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Development of microwave-assisted pyrolysis apparatus and some experiments on agricultural biomass wastes: cashew shells, bagasse, and dried algae

Noor Fachrizal [1*], Hadi Surachman [1*], Titik Nurmawati [1], Ahsonul Anam [2], Sutopo [1], Yosephine Merry Devina [3], Enny Rosmawar Purba [1]

[1] *Research Centre for Energy Conversion and Conservation, National Research and Innovation Agency, South Tangerang, Indonesia*
[2] *Research Center for Process and Manufacturing Industry Technology, National Research and Innovation Agency, South Tangerang, Indonesia*
[3] *Department of Chemical Engineering, University of Indonesia, Depok, Indonesia*

ABSTRACT

Agricultural wastes have huge biomass potency which can convert energy in the form of liquid fuel, solid, and syngas in one process by using the pyrolysis technique. The process can be driven to fast and flash pyrolysis to obtain yield in liquid form by using microwave heating method. This research started by developing an experimental laboratory apparatus by modifying a commercial microwave oven. It worked safely, and some experiments had been carried out. The process could run only 10–20 mins, much faster than conventional heating, which needs more than 2 hrs for pre-heating. By using chopped cashew nuts as raw material, bio-oil results were 9.33% and 25.00%. Dried algae and bagasse needed biochar as the material absorber. The experiments showed, dried algae with 0% and 10% biochar gained bio-oil yields of 3.19% and 15.00%. Feeding bagasse as raw materials with 0%, 10%, and 20% biochar, resulted in bio-oil of 42.45%, 39.10%, and 42.75%. GC/MS analysis of bio-oil identified that it contained 42.24% of non-oxygenatic compounds and 57.76% of oxygenatic compounds. This non-oxygenatic compound contains a long and stable carbon chain. These results proved that the experimental apparatus can work properly and safely.

Keywords: Biomass, Fast pyrolysis, Microwave, Bio-oil.

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Corresponding Author:
Noor Fachrizal
noor004@brin.go.id
Hadi Surachman
hadi012@brin.go.id

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1. INTRODUCTION

Indonesia has a significant and abundant agricultural and plantation organic biomass, such as wood scraps, rice husks, sugarcane bagasse, palm empty fruit bunches, wood dust, coconut fiber, and coconut shells. The potential of biomass from agricultural and plantation residues is shown in Table 1.

Another potential biomass source is microscopic organisms living in water, namely microalgae. In fish and shrimp ponds, as shown in Fig. 1 the microalgae are considered nuisances and wastes as they reduce the penetration of sunlight. Generally, 1 kg of dry biomass is produced from 2.4–6.8 m^3 of microalgae in water (Martins et al., 2018). This represents a significant potential in large ponds if harnessed effectively.

Meanwhile, cashew plantations in Indonesia produced approximately 118,175 tons of cashew nuts in 2014, where the shells constitute 45%–50% (Saenab et al., 2016). This also presents a substantial potential for biomass energy. Additionally, organic waste from residential areas represents another significant and abundant potential source of biomass energy.

All of these biomass wastes will be an environmental burden if left unmanaged, or not getting proper treatment such as direct burning which will produce toxic gases (dioxin and carbon monoxide), or the occurrence of decaying due to piling up waste, which will

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Table 1. Agricultural biomass potential in Indonesia, 2018 (Hidayati and Ekayuliana, 2022)

Raw material	Biomass	Potential residue (tons/year)	GJ/year
Paddy	Rice husk	4,151,858	61,447,491
	Rice straw	12,455,573	176,869,129.5
Corn	Corn husk	5,268,750.71	77,134,510.42
	Corn cob	4,905,077.67	92,313,561.82
Palm oil	Shell	3,001,854	61,538,010.49
	Husk	5,574,872	110,939,953.4
	EFB	9,434,399	174,536,378.2
Coconut	Husk	1,356,170.67	26,309,711
	Shell	422,330.01	8,573,299.15
Rubber	Rubber tree	2,686,464	51,580,112.26
Sugar cane	Bagasse	651,284	12,048,761.4
	Bagasse pith	238,804	4,298,477.04

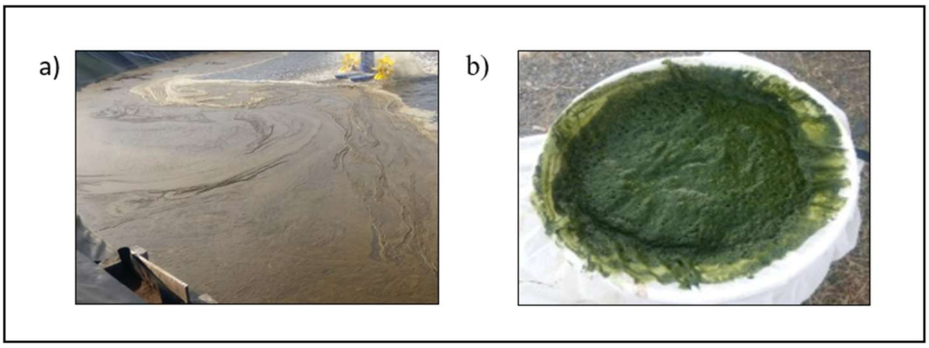


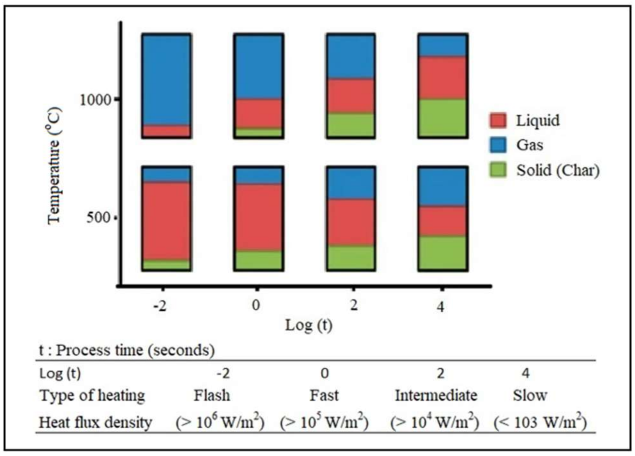
Fig. 1. Microalgae from sewerage and shrimp ponds, (a) Algae covered the surface of the shrimp pond, (b) Algae were removed from the pond

produce methane gas. Various efforts are made to utilize these residues and wastes to be more valuable (Mahmud and Anannya, 2021; Adekanbi and Olugasa, 2022; Leite et al., 2023; Stroescu et al., 2024), one of which is by converting them into energy with various methods.

Pyrolysis is one method of converting this organic material into energy. Organic matter is typically composed of three main components: cellulose, hemicellulose, and lignin, constituting approximately 20%–40%, 40%–60%, and 10%–25% by weight, respectively. Through the pyrolysis process, it decomposes into solid products (charcoal), liquid (bio-oil), and gas (syngas: CO, CO_2, H_2, H_2O, and CH_4) (Yang et al., 2007; Akhtar and Amin, 2012; Heyerdahl et al., 2012; Lin et al., 2015; Waters et al., 2017; Giorcelli et al., 2021). Biomass pyrolysis can be divided into four stages: water content evaporation, hemicellulose decomposition, cellulose decomposition, and lignin decomposition (Yang et al., 2007). Studies have shown that hemicellulose, cellulose, and lignin decompose at different temperature ranges. Generally, hemicellulose decomposes at a lower temperature range (220–315°C) compared to cellulose (300–400°C), while lignin decomposes over a wide temperature range (150–900°C) (Waters et al., 2017; Giorcelli et al., 2021).

Scientifically, pyrolysis is a thermal decomposition process of organic matter in the absence of oxygen at temperatures around 350–550°C. The composition of the products is influenced by the chemical composition of the biomass material and the operational conditions of the process. The faster and at a temperature around 500°C the process occurs, the more liquid is produced, as shown in Fig. 2 (Mohabeer et al., 2022). Tripathi et al. (2016) explained that the pyrolysis process begins with primary cracking, which produces volatile compounds and condensable vapors, and then it is continued with secondary cracking, which is the cracking of heavy compounds into charcoal and gases. Fast and flash pyrolysis with high rapid heating to moderate temperatures (400–600°C) and short residence times, prevents secondary cracking occurred, thereby maximizing the formation of bio-oil (Aboelela et al., 2023). The gas and vapor generated from the pyrolysis process are condensed to separate the gas and liquid materials at room temperature.

Fast pyrolysis is a relatively new technique for producing renewable energy. Compared to combustion, gasification, and carbonization, which are also pyrolysis processes (slower) widely practiced on a commercial scale, biomass pyrolysis, especially fast and flash pyrolysis, continues to evolve (Patel et al., 2020; Raza et al., 2021). Research and development of fast pyrolysis processes and systems for organic materials with conventional heat supply have been extensively conducted. Bridgwater et al. (1999) developed



t : Process time (seconds)

Log (t)	-2	0	2	4
Type of heating	Flash	Fast	Intermediate	Slow
Heat flux density	(> 10^6 W/m^2)	(> 10^5 W/m^2)	(> 10^4 W/m^2)	(< 103 W/m^2)

Fig. 2. Temperature and process time effects on pyrolysis products (Mohabeer et al., 2022)

fast pyrolysis using gas energy sources to drive the process, maintaining temperatures between 400–500°C, with bio-oil products exceeding 50% in weight. Meanwhile, Vamvuka et al. (2023) developed an apparatus that produced energy in solid, liquid, and gas forms with a composition of 28%–45% char, 40%–58% liquid, and 13%–25% gas**.** This investigation included experiments on biomass from forest residues (FOR), pine cones (PCO), and discarded derived fuel (RDF), which were processed in an electric furnace, at moderate temperatures of 350–550°C in atmospheric pressure. Yi et al. (2005) has developed several prototype units with a feed scale of 50–200 kg/hrs and conducted various research on optimizing the pyrolysis process, system configurations, and reactor process variations.

Materials can be classified into three categories based on their interaction with microwaves: conductors, insulators, and absorbers (Fernandez et al., 2011). Microwaves are reflected from the surface of metals and, therefore, do not heat metals, which generally have high conductivity and are classified as conductors. Materials (such as glass and ceramics) that are transparent to microwaves are classified as insulators. On the other hand, materials that can absorb microwaves are called absorbers or dielectrics, and the process of microwave heating is also referred to as dielectric heating (Fernandez et al., 2011; Huang et al., 2016; Diaz et al., 2018) The level of microwave heating of a material can be determined by a term called tangent loss, which is the ratio of dielectric loss factor to the dielectric constant of the material. In general, materials can be divided into three types according to their tangent loss: high (> 0.5), medium (0.1–0.5), and low (< 0.1) for materials in microwave absorbers. The tangent loss of carbon materials such as charcoal, black carbon, and activated carbon ranges from 0.1 to 0.8, comparable to or higher than the tangent loss of distilled water (0.118 at 2.45 GHz and 298K), making carbon materials excellent microwave absorbers, demonstrating high capacity to absorb and convert microwave energy into heat (Huang et al., 2016).

Microwaves cause the movement of molecules with ionic species migration or dipolar rotation, or both, resulting in heat generation due to molecular friction. Materials with high water content, such as food and other biomass, absorb microwave energy. When electromagnetic waves with a frequency of 2450 million cycles per second penetrate food or other organic materials, water molecules tend to align with the cycle's energy. Since microwaves change their polarity every half cycle, these water molecules also follow these alternations, resulting in the material's molecules changing direction about 4.9 billion times per second, causing intermolecular friction and generating heat energy (Metaxas, 1991). This principle is used to harness microwaves as a heating tool. Unlike conventional heating that heats from the surface, heating with a microwave oven is called volumetric heating because the heating is generated internally (Motasemi et al., 2015; Robinson et al., 2015; Ethaib et al., 2020).

The application of microwave heating technology as an energy source for biomass pyrolysis processes is a new approach. Due to the nature of intrinsic heating within the heated material, the response can be much faster and more uniform than conventional heating. It is hypothesized that this process can be driven towards fast and flash pyrolysis, leading to an increased production of liquid products (bio-oil), as well as energy products in the form of syngas and residual char (Zhang et al., 2017).

The development and implementation of microwave technology as a heating source for biomass pyrolysis processes, as obtained from articles found through web searches, were initially conducted by researchers from several foreign institutions, namely Prof. Heyerdahl from the Norwegian University and Dr. Roger Ruan from the University of Minnesota (Heyerdahl et al., 2012). Afterward, more researches had been carried out using this microwave method (Lin and Chen, 2015; Huang et al., 2016; Zhang et al., 2017; Giorcelli et al., 2021; Pratiwi et al., 2022). Several experiments yield on various raw materials with various pre-treatments and process conditions, were shown by Huang et al. (2016).

The use of microwaves carries radiation risks, which are higher compared to conventional heating systems. There are specific safety requirements that must be implemented for the security and safety of each microwave oven unit when utilizing this technology in biomass pyrolysis processes.

The threshold limits for radiation exposure according to the Title 21, CFR, Part 1030 from The Center for Devices and Radiological Health (CDRH) of the United States Food and Drug Administration (FDA) (Kawahara et al., 2013), the power density in the vicinity of the external oven surface shall not exceed 1 mW/cm^2 at any position 5 cm or more from the external surface of the oven as measured before purchased by the purchaser, and thereafter, 5 mW/cm^2 at any point.

Similar to all electromagnetic waves, the intensity of radiation decreases significantly as the distance from the microwave oven increases. For instance, if ones experience a radiation level of 5 mW at a distance of 2 inches, moving away to 20 inches would reduce the radiation to 1/100th, which is 0.05 mW. Therefore, considering a microwave heating power of 600–700 W, the exposure of 0.05 mW becomes negligible. The greater the distance, the lower the radiation exposure. At a distance of 1 meter, the radiation exposure would be extremely minimal.

This paper presents the development of this technology with some experimental results. These experiments were conducted on a laboratory scale (batch process) and aimed to prove whether the microwave pyrolysis process could be successfully performed more quickly compared to the conventional pyrolysis process. Further research will characterize various biomass raw materials with this equipment.

The research began with the construction of a simple laboratory experimental apparatus. The assembly of this experimental apparatus was done by utilizing a household microwave oven, modified for the purpose of laboratory experimentation on biomass pyrolysis processes. The modification considered aspects of microwave properties during the heating process, safety factors, and the anticipated heating effects on the oven. The design and initial experiments focused on proving the hypothesis that with microwave heating, the process could be achieved faster, and this household oven could be modified and used safely as a laboratory experimental tool. The experimental materials used were cashew shells, dried algae, and sugarcane bagasse, which are abundant biomass waste in Indonesia. It is expected that this laboratory device will be utilized as a facility to study the pyrolysis process in the laboratory, providing a foundation for developing continuous production system designs.

2. MATERIALS AND METHODS

2.1 Designing Experimental Apparatus Configuration

The criteria for selecting materials and assembling the experimental apparatus are as follows:

a. Selecting the microwave oven

The microwave oven to be used is a commercial oven, utilizing the commonly used operating frequency for household microwave ovens, which is 2.45 GHz. The selection of this oven should also consider the power required for the process, based on literature, it should have an output power of at least 600–900 (Heyerdahl et al., 2012; Huang et al., 2016). A microwave oven with manually adjustable power supply and time settings is chosen to provide flexibility in changing experimental parameters.

b. Selecting the reactor process material

Glass is a material that allows microwaves to pass through and is also insulating. Therefore, a 500 mL round-bottom flask or Erlenmeyer flask is selected (Huang et al., 2016; Diaz et al., 2018) equipped with a glass pipe connector that will link the reactor to other devices outside the oven.

c. Selecting the condensation device:

This device is used to condense the vapors produced in the pyrolysis process. It consists of a glass distillation column commonly used in chemical laboratories, which is arranged and modified according to the needs of this experiment. The distillation column is equipped with a water-cooling bath with circulated water, and it acts as a container for collecting the condensed pyrolysis-oil. At the outlet of the distillation device, a suction fan is installed to help remove the pyrolysis vapors to the condensation device while simultaneously extracting the syngas, which is also a product of the pyrolysis process

d. Modifying the oven

To connect the glass reactor vessel inside the oven to the condensation device, heat-resistant glass tubing connectors are used. This requires drilling holes in the oven walls. The drilling process must consider the properties of microwaves to prevent radiation leakage that could harm people outside the oven. The wavelength of microwaves is approximately 12 cm, so a hole with a diameter of 2 cm is safe to block radiation leakage.

e. Selecting additional insulation for the reactor

In household microwave ovens, the heating chamber and the electronic wave generator compartment are separated by only a thin metal wall. The pyrolysis process, which requires temperatures around 350–550°C, can potentially generate excess heat that may affect the electronic components. Although the glass reactor is already insulating, the heat radiation from the heated material can still impact the electronic devices. To address this, an insulating container is needed that does not impede microwave radiation but can block thermal radiation. For this experiment, a ceramic container is chosen (Huang et al., 2016; Diaz et al., 2018).

The configuration of the microwave-assisted biomass pyrolysis experimental apparatus is schematically presented in Fig. 3. Throughout the process, the experimental chamber was equipped with a microwave leakage detector and an emergency shutdown system. Temperature measurements during the process could only be carried out using an infrared thermometer immediately after the process is completed. The temperature of the glass reactor vessel was measured, and a thermocouple sensor was inserted into the glass tubing to measure the temperature of the outgoing pyrolysis product vapors.

2.2 Assembling and Experimental Scheme

The experimentation on the pyrolysis process using microwaves aimed to validate the hypothesis that microwave heating could drive the process towards fast and flash pyrolysis. It also aimed to demonstrate the functionality of the designed and assembled experimental setup to support this laboratory experiment effectively. Furthermore, it sought to confirm the safety of the modified oven for use. This assembling process took the important step to find out the optimum configuration to support the process driven by microwave method.

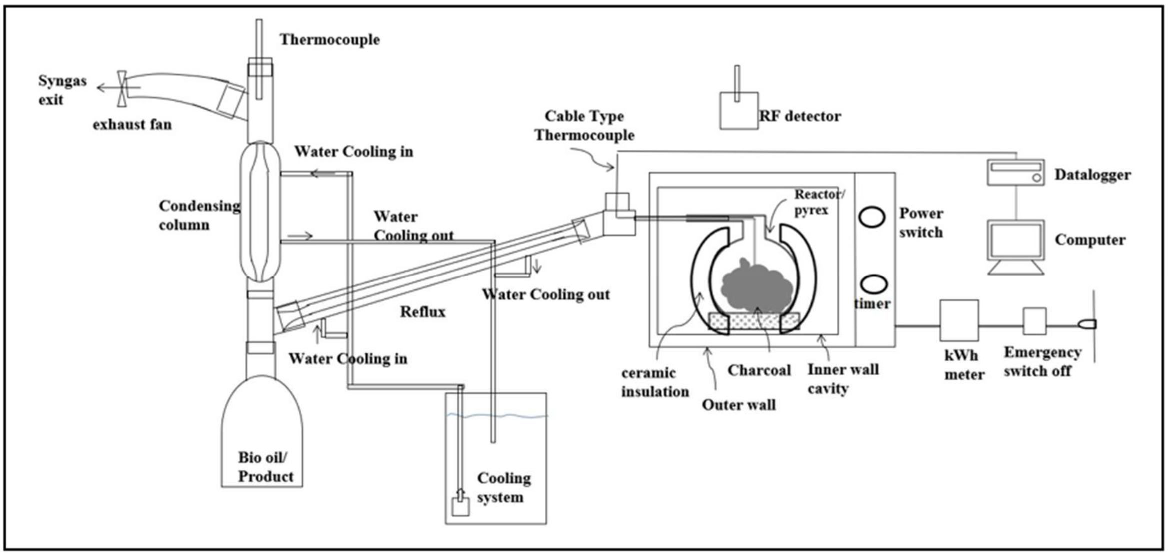


Fig. 3. Schematic configuration of the microwave pyrolysis experimental apparatus

The raw materials were weighed and placed into the glass reactor. Before that, the raw materials were slightly crushed to reduce air gaps in the glass reactor, ensuring that the oxygen-free process can be maximized. After placing the reactor inside the oven and connecting it to the condensation device through tubing, the suction fan was turned on for a while to reduce the presence of oxygen in the system, increasing the chances of a process with minimal oxygen presence. The suction fan and cooling device had to be turned on before the process began.

For conducting these experiments, biomass from dried cashew nut shells and microalgae was used, with weights ranging from 150 to 300 g, except for sugarcane bagasse, which weighs 40 g. The experiment aimed to achieve rapid process temperatures to produce pyrolysis oil and residual char, as well as syngas at the outlet of the suction fan. However, a flare gas test to prove the existence of syngas could not be performed due to the absence of a flare gas pipeline. Visual observation would be made on the liquid product and residual char. The experiment would be conducted using microwave output power ranging from 380 to 850 W, with a hypothesized processing time of 5 to 20 minutes for biomass weighing between 40 and 300 g.

2.3 Product Testing

Bio-oil products would be tested visually to see how the bio-oil products were responsive to direct burning. And other test was composition content of the bio-oil produced by the experiment, using GC/MS. This characterization was carried out using GC/MS-QP2010 Ultra Shimadzu. The sample was extracted using diethyl ether solvent and concentrated with nitrogen gas, then analyzed using the gas chromatography-mass spectrometry (GC/MS) method. The chromatogram obtained can be used to classify compounds based on functional groups, such as aromatic, aliphatic, phenolic, furan, acid, alcohol, aldehyde, ketone, sugar, and nitrogen complex compounds. Fractions of each functional the group was calculated from the percentage area of the functional group in the chromatogram. The chromatogram referred to the residence time database of various functional groups owned by the NIST (National Institute of Standards of Technology) Library. The functional groups contained in bio-oil were identified based on the % area value of the peak in the GC/MS chromatogram. The values listed were not specific amounts, but only general comparisons.

3. RESULTS AND DISCUSSION

3.1 Experiment Results

This research began by designing a laboratory-scale apparatus for microwave-based biomass pyrolysis, which would be used to conduct experiments. In these trials, various changes were made to the tubing and distillation column configuration to find the most effective setup. The objective was to ensure that the vapors produced during heating in the reactor could flow through the distillation column and be efficiently cooled, resulting the optimal production of bio-oil. Assembly and testing were carried out to obtain the best experimental configuration, that effectively pushed the vapor resulting from the process in the glass reactor in the oven, so that it could rise and flow into a series of cooling tubes so that the vapor resulting from the pyrolysis reactor could be condensed into a liquid bio-oil product. The location and dimension of the oven exit hole, the configuration of the condensation tubing, and the location of the bio-oil collection glass would determine the amount of collected bio-oil product. These configurations were tested by visually observing the process and flow of steam in the glass tubing, carried out without measurements. Several arrangements from configuration 1 to configuration 5, had been tested, are shown in Fig. 4, resulting the optimum configuration on Fig. 4 (e). This optimum configuration was used for all experiments.

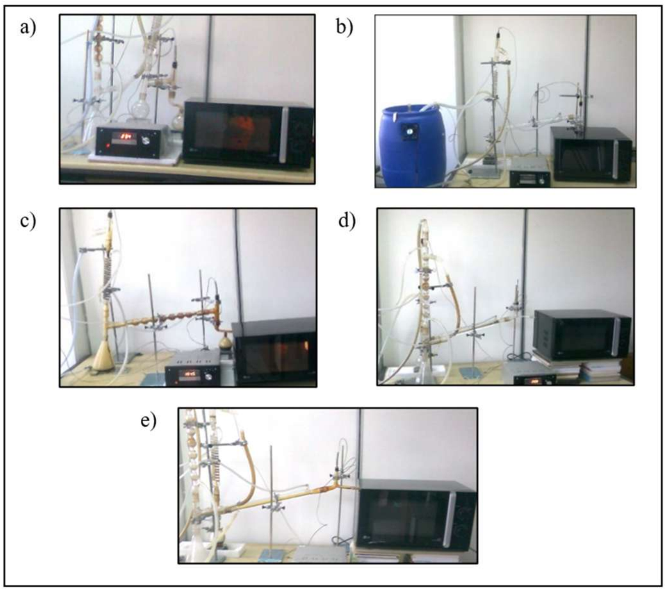


Fig. 4. Design and testing of experimental apparatus configurations: (a) Configuration 1, (b) Configuration 2, (c) Configuration 3, (d) Configuration 4, (e) Configuration 5, the optimal setup

Along with assembly and testing to obtain the best configuration of the experimental apparatus, a radiation leakage test had to be carried out to ensure that no microwaves could penetrate outside the oven, especially through the holes made for the glass reactor pipe to the condensation device. The measurement of microwave radiation leakage is shown in Table 2.

Table 2. Radiation leakage from the oven

Configuration	Radiation leakage at oven hole (mW/cm^2)
1	0.0001
2	0.0001
3	0.001
4	0.000
5	0.000

It can be observed that the radiation leakage was very small, far below the standard safety threshold, especially around the holes that were made. Thus, there was no radiation impact that exceeded the safety limit in the oven modifications that were carried out.

The experiments were conducted using chopped cashew nut shells, dried algae and sugarcane bagasse, each with the same as reactor volume of 500 mL, because of different sizes, there were different weights resulted. A total of 7 experiments were performed, and the results are presented in Table 3.

The first experiment was able to achieve a process temperature of 368°C, as measured shortly after the experiment concluded. The vapor temperature at the inlet of the condensation column reached over 150°C.

Tabel 3. Experimental results for different biomass

Experiment	Raw material	Power	Time	Process temperature	Yield	
		(Watt)	(min)	(ºC)	Bio-oil (g)	Charcoal (g)
1	Cashew nut shells chopped 300 g	620	10	368	28 (9.33%)	60 (20.00%)
2	Cashew nut shells chopped 240 g	850	11.5	443	60 (25.00%)	63.5 (26.45%)
3	Dried algae chopped 188 g	850	17.5	347	6 (3.19%)	135 (71.80%)
4	200 g dried algae with 20 g biochar	850	18	426	30 (15.00%)	130 (65.00%)
5	40 g bagasse with 20% biochar	380	10	515.9	17.1 (42.75%)	12.01 (30.03%)
6	40 g bagasse with 10% biochar	620	10	640.8	15.64 (39.10%)	10.38 (25.95%)
7	40 g bagasse, 0% biochar	850	10	398.52	16.98 (42.45%)	19.42 (48.55%)

The products obtained from this experiment were 28 g of bio-oil and 60 g of residual charcoal from 300 g of crushed cashew nut shells. The electric power consumption of the oven for this experiment was measured to be 0.256 kWh.

Because of the results were not entirely satisfactory, as a significant amount of liquid product was trapped in the distillation column, and the process temperature had not reached 400°C, it was decided to repeat the experiment by increasing the oven power and extending the process duration. In the second experiment, it was still not possible to install a thermocouple sensor at the reactor outlet outside the oven hole. Therefore, the process temperature was still measured shortly after the experiment was completed, and the reactor indicated a temperature of 443°C. Directly placing a stick-shaped metal temperature sensor inside the reactor was estimated to interfere with the microwave resonance in the oven, leading to the risk of radiation leakage. Due to this concern, the installation of a temperature sensor inside the Pyrex glass reactor has not been conducted up to this point. The second experiment was more effective, resulting in better yields compared to the previous trial. By applying 850 W for 240 g chopped cashew nut shells, in 11.5 minutes raw material in glass reactor reached 443ºC, resulted 60 g bio-oil dan 63.5 g charcoal. Improving the microwave power dan process time indicated the increase of process temperature and bio-oil yield.

These results were still far from the 45% bio-oil yield achieved from 150 g cashew nut shells, 20 mins and 400ºC process, power of 400 W, conducted by Amaliyah and Putra (2021), but these were an improvement over the 12% liquid product yields obtained from 800 g cashew nut shells, 400–600ºC process, 30 mins duration, from the research conducted by Ábrego et al. (2018).

Experiment 3, using dried microalgae as feedstock, showed unsatisfactory results, only 6 g bio-oil achieved from 188 g raw material, by applying 850 W of power in 17.5 mins process, reached 347ºC reaction temperature. It was possibly due to the limited responsiveness of dried microalgae to absorb microwave radiation, hence requiring an absorber. According to Salema and Ani (2010), not all organic materials can be pyrolyzed using microwave radiation due to differences in their absorption capacity towards radiation. Some materials, such as palm fibers and sugarcane bagasse, have low absorbance of microwave radiation. Mixing the feedstock with conventional biochar powder will increase the absorption of microwave radiation. Therefore, in Experiment 4, the dried microalgae was mixed with 10% wood biochar powder. The 10% amount was chosen arbitrarily as this experiment was in its early stage to demonstrate that the addition of conventional charcoal could enhance the process temperature and increase the yield of bio-oil.

Experiment 4 showed a significant increase in bio-oil yield to 15.00% by weight. However, this result is still far below the findings of Czarnocka (2015), who conducted various experiments using Chlorella sp. algae with microwave power ranging from 500–1260 W, correlating with process temperatures between 460–625°C, and achieved the best bio-oil yield of 28.5% by weight. In Czarnocka's study, the algae biomass was also mixed with biochar as a microwave absorber. A similar result was obtained by Du et al. (2011) with a bio-oil yield of 28.6% using a microwave power of 750 W.

These results indicate that the microwave method may not be universally applicable to all organic materials. However, when applied to complex municipal waste compositions, like the typical waste composition in Indonesia, which is often mixed, there is a high likelihood that some mixed materials will have good absorption capacity towards microwave radiation. Therefore, the limitations of the microwave method can be anticipated.

The subsequent experiments, namely Experiments 5 to 7 in Table 3, also used materials with low microwave absorption. Sugarcane bagasse, chopped and sieved to a size of 500µm, was prepared with a moisture content of 15%–20%. This sugarcane bagasse was mixed with biochar, varied at 0%, 10%, and 20% of the bagasse weight, with a bagasse weight of 40 g. The microwave power was varied at 380 W, 620 W, and 850 W, respectively. According to the experimental results of bagasse pyrolysis, a bar graph of the yields of experiments 5 to 7 is displayed in Fig. 5, where gas yields were calculated from the remaining percentage of charcoal and bio-oil yields.

Experiments 5–7 showed the bio-oil yields for each power level. The temperature of the vapor leaving towards condensation was measured by inserting a cable-type thermocouple sensor into the Pyrex reactor through the steam outlet tubing and recorded with a datalogger. Table 3 shows the highest bio-oil yields, namely 42.75% and 42.45%, obtained at sugarcane bagasse temperatures of 515.9°C and 398.52°C, respectively. These results were better than those obtained by Lin and Chen (2015), who achieved bio-oil yields between 14.4% and 22.0% by weight, using a controlled process at 550°C for 30 mins with a bio-char fraction between 0.1 and 0.3.

The results from the sugarcane bagasse experiments show that when the feedstock does not use biochar as a microwave absorber, the process temperature does not exceed 400°C. Bar graph on Fig. 5 shows that the majority of the products obtained in experiment 7 were charcoal (48.55%), with very little gas. Without the existence of biochar as absorber, although a 42.45% bio-oil yield was obtained, it needed microwave power of 850 Watts to proceeded temperature of 398.52ºC asymptotically. This indicates that a pyrolysis temperature lower than 400°C is not yet optimal for bio-oil production (Terry et al., 2021).

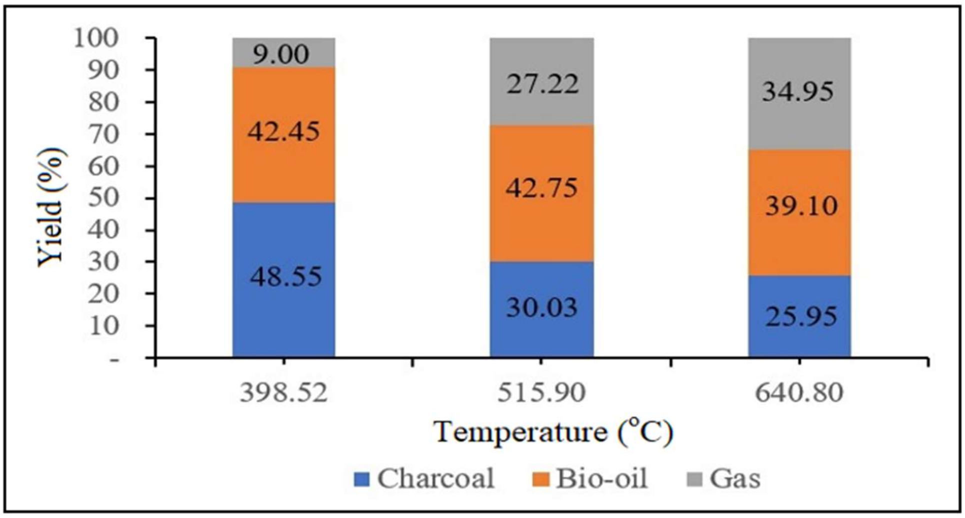


Fig. 5. The effect of temperature on the yield of the bagasse pyrolysis process in the experiment 5 (515.90°C), experiment 6 (640.80°C) and experiment 7 (398.52°C)

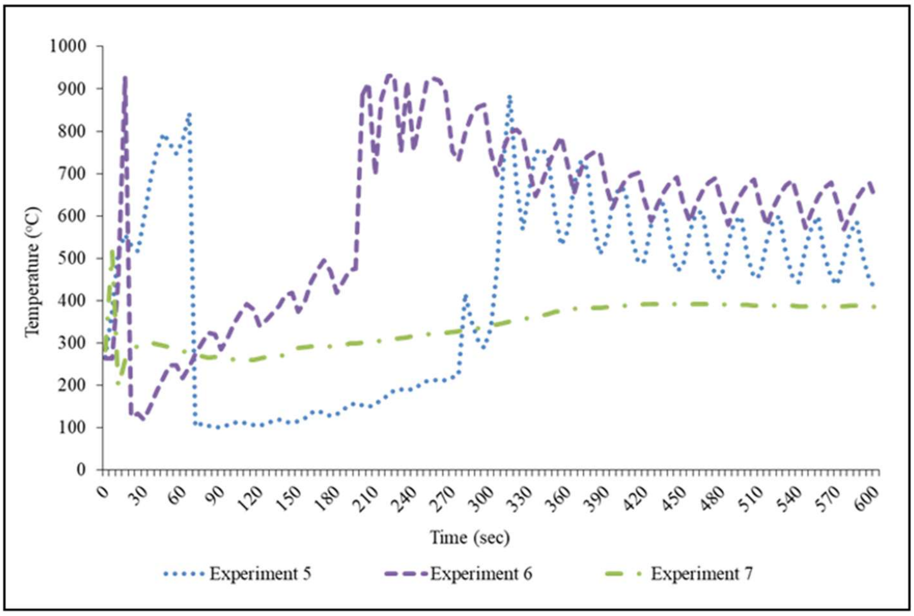


Fig. 6. The effect of bio-char content in sugarcane bagasse on the pyrolysis process temperature profile

The influence of adding biochar absorber on the pyrolysis process temperature of sugarcane bagasse is shown in the Fig. 6. The pyrolysis temperature of bagasse without microwave absorbers was lower than 400°C, most of the gas produced was still water vapor and a little volatile substance. Bagasse is a poor microwave absorber. The increase in temperature that occurred in the early stages was influenced by the water content in the bagasse (Salema and Ani, 2010). Water is a good microwave absorber, because of its nature as a polar compound and is easily subjected to dielectric heating when exposed to microwaves (Fernandez et al., 2011). When the water has evaporated, other biomass constituents were not ready to absorb microwaves (Salema and Ani, 2010), so the temperature of the bagasse were relatively constant

By adding 10% of biochar as absorber, with power of 620 W, process temperature of bagasse in the experiment 6, asymptotically reached 640.8°C. This indicated the biochar effect as microwave absorber is to increase temperature, but the bio-oil yield decreased to 38.99%. When the temperature of sugarcane bagasse is higher than 600°C, the amount of bio-oil and charcoal obtained decreased, indicating that more gas was produced during the decomposition. This shows that a pyrolysis temperature that is too high, i.e., above 600°C, is less optimal for bio-oil production (Terry et al., 2003; Huang et al., 2016).

In the experiment 5, the use biochar of 20%, with power of 380 W only, the temperature process had been driven to 515.9°C, which resulted the highest bio-oil yield of 42.75%. These results were correlated to the graph shown in a Fig. 2.

(Mohabeer et al., 2022). Therefore, maintaining the process temperature between 400–550°C can result in optimal liquid bio-oil production. Similar results were shown by Robinson et al. (2015), with bio-oil yields ranging from 50%–60% by controlling the process temperature at 500°C.

3.2 Bio-oil Product Testing

In addition to operational tests of the experimental setup, a flammability test was also conducted on the bio-oil product. The visualization of the tests on the oil product is shown in Fig. 7. According to Jatropha seeds extraction research (Kang et al., 2013), Jatropha oil could not be directly burned. However, the burning test of pyrolysis oil (bio-oil) showed that the oil was flammable enough, it could be easily burned directly by dipping a wooden stick into the oil and lighting it, or using a kerosene lamp with a wick, although the burning response was not as rapid as kerosene. It proved that

Fig. 7. Pyrolysis oil flamability test

Analysis of the content of compounds in bio-oil products from experiments was carried out by analyzing bio-oil sample with the GC/MS test. The sample tested were selected from the result of experiment 5 with a process temperature of 515.9ºC, which produced a yield of 42.75%. Experiment 5 was chosen because the process occurred at a temperature that produced optimal bio-oil yield, namely at a temperature of around 500ºC (Aboelela et al., 2023). The GC/MS results on bio-oil from experiment 5 are given in Fig. 8.

The X-axis represents the retention time, which indicates the time it takes for each component to pass through the GC column. The Y-axis represents intensity, which is related to the relative concentration of each component. Early Peaks (5.0–10.0 mins) depict volatile compounds with low boiling points. Examples include light hydrocarbon compounds, such as short-chain alkanes or alkenes. Middle Peaks (10.0–20.0 mins) depict compounds with moderate boiling points. Examples include organic compounds, such as aromatic compounds, phenols, or carboxylic acids. The peak points in this area indicate the complexity of the bio-oil composition. Late Peaks (20.0–27.5 mins) depict compounds with high boiling points (less volatile compounds), long-chain hydrocarbon compounds, polyaromatic compounds, or heavy oxygenated compounds. The highest peak indicates the presence of a compound with a significant concentration that has a long retention time. GC/MS analysis of the bio-oil content in experiment 5 bio-oil sample is shown in Table 4 and Table 5.

The GC/MS test results on the bio-oil samples from experiment 5 identified compounds that can be classified as non-oxygenate compounds because they do not contain oxygen, such as: aromatic and aliphatic, and oxygenate compounds because they contain oxygen, such as aliphatic hydroxyl and phenol, furan and its derivatives, aldehydes, ketones, acids, nitrogen complex compounds, and other components in small quantities. According to previous research, bio-oil is made up of an intricate blend of aromatic and oxygenated aliphatic compounds, which are divided into two primary fractions: the aqueous (acid phase) and the organic (oil phase). The aqueous fractions may be present.

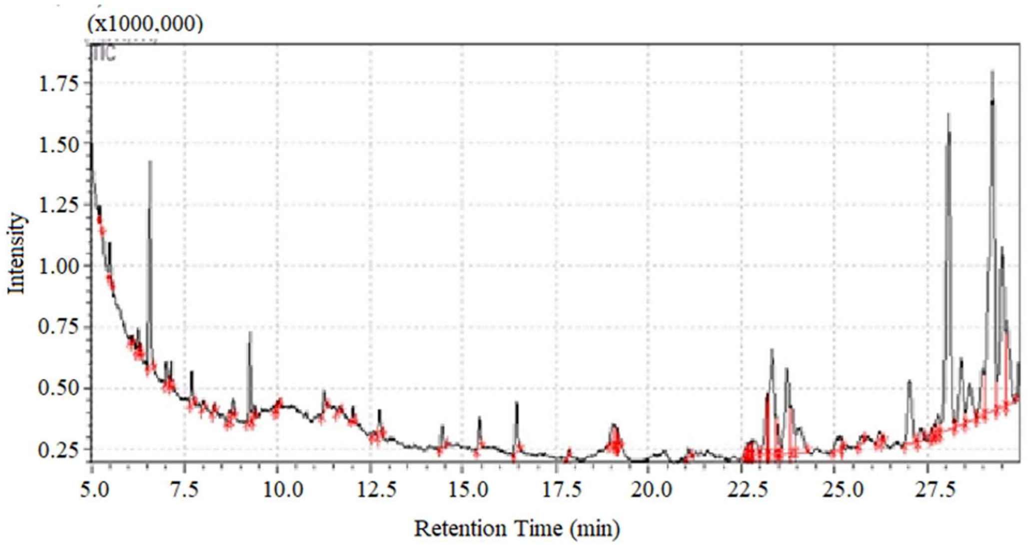


Fig. 8. Chromatogram result of bio-oil sample from experiment 5

Table 4. Non-oxygenatic compound content in bio-oil sample from experiment 5

Compounds	%
Non-oxygenatic compounds:	42.24
Aromatics	20.31
1,3-Dimethyl-(3,7 dimethyloctyl) cyclohexane	0.71
Cyclodocosane, ethyl-	15.09
Cyclohexane, 3-ethyl-5-methyl-1-propyl-	0.25
Cyclopentane, 1-pentyl-2-propyl-	3.74
Cyclotetradecane, 1,7,11-trimethyl-4-(1-methylethyl)-	0.52
Aliphatics	21.93
2-Pentene, 3-ethyl-4,4-dimethyl-	0.39
1-Nonadecene	5.37
erythro-7,8-Dichlorodisparlure	0.22
17-Pentatriacontene	0.66
Docosane	8.17
Heneicosane	6.55
1-Tricosene	0.35
1-Hexacosene	0.22

The oil phase consists of carbonyl and phenolic compounds, while bio-oil can contain more than 200 organic compounds. These include alcohols, aldehydes, ketones, organic acids, esters, phenols, alkenes, nitrogen compounds, sugars, furans, various oxygenates, and inorganic metals (Miranda et al., 2021). The bio-oil in the sample tested contained 42.24% non-oxygenate compounds, consisting of 20.31% aromatic components and 21.93% aliphatic components, and the remaining 57.76% oxygenate compounds. All aromatic components found in bio-oil are MAHs (mono-aromatic hydrocarbons) compounds, such as cyclopentane, cyclohexane, cyclotetradecane, and cyclodocosane. These aliphatic components are quite diverse, including saturated, unsaturated, single bond and double bond compounds, such as pentene, nonadecene, pentatriocontene, heneicosane, docosane, tricosane and hexacosane.

Bio-oil resulting from the pyrolysis process can be converted into bio fuel such as biodiesel, bio ethanol, or bio kerosene. The aim is to upgrade the properties of bio-oil so that it has a higher calorific value (energy) and better stability (Pawar and Salvi, 2020). Potential for upgrading to biodiesel, bio-oil has high organic acids, can be converted into esters (biodiesel) by adding alcohol and several catalysts such as zeolites and resins using the esterification or transesterification process (Panwar and Paul, 2021). Compounds that have the potential to be converted into biodiesel from the results of experiment 5 are fatty acid compounds (14-Pentadecenoic acid (1.97%), Chloroacetic acid, dodecyl ester (0.72%), Chrysanthemic acid (0.67%) and alcohol (6.6%). The total potential to become biodiesel is around 7.11% (alcohol and fatty acids) of bio-oil.

Even though the sample bio-oil contains non-oxygenate compounds and does not contain PAHs (poly-aromatic hydrocarbons), these non-oxygenate compounds are apparently composed of several long carbon chain compounds. The long carbon chains in bio-oil include cyclodocosane, docosane, tricosane and hexacosane, the percentage of which reaches 23.83%. Long carbon chains are hydrocarbon compounds that are stable and do not easily crack into shorter carbon chains. Thus, this compound is more difficult to break down into carbon chains that comply with fuel oil or diesel oil specifications.

Bio-oil can be converted into bio ethanol. Based on experimental results, compounds that have the potential to become bioethanol are alcohol (6.6%) and several oxygenatic compounds (Phenol, 2-methoxy-1.99%; 2-Furancarboxaldehyde, 5-methyl-0.44%; 1,2-Cyclopentanedione, 3-methyl-0.80%; Maltol: 0.11%) the potency is 3.34% of bio-oil. So, the total potential to become bio ethanol is around 9.94% of the total bio-oil. The alcohol in bio-oil directly contributes to bioethanol production through purification and conversion processes. Oxygenatic compounds become alcohol by hydrogenation reactions (Kong et al., 2018). Potential conversion to bio-kerosene, compounds that contribute to this include aromatic components (for example, Cyclodocosane, ethyl-, Cyclohexane, 3-ethyl-5-methyl-1-propyl-, and others). Total aromatic components: 20.31%, Aliphatics: Several aliphatic compounds that have larger chain lengths, such as Docosane (8.17%), Heneicosane (6.55%), and 1-Nonadecene (5.37%), also have the potential to be converted into bio-kerosene, because they have long chains. Total aliphatic components: 21.93%. So, the total potential is 42.24% of the total bio-oil. Fractional Distillation can also be used to produce bio kerosene (Castro et al., 2021). However, there are several challenges related to the conversion process into bio kerosene, requiring more sophisticated equipment and technology.

Table 5. Oxygenatic compound content in bio-oil sample from experiment 5

Compounds	%
Oxygenatic compounds:	57.76
Phenols	11.78
2-Methoxy-4-vinylphenol	0.79
Benzene, 1,2,3-trimethoxy-5-methyl-	0.28
(+)-4a,7,7a,8,9,10,11a-Octahydro-3,3,7,10,11-pentamethyl-6-oxo-1,2,4-trioxino[6,5-j]-2-benzopyran	0.31
Phenol	4.78
Phenol, 2-methyl-	0.17
Phenol, 2-methoxy-	1.99
Phenol, 4-ethyl-	0.54
Phenol, 4-ethyl-2-methoxy-	0.65
Phenol, 2,6-dimethoxy-	1.42
p-Cresol	0.56
Creosol	0.29
Furans	0.74
Benzofuran, 2,3-dihydro-	0.74
Alcohols	6.60
1-Decanol, 2-hexyl-	0.81
1,5-Naphthalenediol, decahydro-	0.30
1,22-Docosanediol	0.13
Tricyclo[4,2,1,0(3,7)] nonane-3,8-diol, (anti-8)-	2.07
2-Furanmethanol, tetrahydro-	0.48
11-Hexadecen-1-ol, acetate, (Z)-	2.31
cis-9,10-Epoxyoctadecan-1-ol	0.50
Aldehydes	0.90
2-Furancarboxaldehyde, 5-methyl-	0.44
3-Chloropropionic acid, tridecyl ester	0.14
7-Hexadecenal, (Z)-	0.16
Vanillin	0.16
Ketones	2.69
1,2-Cyclopentanedione, 3-methyl-	0.80
1,3-Cyclopentanedione	0.74
2-Cyclopenten-1-one, 3-methyl-	0.13
2-Cyclopenten-1-one, 2,3-dimethyl-	0.19
2-Cyclopenten-1-one, 3-ethyl-2-hydroxy-	0.23
Ethanone, 1-(2-furanyl)-	0,23
Pantolactone	0.26
Maltol	0.11
Acids	6.08
1-, beta, -d-Ribofuranosyl-1,2,4-triazole-3-carboxylic acid	0.11
1-Heneicosyl formate	2.03
11-Dodecen-1-ol difluoroacetate	0.14
11,13-Dimethyl-12-tetradecen-1-ol acetate	0.44
14-Pentadecenoic acid	1.97
Chloroacetic acid, dodecyl ester	0.72
Chrysanthemic acid	0.67
Nitrogen Complex Compounds	0.57
3-Hydroxymethyl-piperazine-2,5-dione	0.57
Others	28.4
1,3,4-Oxadiazole-2(3H)-thione, 3-(4-morpholylmethyl)-5-phenoxymethyl-	0.10
2-Piperidinone, N-[4-bromo-n-butyl]-	0.33
2,5-Dibora-1,4-dioxane, 2,3,5-triethyl-6-propyl-	3.08
Borane, 2,3-dimethyl-2-butyl- (dimer)	0.17
Trifluoroacetoxy hexadecane	20.87
4-Trifluoroacetoxyhexadecane	3.24
Triallylsilane	0.61

There are advantages and disadvantages of Bio-oil Components Based on GC-MS Analysis. The advantages of the existence of aromatic compounds (benzene and its derivatives, which are approximately 20.31%) are as follows. Aromatic compounds such as benzene and its derivatives have a high-octane number and a relatively high calorific value, thus enhancing engine performance and combustion efficiency. Aliphatic Hydrocarbons (such as docosane and heneicosane, long-chain aliphatics like 1-Nonadecene and 17-Pentatriacontene) approximately 21.93% have a relatively high calorific value. Ketones (1,2-Cyclopentanedione and 1,3-Cyclopentanedione) approximately 2.69% can increase the octane number of fuel. Additionally, some ketone compounds can act as fuel additives. Phenols and their derivatives approximately 11.78%, although they have some disadvantages, possess a reasonable calorific value (AL-Mashhadani et al., 2014; Xia et al., 2022). However, there are also disadvantages due to the presence of oxygenatic compounds (which amount of 57.76%) as follows. High oxygen content generally lowers the calorific value of bio-oil. Acids (Carboxylic acids such as 14-Pentadecenoic acid) around 6.08%: corrosive, increasing the instability of bio-oil (Wang et al., 2012). Aldehydes (0.90%) cause the formation of peroxides and are corrosive which can damage engines and fuels. Alcohols (6.60%), some of these compounds are corrosive and lower the calorific value of fuels. Furans (0.74%) have the effect of forming deposits and reducing the stability of bio-oil. Nitrogen Complex Compounds (0.57%) affect the formation of NOx in the combustion process (Jahirul et al., 2012; Pidtasang et al., 2013).

Fast pyrolysis has the advantage of producing more liquid. Fast pyrolysis produces liquid faster and in greater quantities. This occurs because the operating conditions are different compared to slow pyrolysis, especially the heating rate and vapor residence time. Fast pyrolysis uses a relatively high heating rate so that biomass can decompose quickly, and produce vapor with a high concentration. In addition, the residence time in the reactor is quite short, which can prevent vapor from decomposing into gas and char. Usually, the very small size of biomass particles increases the heat transfer rate, thereby accelerating the decomposition process and vapor production. Vapor cooling also occurs very quickly, which can minimize vapor decomposition and maximize condensation into liquid (Onay, 2007; Bridgwater, 2012; Solar et al., 2016).

The conducted experiments have shown a significant difference in response time between conventional surface heating and microwave volumetric heating. Experience in testing and operating biomass carbonization systems indicated that the carbonization process, which is also a slow pyrolysis process, took at least 45 mins to 3 hrs to produce mature charcoal (Fachrizal el al., 2008) The response time from room temperature to 300–400°C takes at least 30 mins for heating up (Sadaka et al., 2014). If the completion of the process was visually indicated by the maturity of charring, the experiment using microwave radiation showed a much faster response time, taking only about 10 mins, although the cooling column configuration was not yet effective.

The assembly of the equipment and this preliminary stage of the experiment were mainly focused on proving that a household microwave oven can be modified into a laboratory experimental facility for fast pyrolysis of organic materials, while considering the necessary aspects to achieve the experimental goals and ensure safety. Many process parameters still need to be studied, such as the ceramic insulation material which was still not effective in containing the heat, as the air surrounding the reactor inside the oven had a much lower temperature, resulting in significant heat loss from the reactor.

The main advantage of using microwaves is because of the heating is intrinsic (volumetric), heat is generated from within the material due to the dipole moment of the material molecules induced by microwaves (Motasemi et al., 2015), so that the pyrolysis process can be driven to fast pyrolysis. The use of conventional methods can produce fast pyrolysis, such as the system developed by Yi et al. (2005) and Bridgwater et al. (1999). However, to obtain fast pyrolysis with conventional energy, a large energy density is required (Patel et al., 2020; Raza et al., 2021; Mohabeer et al., 2022), because the heating comes from surface to insaide. The use of other method can also be done as reviewed by Novita et al. (2022), which utilizes concentrated solar power, but it requires high investment. The alternative use of microwave applications is quite rational, because the system can be made by local manufacturers, and microwave generator components are also available at domestic market. The use of microwaves does have risks, in this study due to modifying a commercial oven, especially making holes in the oven wall. However, a commercial oven with a working frequency of 2.45 GHz, a small hole in the wall (2 cm) will not pass radiation with a wavelength of 11–12 cm, this was proven in all experiments.

However, this experimental apparatus was already adequate as equipment for studying biomass pyrolysis processes in the laboratory and can be further improved in the future, such as by accomplishing with temperature control system. The results can be used to design a continuous model microwave pyrolysis system for bio-oil production. According to the nature of microwaves that reflected by metal walls (Fernandez et al., 2011), the mini continuous system model to be developed will be designed comprehensively by considering all aspects including safety, the reactor and microwave generator can be covered with metal plates so that they are safe from radiation leakage, and equipped with various electrical safeguards. Thus, the system can be applied safely to convert agricultural, plantation, and household wastes into alternative energy resources.

4. CONCLUSION

The experiment results showed that the modified commercial microwave oven functioned well and was safe to use. The configuration of the condensation system still needs to be optimized to achieve better results. This experimental setup can be utilized as a means to study the pyrolysis process and microwave physics in school or university laboratories. However, due to the risk of microwave radiation leakage, it is essential to have microwave detectors, metal plate insulation for radiation protection, and an emergency shutdown for the microwave oven during experiments. Additionally, the gas product discharge channels should be positioned outside the lab to ensure respiratory safety.

The results of this experiment were quite promising, as they advance the process towards fast and flash pyrolysis, potentially yielding a significant amount of liquid products. The pyrolysis oil produced was 60 g (25.00% by weight) from 240 g of cashew nut shell material without the need for an absorber since it was already responsive to microwave radiation. The other materials, such as dried microalgae and sugarcane bagasse, which were less responsive to radiation, were assisted by 10% and 20% biochar as absorber, resulting in bio-oil yields of 15.00% and 42.75% by weight, respectively. These results are hopeful, and visually, the flammability tests indicated that the bio-oil was readily burned directly, although not as reactive as kerosene. All experiments showed that, to obtain bio-oil higher, the process temperature should be reached over 400ºC, but not exceeded 600ºC. Applying higher power, or by adding biochar, will be able to increase temperature higher, but can even exceed 600ºC. It needs more investigation regarding absorber material addition, and control system to maintain process temperature within the range of 400–600°C is required.

The content of compounds in bio-oil from the results of GS/MS analysis includes aromatics and aliphatics that do not contain oxygen, aliphatic hydroxyls and phenols, furans and their derivatives, aldehydes, ketones, acids, nitrogen complex compounds, and other components in small quantities. This composition contains 42.24% non-oxygenate compounds and 57.76% oxygenate compounds, these non- oxygenate compounds contain stable long carbon chains. It is necessary to study further the bio-oil content in processes with controlled temperatures, the influence of raw materials and absorbers.

This experiment, and further experiments to be conducted, can serve as a basis for designing and building a continuous system model for bio-oil production, in order to overcome waste problem to be energy production.

DECLARATION OF COMPETING INTEREST

We wish to confirm that we have no known competing financial interests or personal relationships that could have appeared to influence outcome of the work reported in this paper.

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