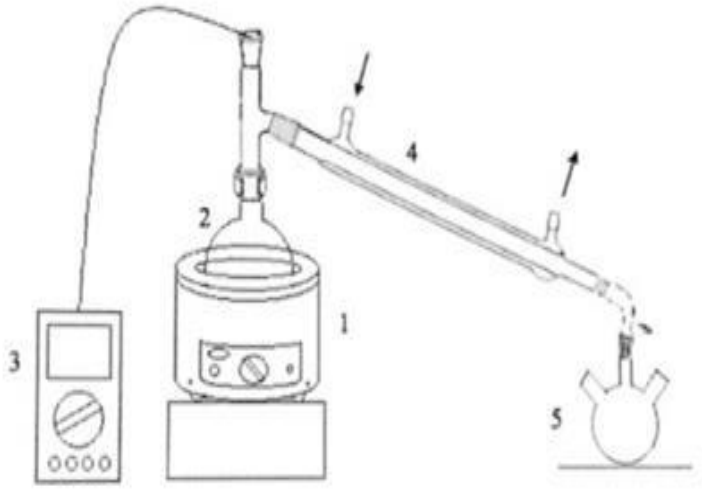

Figure 4. Schematic Heating of Palm Kernel Shell by conventional heating (1-Heating mantle, 2-250ml flask, 3-Multimeter, 4-Liebig Condenser, 5-250ml collecting flask.

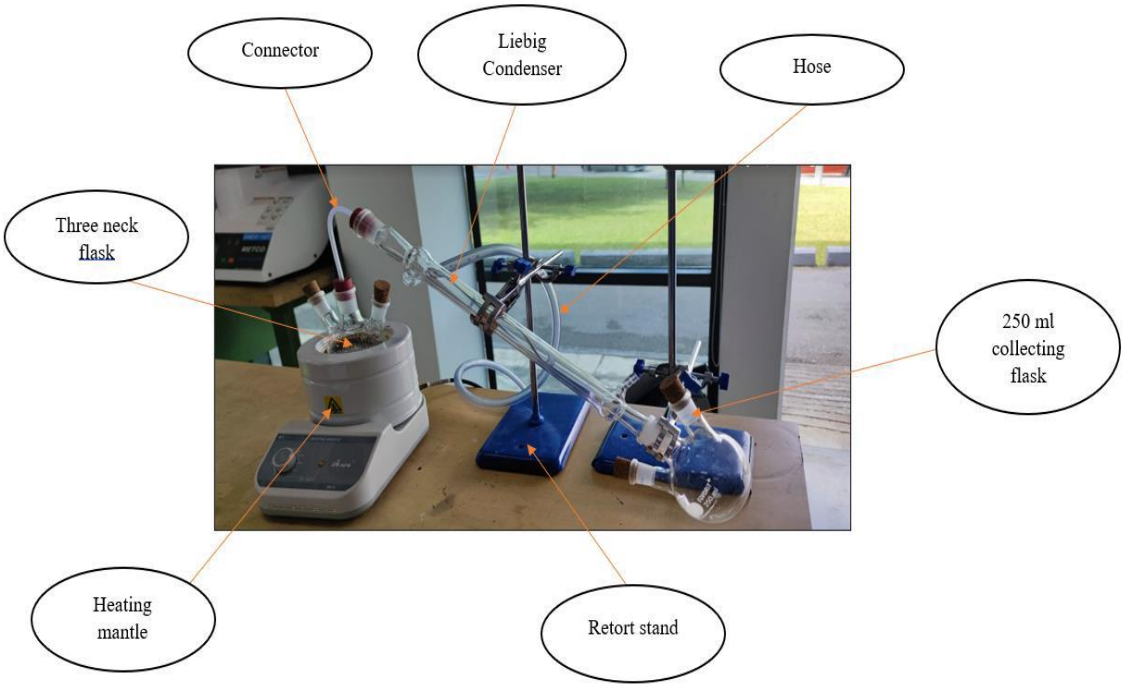

Figure 5. Experimental setup of conventional heating process

20 g of PKS sample feedstock was added to the 250 ml round bottom flask and placed on top of the heating mantle. A digital multimeter thermocouple was attached from the top to the T-shaped connector and securely closed to monitor the temperature within the flask. The heating mantle was then configured to a maximum level of 10, which can be adjusted to achieve the required temperature. In this study, conventional pyrolysis temperatures were established at 350°C, 400°C, and 450°C, with conventional pyrolysis times of 30 minutes, 40 minutes, and 50 minutes for each temperature. Volatile chemicals were extracted and condensed in the collecting flask, and the weight of the resulting bio-oil was measured.

Yield calculation of Bio Oil, Bio Char and Biogas

The mass of PKS feedstock samples was measured before starting the experiment, while the mass of the bio-oil yielded at the end. The following Eq. (1) - (3) [9-11] were adopted to calculate yield of Bio Oil, Biochar and Biogas.

$$\textit{Bio-oil yield: } Y_{bio\text{-}oil} = m_{bio\text{-}oil}/m_{sample} \times 100\% \qquad (1)$$

Where: $m_{bio\text{-}oil}$ *= mass of the bio-oil produced after pyrolysis process,* m_{sample} *= mass of the biomass feedstock before starting of pyrolysis process*

$$\textit{Biochar yield: } Y_{biochar} = m_{biochar}/m_{sample} \times 100\% \qquad (2)$$

Where: $m_{biochar}$ *= mass of the bio char obtained from pyrolysis process,* m_{sample} *= mass of the biomass feedstock before starting of pyrolysis process*

$$\textit{Biogas yield: } Y_{biogas} = 100\% - Y_{bio\text{-}oil} - Y_{biochar} \qquad (3)$$

RESULTS AND DISCUSSION

Physical appearance of bio-oil and biochar

The bio-oil was recovered from PKS using a microwave-assisted hydrothermal liquefaction method and conventional heating. Microwave aided hydrothermal tests were carried out at 350°C and 450°C for periods of 6 minutes, 9 minutes, and 12 minutes. In the standard heating procedure, the temperatures are 350°C, 400°C, and 450°C, with pyrolysis times of 30 minutes, 40 minutes, and 50 minutes, respectively. The bio-oil collected from both conversion techniques is bright orange in colour, while the biochar is black. Figure 6 and Figure 7 show specimens of bio-oil and biochar collected throughout the experiment.

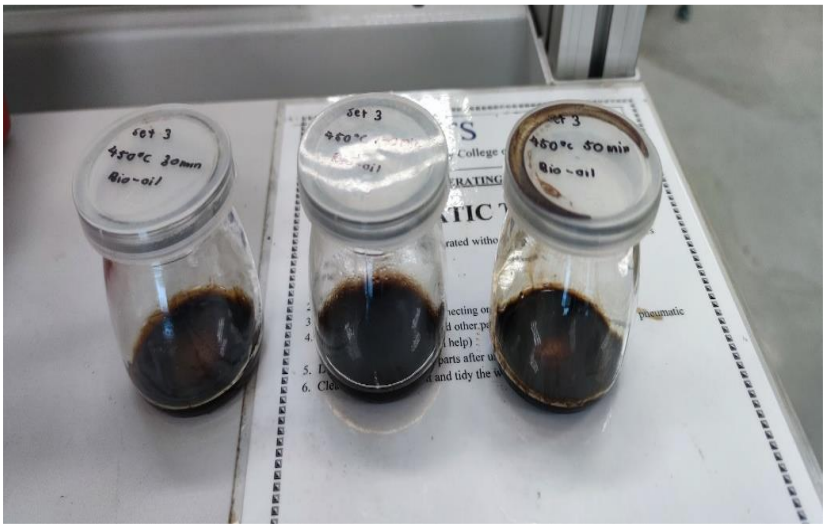

Figure 6. Samples of bio-oil obtained from experiment.

Figure 7. Samples of biochar obtained from experiment.

Yield of different fraction

Each run of the experiment utilized 20-gram samples of Palm Kernel Shells (PKS) for all processes. Each of the following subjects are centered on the production yield of the good generated from 20g of the Palm Kernel Shell (PKS) and their respective procedures. The total product yield of Palm Kernel Shell is discussed and provided in Tables 1 and 2. Collecting and calculating biogas results proved to be a complex process due to the difficulty in capturing the vapor during conversion. Therefore, the percentage yield of biogas was computed by subtracting the combined yield of bio-oil and biochar from 100 percent. In the case of bio-oil and biochar, the amount of raw material remains constant, allowing for direct measurement.

HTL under microwave heating

The percentage yields of bio-oil derived from Palm Kernel Shells are displayed in Table 1 and Figure 8. Table 1 presents the data obtained from the measurement and analysis of the bio-oil yield at temperatures of 350°C and 450°C during the pyrolysis of Palm Kernel Shells with the use of microwave heating. The maximum bio-oil yield is 25.60 wt% achieved at a temperature of 450°C after 9 minutes, whereas the minimum recorded bio-oil yield is approximately 10.70 wt% obtained at a temperature of 350°C after 6 minutes. Another by-product produced during hydrothermal processes is biochar, which refers to the carbon-rich residue, along with the generation of biogas. Each of these products had a peak yield of 49.35 wt% and 54.65 wt% after 6 and 9 minutes, respectively, at a constant temperature of 350°C. Figure 7 readily shows that the yield of bio-oil gradually rose as the pyrolysis temperature increased. The pyrolysis temperature of 350°C resulted in a yield ranging from 10.70 wt% to 21.30 wt%, whereas the hydrothermal temperature of 450°C yielded between 17.65 wt% and 25.60 wt%. Both temperatures exhibit the maximum percentage yield while the raw materials are subjected to a hydrothermal time of 9 minutes, suggesting that both temperatures have attained an optimal value at this duration. Nevertheless, the yield decreased after the duration exceeded 9 minutes. These findings demonstrate that microwave-assisted pyrolysis requires precise durations and temperatures in order to attain an optimal yield of bio-oil. The bio-oil yield was maximum at 25.60 wt% when microwave-assisted pyrolysis was conducted at a temperature of 450°C, resulting in a reduced biochar yield.

Table 1: Product yield of palm kernel shells using a microwave-assisted hydrothermal liquefaction technique at different times and temperatures.

Temperature (°C)	Time (min)	Bio-oil (wt%)	Biochar (wt%)	Biogas (wt%)
350	6	10.70	49.35	39.95
	9	21.30	24.05	54.65
	12	16.30	29.55	54.15
450	6	23.81	27.62	48.57
	9	25.60	25.12	49.28
	12	17.65	33.45	48.90

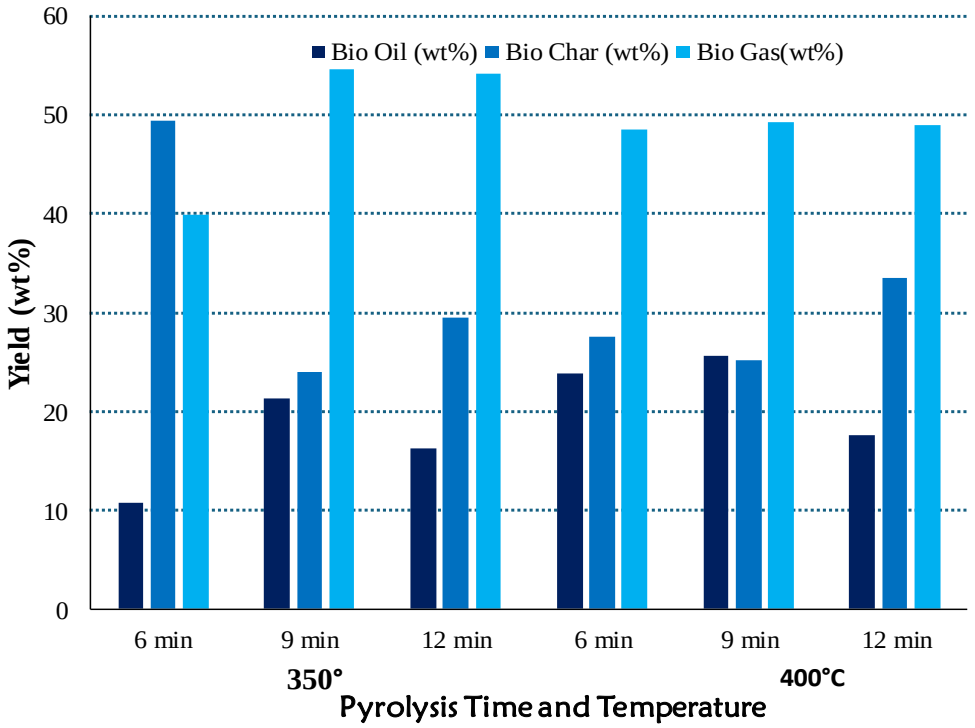

Figure 8. Yield of hydrothermal liquefaction aided by microwave.

Conversion under conventional heating

This experiment utilizes conventional heating pyrolysis, which is a modified type of conventional heating employed in research methodology to extract bio-oil from palm kernel shells. Subsequently, the quantities of bio-oil and associated by-products, such as charcoal and biogas, are computed. The yields obtained from palm kernel shells using traditional heating technique at pyrolysis temperatures of 350°C, 400°C, and 450°C are tabulated and reported in Table 2. Figure 9 illustrates the overall product yield achieved through traditional thermal pyrolysis. The bio-oil yields varied from 11.13 wt% to 36.07 wt%, as indicated in Table 2. The highest bio-oil yield was found at a temperature of 400°C, rather than 450°C, with a percentage drop ranging from 11.13 wt% to 13.03 wt%. A higher temperature might trigger a violent response during thermal decomposition, leading to secondary pyrolysis that produces a greater amount of biogas compared to bio-oil. The data presented in Figure 9 shows that the product yield for bio-oil increased as both the temperature and time increased. However, there was a slight decrease in the bio-oil yield when the pyrolysis temperature exceeded 450°C. On the other hand, the yield for biochar and biogas showed marginal increases with some inconsistencies. The maximum recorded weight percentage of biochar was 63.97 wt%, while the maximum recorded weight percentage of biogas was 50.45 wt%. According to the tabulated data and graph, it is evident that the palm kernel shell produces the largest amount of bio-oil at a pyrolysis temperature of 400°C and a pyrolysis period of 40 minutes, resulting in a bio-oil yield of 36.07 wt%. Furthermore, the pyrolysis temperature results in a poor yield of biochar, amounting to just 40.62 wt%, and a similarly low output of biogas, amounting to 23.31 wt%.

Table 2: Product yield of palm kernel shell utilizing conventional heating at different times and temperatures.

Temperature (°C)	Time (min)	Bio-oil (wt%)	Bio-char (wt%)	Bio-gas (wt%)
350	30	15.42	46.48	38.1
	40	22.47	63.97	13.56
	50	29.01	46.8	24.19
400	30	32.09	47.67	20.24
	40	36.07	40.62	23.31
	50	31.48	42.5	26.02
450	30	11.27	33.28	50.45
	40	13.03	55.83	31.14
	50	11.13	56.72	32.15

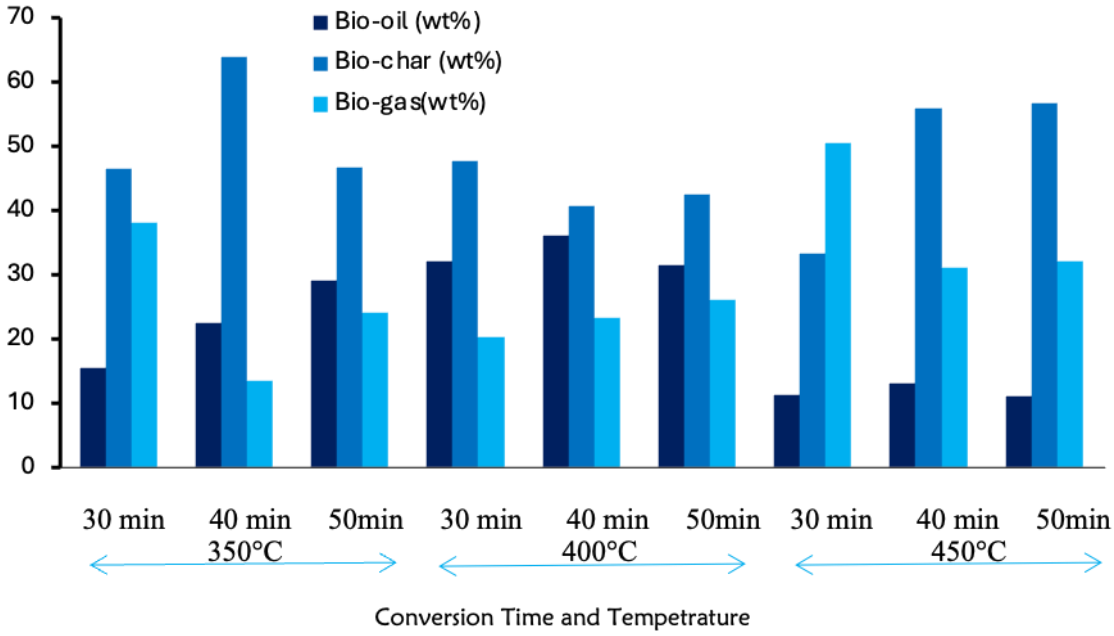

Figure 9. Product yields of conventional heating process.

CONCLUSION

The extraction of bio-oil from palm kernel shells is achieved by the utilization of both microwave-assisted and conventional heating methods. The results indicate that many parameters have an impact on both conversion processes used for bio-oil production. The highest amount of bio-oil obtained with the use of microwave-assisted conversion is 25.60 weight percent at a temperature of 450°C and a duration of 9 minutes. The highest amount of biochar achieved using these methods is 350°C and 6 minutes, resulting in a yield of 49.35 wt%. The highest value is recorded for biogas at a temperature of 350°C, with a duration of 9 minutes, and a yield of 54.65 weight percent. The maximum bio-oil output achieved with

traditional heating at a temperature of 400°C during a duration of 40 minutes was 36.07 wt%. The biggest amount of biochar obtained was at a temperature of 350 °C, with a duration of 30 minutes, resulting in a yield of 63.97 wt%. Additionally, the greatest yield of biogas was achieved at a temperature of 450°C, again within a 30-minute timeframe, with a yield of 50.45 wt%. These findings highlight the impact of the heating method and operating conditions on the yield and distribution of products, providing valuable insights for optimizing bio-oil production from palm kernel shells.

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