

Microwave-assisted pyrolysis and distillation of cooking oils for liquid bio-fuel production

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ABSTRACT

The goals of this study are to produce and to characterize bio-oil from pyrolysis and distillation of liquid samples in a microwave reactor. Waste cooking oil (WCO) and fresh cooking oil (FCO) were employed as the feedstocks. A 900 W microwave reactor was modified and filled with 300 g of charcoal. It was used for both pyrolysis and distillation experiments. The pyrolysis experiments were performed at various feed flow rates and temperatures ranging from 0.051 kg/h to 0.306 kg/h and 400 °C–550 °C, respectively. The sample was introduced continuously into the microwave reactor for 60 min. Nitrogen gas was also supplied to ensure pyrolysis condition. The bio-oil product was then distilled to obtain fuel fractions. The results showed that the products yield and bio-oils composition were strongly influenced by pyrolysis temperature and type of cooking oils, while there was almost no influence of feed flow rate under the investigated condition. The bio-oil contained 90–100 wt.% aliphatic hydrocarbons and the rest were aromatic hydrocarbons. The bio-oil produced from WCO contained more than 50 wt.% green-diesel (C₁₀–C₁₅) which was comparable to that from FCO. The green-diesel fraction showed good fuel properties agreement to that of fossil diesels. Overall, microwave-assisted pyrolysis and distillation processes are potential ways to convert problematic WCO into useful liquid bio-fuel and green-diesel fuel.

1. Introduction

Until now, the dependency on energy supplies from fossil fuels is still very high. This raises a variety of crucial issues including economic, security of energy supply, and as well as environmental problems [1]. As such, concerns about the high demand for fossil fuels and global climate change have attracted increasing interest in the development of renewable energy [2,3]. Sustaining bioenergy production from natural resources such as palm oil, fat, and other vegetable oils is considered as the main alternative energy sources for bio-fuel production [4,5]. In addition, waste cooking oil (WCO) can also be a potential source for bio-fuel production [6]. This is due to the availability of easy-to-obtain materials with low price. For instance, the technical potential of WCO production from restaurants, hotels, schools, hospitals, rural and urban households in Indonesia can reach more than 3 million tons/year [7].

The pyrolysis process is believed as one of the best ways to convert WCO into alternative fuels. This process involves the breakdown of more complex chemical compound to be simpler one at medium to high temperatures with little or without oxygen. The product of this process can be in the form of gas, solid (char), and liquid (bio-oil) [8]. The

pyrolysis oil and gas products can be utilized as fuel or chemical feed-stock, while the char product can be utilized as a substitute for activated carbon although this is not yet so popular. Pyrolysis process can be carried out by applying conventional heating or microwave heating. The literature showed that bio-oil production process with conventional heating methods requires a relatively longer time due to transferring heat energy to raw materials from the reactor wall to the surface of the material to be processed [9]. Therefore, conventional heating consumes more energy as it suffers from heat losses to the environment [10]. Meanwhile, the heating process by using microwave irradiation is faster than conventional heating. There are several advantages in microwave heating including fast and environmentally friendly processes, more product yields, uniform heating, direct use of large-sized raw materials, and can be used to treat various types of waste [11–13].

It was revealed that microwave heating has been successfully applied for processing of various wastes including liquid waste of WCO [14–16]. In general, the use of microwaves is widely applied to the transesterification process for bio-oil production from liquid waste. Instead of transesterification, the use of microwaves for pyrolysis of liquid waste has also been gained attention in recent years. This is because pyrolysis

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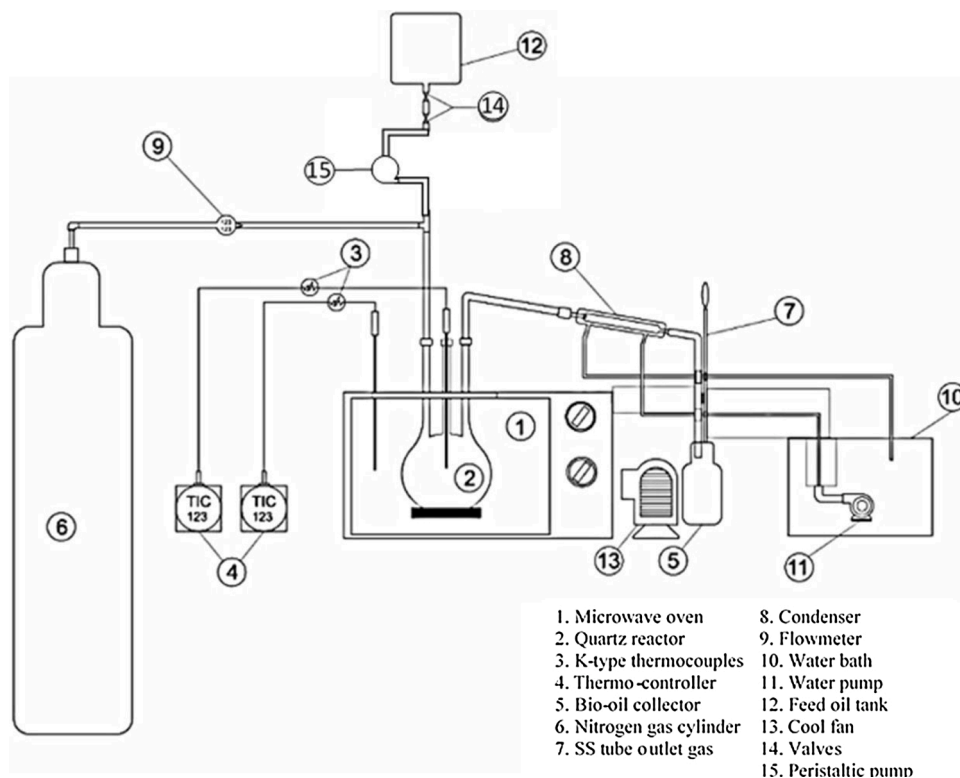


Fig. 1. Schematic diagram of the experimental equipment for pyrolysis process.

has several advantages including the flexibility to use various types and compositions of raw materials, and several valuable fuels and chemicals can be produced. The literature showed that waste oils derived from engine oil have been successfully converted into useful products under microwave pyrolysis [15,17,18]. However, information regarding WCO processing through microwave-assisted pyrolysis process is rarely available in the published literature. Diesel-like fuel recovery from microwave pyrolysis of waste palm cooking oil has been done [19]. In the study, waste palm cooking oil collected from a fried chicken restaurant

was processed under batch microwave pyrolysis for diesel-like fuel recovery at some process parameters such as temperature and type of absorber. A recent study also reported about bio-oil production from WCO at a low temperature of 400 °C under continuous microwave pyrolysis [20].

Considering the preceding description, instead of batch process, research on continuous pyrolysis process of WCO in a wide range feed flow rate and pyrolysis temperature under microwave irradiation is important to be performed. Therefore, this work is conducted to

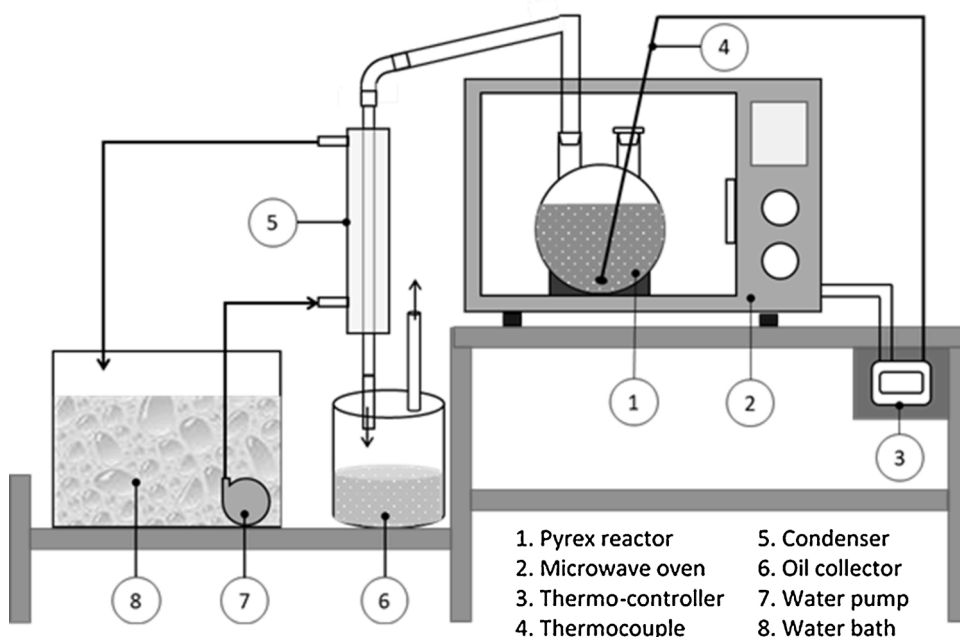


Fig. 2. Schematic diagram of the experimental equipment for distillation process.

investigate the effect of three main parameters including feed flow rate, pyrolysis temperature and type of cooking oils (waste and fresh cooking oils) on the yield of bio-oil and its composition produced from continuous microwave-assisted pyrolysis. The bio-oil was then distilled in a microwave reactor to obtain fuel fractions. Special attention was also performed to get fuel properties of bio-diesel fraction and to estimate the energy consumption and recovery. The waste cooking oil employed in this study was collected from different sources, resulted in having relatively complex compounds. This is important because in fact, the abundant availability of waste cooking oil comes from various sources such as restaurants, traditional food stalls, and households.

2. Materials and methods

2.1. Materials

Waste cooking oil (WCO) collected from some local restaurants and traditional food stalls was used for bio-oil production. Commercial fresh cooking oil (FCO) obtained from local market in Semarang, Indonesia was also used for comparison purposes. Prior to use, the water content in WCO and FCO were removed by heating them at ± 103 °C for 30 min, as given in a previous study [12]. After cooling, filter papers were employed to separate solid particles within the samples. For absorbing and converting microwave energy into heat, commercial particulate charcoal was employed. The charcoal was used in this work as it available commercially and generally has good microwave absorption ability compared to others absorber material. High purity nitrogen of 99.9 % was also used to remove oxygen within reactor and piping systems and to push pyrolysis products towards the condensation system.

2.2. Experimental setup

Fig. 1 presents a schematic diagram of the research equipment for pyrolysis process. In this study, continuous system of microwave-assisted pyrolysis of waste and fresh cooking oils was performed. To allow continuous process of the sample, a 2.5 L feed oil tank equipped with a peristaltic pump and valves as flowrate regulator was applied and settled over the microwave oven. A 2450 MHz microwave oven with 12.23 cm wavelength and 900 W maximum output power was used as the heating source. The microwave oven was modified to allow each pyrolysis experiments to be carried out properly. A reactor made of quartz glass with 1000 mL capacity was used and vertically installed inside microwave cavity. To measure the temperatures inside reactor and microwave cavity, K-type thermocouples were used. The pyrolysis reaction temperature was controlled by using a temperature controller. To prevent damage to the microwave oven, the temperature of the microwave cavity must be maintained below 200 °C. To condense the pyrolysis vapor product, a double glass-tube condenser was used. It was supported by a pump to circulate cooling fluid from a water bath containing a mixture of water and ice. Glass bottles with a capacity of 500 mL were used as containers to collect bio-oil products.

Fig. 2 presents a schematic diagram of the research equipment for distillation process. In this study, microwave-assisted distillation of bio-oil was performed. Similar to pyrolysis process, a 2450 MHz microwave oven with 900 W maximum output power was used as the heating source for distillation experiments. A Pyrex reactor of 1000 mL was vertically positioned inside the microwave cavity, and filled with charcoal as microwave absorber. The temperature inside the reactor was measured by using a K-type thermocouple. The distillation reaction temperatures were adjusted with the aid of a temperature controller. A double glass-tube condenser equipped with a pump for cool water circulation was employed to condense fuel fraction products. The fuel fraction products were then collected using 500 mL glass bottles.

Table 1
Experimental pyrolysis condition.

Manipulative variable	Operating condition	Constant variable	Respond variable
Feed flow rate	0.051–0.306 kg/h	Temperature: 450 °C	Temperature profile
		Feedstock: WCO Amount of charcoal: 300 g N ₂ flow rate: 100 mL/min Process duration: 60 min Microwave power: 900 W	Mass yield of products (bio-oil, gas, solid)
Temperature	400–550 °C	Feed flow rate: 0.102 kg/h	Mass yield of products (bio-oil, gas, solid)
		Feedstock: WCO, FCO Amount of charcoal: 300 g N ₂ flow rate: 100 mL/min Process duration: 60 min Microwave power: 900 W	Chemical composition of WCO, FCO, bio-oil

2.3. Pyrolysis and distillation experiment

This study relates to the use of microwave reactors for waste cooking oil pyrolysis process and for pyrolytic-bio oil distillation process. In the pyrolysis process, the feed was supplied continuously. This research was carried out sequentially to get a fundamental point of view of the continuous processing of waste cooking oil using a microwave reactor. **First**, research was begun by examining the thermal capability of the reactor at various feed flow rates. This information is important to assess the ability of microwave system to generate sufficient heat for maintaining the targeted reaction temperature. The reaction temperature profile was observed at feed flow rates ranging from 0.051 kg/h to 0.306 kg/h. The feed of waste cooking oil was injected after the targeted reaction temperature of 450 °C was achieved. This temperature was chosen because it is widely proven to be a good condition for materials decomposition through pyrolysis process. The reaction temperatures were then recorded using a camera during 60 min of irradiation. At the same time, pyrolysis process of waste cooking oil as the effects of feed flow rate as well as weight hourly space velocity (WHSV) and power load (WHSV/microwave power, MP) were also examined and evaluated to get the best conditions of the applied system. **Secondly**, pyrolysis process was then carried out to investigate the effect of reaction temperatures at a fixed feed flow rate of 0.102 kg/h using WCO and FCO. The reaction temperatures were varied from 400 °C to 550 °C. Table 1 shows the experimental condition of pyrolysis studies.

Prior to conducting the thermal and pyrolysis experiments, 300 g of charcoal was poured into the quartz glass reactor aimed at absorbing microwave energy. Addition of microwave absorber is needed as WCO and FCO samples are non-polar substances or insulator materials that difficult to be heated under microwave irradiation [12]. After that, the microwave reactor was then turned on until the specified pyrolysis temperature was reached. This condition was maintained for a while, then WCO or FCO sample was introduced continuously into the reactor at a predetermined flow rate for 60 min. The product of pyrolysis volatiles in the reactor was led into a condensation system to condense the bio-oil. Before and during the process, nitrogen gas was injected at a flow rate of 100 mL/min to ensure the pyrolysis process takes place without the presence of oxygen. Once the reaction time was reached, the microwave reactor was switched off and allowed to cool to ambient temperature by the aid of a fan.

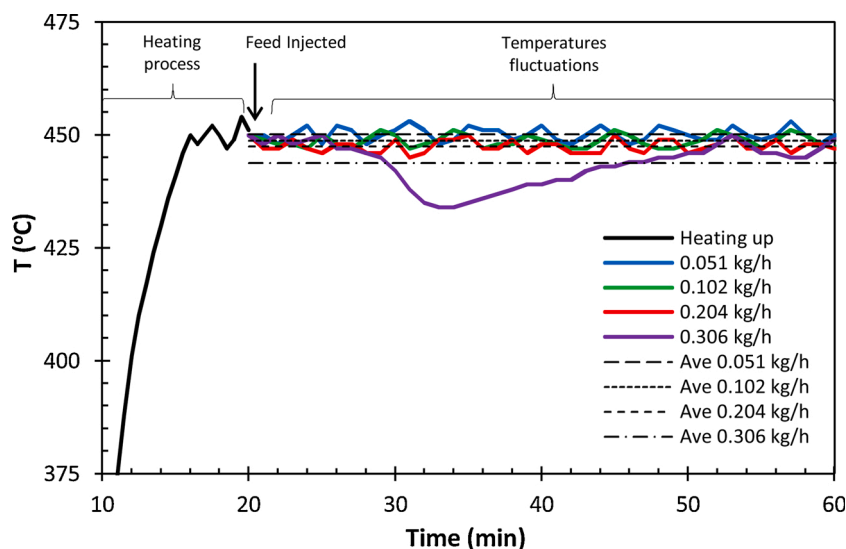


Fig. 3. Pyrolysis temperature profile at 450 °C under various feed flow rates.

The amount of bio-oil product was determined via weight difference of the oil collector and condenser before and after the experiments. The weight change in the charcoal-contained reactor was also measured to obtain the yield of solid product. The equipment could be easily dismantled for weighing purposes and reconnected back for further experiments. The yield of gas product was determined by mass balance. Evaluation of the yield of products is given in the following equations:

$$Y_{BO} = \frac{m_{fB} - m_{iB}}{m_O} \times 100\% \quad (1)$$

$$Y_S = \frac{m_{fS} - m_{iS}}{m_O} \times 100\% \quad (2)$$

$$Y_G = 100\% - (Y_{BO} + Y_S) \quad (3)$$

where Y_{BO} , Y_S , and Y_G are the yields of bio-oil, solid, and gas products, respectively in wt.%; m_{fB} and m_{iB} are the final and initial weight of the oil collector and condenser, respectively; m_{fS} and m_{iS} are the final and initial weight of the reactor contained charcoal, respectively; while m_O is the total weight of WCO or FCO during the pyrolysis process.

The last stage was distillation of bio-oil. In this work, the obtained bio-oil from continuous microwave-assisted pyrolysis of WCO at 450 °C was then processed under single stage distillation in a microwave reactor to obtain liquid bio-fuel fractions. The hydrocarbon components of bio-oil are separated according to their boiling temperature range to obtain fuel fraction which are 230 °C–340 °C for diesel fraction, 180 °C–230 °C for kerosene fraction, 30 °C–180 °C for gasoline fraction, and the rest are grouped as heavy fuels fraction. Three samples of each fraction were taken for obtaining the average.

2.4. Analytical method

A Perkin Elmer GC Clarus 680 MS Clarus SQ 8 T was applied for chemical composition analyses of the feedstocks and bio-oil products. The GC–MS has 30 m length of column with 250 μ m of diameter. The maximum oven temperature was conditioned at around 300 °C, while the injector split ratio was prepared at 42.2:1. To avoid sample oxidation, helium gas was used to purge the sample along the column at 0.8 mL/min. Moreover, oven temperature was set at 60 °C for 5 min, ramp at 5 °C/min to 280 °C and then hold at 280 °C for 5 min. Sample analysis process was run for 54 min. The sample was filtered by using a filter paper before injected into the GC at one μ l. All samples were treated and analyzed at similar program to obtain good result with accuracy of more than 98 % (or less than 2 % error) as recommended. NIST

MS 2.0 software was employed for identification of the characteristic peaks to compounds spectrum and quantitative analysis was carried out based on the peak area integral of each identified compounds. Three oil samples were taken for obtaining the average.

The obtained bio-diesel fraction from microwave-assisted distillation was also analyzed to get its fuel properties including density (ASTM D4052-18), kinematic viscosity (ASTM D445-18), total acid number (ASTM D664), flash point (ASTM D93-18), pour point (ASTM D97-17b), calorific value (ASTM D240-17), and cetane index (ASTM D4737-10, 2018). The fuel properties of bio-diesel were then compared with commercial fossil diesel to obtain better point of view regarding the quality of the produced bio-diesel.

3. Results and discussion

3.1. Temperature profile

Fig. 3 shows the temperature profile at the targeted reaction temperature of 450 °C at various feed flow rates. The figure shows that addition of feed flow rate affects reaction temperature. In general, the microwave system was able to maintain the reaction temperature of around 450 °C from the beginning to the end of the feed injection process into the reactor for feed flow rate of 0.051 kg/h, 0.102 kg/h, and 0.204 kg/h. It was found that the average reaction temperatures were 450.1 °C, 448.7 °C, and 447.5 °C in the same order of feed flow rates, respectively. The maximum temperatures at the condition were found to be 453 °C, 451 °C, and 450 °C, whereas the minimum temperatures were 448 °C, 447 °C, and 445 °C, respectively. This indicates that under these feed flow rate conditions, the interaction of microwave with absorber is capable to convert microwave energy into heat to supply and maintain the specified reaction temperature.

In the meantime, for feed flow rate of 0.306 kg/h, the reaction temperature initially dropped from about 450 °C to 434 °C after eight minutes of injection of the feed into the reactor, and then gradually increased until the targeted reaction temperature can be maintained. The result indicated that the average reaction temperature was 443.8 °C under this feed flow rate condition. The high feed flow rate with lower temperature could cause convective cooling effect at the absorber surface that consequently requires more energy to be heated. This illustrates the maximum thermal capability condition of the experimental equipment used in this work in which the microwave oven takes longer to produce enough heat to maintain the desired reaction temperature from the beginning to the end of the feed injection process.

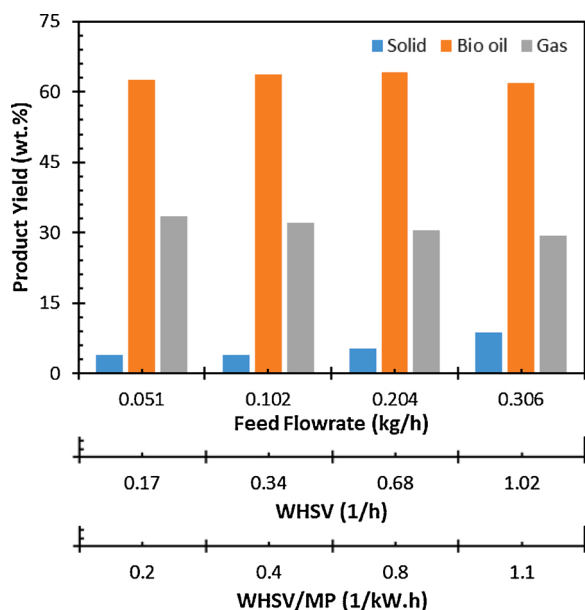


Fig. 4. Yields of products from pyrolysis of WCO as the effect of feed flow rate and its relationship with WHSV and WHSV/MP.

Based on the above results, it is expected that for higher process capacities, more amount of absorber material in the reactor and higher output microwave power are required to get better thermal stability. This phenomenon is also observed in the previous studies regarding the potential of microwave heating to handle high feed rates at a fix microwave power by regulating the amount and type of absorber in increasing the reaction temperature [10,12,18].

3.2. Yield of product

3.2.1. Effect of feed flow rate

Fig. 4 depicts the yield of products generated from pyrolysis of waste cooking oil in a microwave reactor as the effect of feed flow rate at a fix reaction temperature of 450 °C. It could be observed that almost no influence of WCO feed flow rate on the yield of bio-oil. This is indicated by only about 3.4 % maximum difference in the yield of bio-oil from WCO pyrolysis at various feed flow rates ranging from 0.051 kg/h to 0.306 kg/h. The slightly decrease of bio-oil yield at the highest feed flow rate applied in this study might be occurred due to significant temperature fluctuations that tended to produce a lower average pyrolysis temperature than others as shown previously in Fig. 3. This elucidates that there is a high relationship between the yield of products with temperature stability during pyrolysis process. Another reason is the increase of pressure inside the reactor that causes the pyrolysis volatiles product being forced out of the reactor toward condensation and product collection systems, as was also observed in other cases in a previous study [18]. The increase in pressure is due to an increase in volatile volume from the process of phase change of liquid into vapor resulting from the cracking of WCO into shorter molecules.

Fig. 4 also shows that the increase of feed flow rate not only tended to produce more amounts of bio-oil but also slightly higher number of solid products. This might be related to an increase in the volatile volume and pressure in the reactor as described earlier and also associated to a decrease in cracking reaction intensity of WCO that flows at a high quantity into the reactor especially for feed flow rates over 0.204 kg/h. This condition provides an opportunity for the formation of solid products both in the form of char and un-pyrolyzed WCO.

The relationship between yield of bio-oil with WHSV and WHSV/MP is also provided in Fig. 4. The WHSV is calculated as the feed flow rate of WCO divided by the mass of absorber material. This is one of the

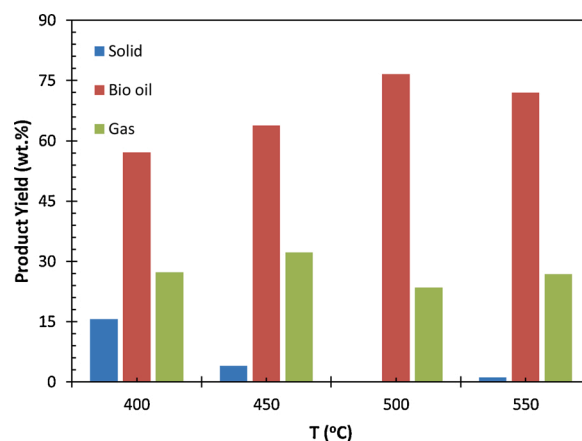


Fig. 5. Yields of products from pyrolysis of WCO at different temperatures.

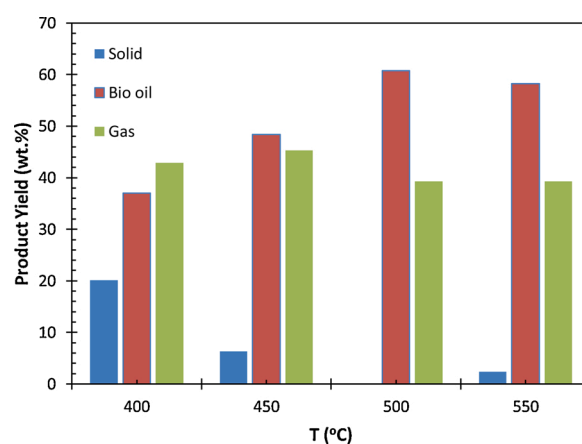


Fig. 6. Yields of products from pyrolysis of FCO at different temperatures.

important parameters commonly used in the chemical reactor design process. As shown in the figure, under the investigated condition, the used microwave-assisted pyrolysis system is capable to process each hour feed of WCO up to about one time the mass of absorber material with a relatively high yield of bio-oil product. In the meantime, the WHSV/MP represents the power load of the system that is calculated as the WHSV divided by the maximum output microwave power. This provides more useful information about the highest possible load capacity of the microwave reactor by considering the available output microwave power. As can be observed in Fig. 4, the microwave reactor had a maximum power load of around one per hour per kilowatt which illustrates the magnitude of WHSV is approximately equivalent to the magnitude of the maximum output microwave power. This means that when the microwave reactor has an output power of 3 kW, the maximum WHSV is also around 3 per hour for obtaining the power load of one per hour per kilowatt. Furthermore, when the amount of absorber that can be accommodated in the reactor is about 0.9 kg, the feed flow rate that can be processed is 2.7 kg/h. It should be noted that this is a rough estimate and may only apply for waste oil processing in microwave reactor with charcoal as absorber material under the investigated condition. Nevertheless, this approach seems to be applicable for evaluating the results of a previous study [18] in which the optimum pyrolysis process is obtained at a WHSV of 5 per hour with an output microwave power of 5 kW, showing the system has also a power load of one per hour per kilowatt.

3.2.2. Effect of temperature

Figs. 5 and 6 show the yield of products generated from pyrolysis of

Table 2
Comparison of yield of bio-oil from pyrolysis of WCO.

Heating method	Pyrolysis process	Max. carbon chain of feedstock	Temperature (°C)	Bio-oil yield (wt.%)	Reference
Microwave	Continuous	C ₃₀	450	64	Present study
			500	77	
			550	72	
Microwave	Batch	C ₁₈	450	70	[19]
			500	69	
			550	66	
Electric resistance	Batch	C ₂₂	550	8	[25]
			800	80	
Electric resistance	Batch	C ₁₈	500	56	[26]
			550	78	
			600	72	
Electric resistance	Continuous	C ₁₈	450	73	[23]
			500	62	
			550	35	
Electric resistance	Continuous	–	500	63	[24]

waste and fresh cooking oils, respectively, at different process temperatures. Pyrolysis experiments were performed at temperatures above 350 °C. This is caused by the fact that only small amounts of pyrolysis-volatile were generated below 350 °C. A previous study reported that at a temperature of 350 °C, a lot of un-pyrolyzed oils product (77 wt.%) and small amounts of bio-oils and gases have been obtained [19]. This phenomenon was also reported in other study that evaporation started at 325 °C for waste cooking oil [21].

The pyrolysis product of bio-oil is directly proportional to the temperature rises. The increase of temperature accelerates the rate of decomposition of raw materials. It was found that temperature of lower than 400 °C; less bio-oil was obtained compared to other temperature variations for both WCO and FCO. It might be due to the incomplete decomposition of the samples lead to less oil converted into bio-oil and gas. This incomplete decomposition impacts on the high yield of solid products. In this condition, the solid product obtained refers to a mixture of un-pyrolyzed oil with charcoal.

The highest yields of bio-oil product in this work were obtained at temperature of 500 °C for both WCO and FCO. It was influenced by decomposition rate in the pyrolysis process where the heat generated at this temperature was enough to break chemical bonds of raw material that the volatile content was evaporated. The faster the rate of chemical decomposition, the faster the breakdown process of chemical molecules of raw material. At that condition, volatile compounds of raw materials rapidly evaporated and tended to form bio-oil as the main product. The yield of bio-oil increased as the increase of reaction temperature, and then decrease after being reached the maximum point [22].

At a pyrolysis temperature of 550 °C, the yield of bio-oil dropped to 72 wt.% and 58 wt.% for WCO and FCO, respectively. The reduction of bio-oil yield at 550 °C is might be due to the secondary cracking and carbonization reaction of WCO and FCO to generate higher quantities of incondensable gas and carbonaceous char products. This result can be inspected from the increased yield of both the pyrolysis gas and char products. At high pyrolysis temperatures of 550 °C and above, the yields of gas product increase due to secondary cracking reactions. It was also possible that carbonization has also taken place during the pyrolysis process at temperatures of more than 500 °C and this has led to an increase in the yield of carbon char products of 1 wt.% and 2 wt.% for WCO and FCO, respectively.

Table 2 shows that the bio-oil obtained from microwave-enhanced pyrolysis exhibited higher yields at relatively lower temperatures compared to those produced from WCO pyrolyzed under conventional electric heating. Pyrolysis of WCO under conventional electric heating needed higher temperatures in order to obtain the maximum bio-oil yield. These results indicate that faster thermochemical decomposition rate of raw materials can be achieved with the assist of microwave heating. Table 2 also indicates that continuous pyrolysis processes provided better performance compared to batch processes. This happened because the continuous pyrolysis process is able to perform fast or flash pyrolysis process which is generally applied for bio-oils production as also reported in other studies by Chang et al. [23] and Chiaramonti et al. [24].

In this study, the largest yield of bio-oil product was achieved at relatively high temperature of 500 °C. This is mainly due to the carbon

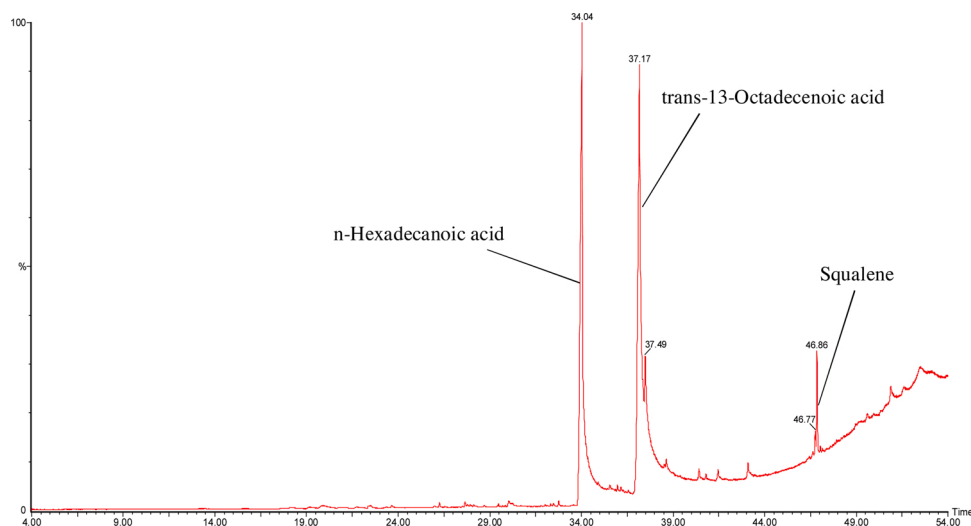


Fig. 7. Chromatogram of WCO from GC-MS analysis.

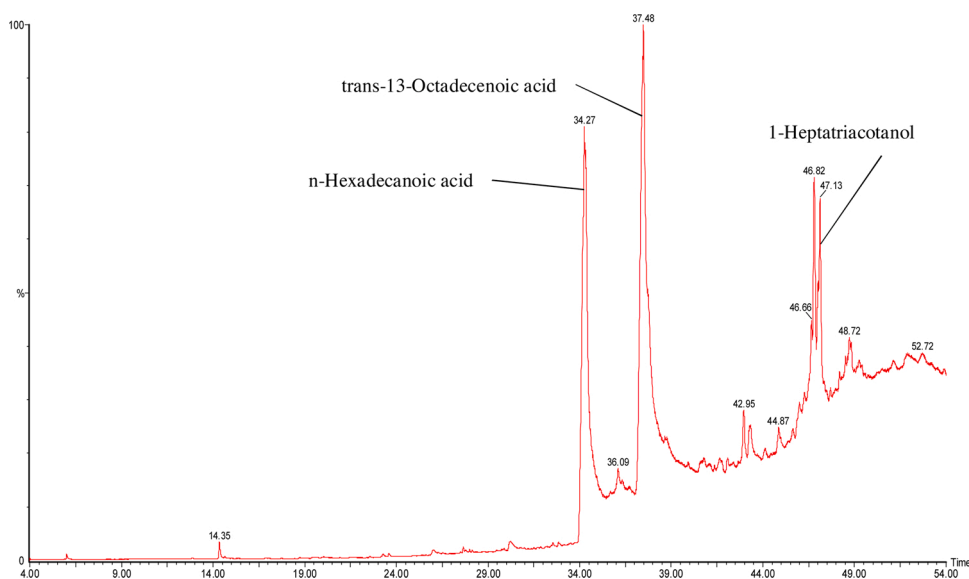


Fig. 8. Chromatogram of FCO from GC-MS analysis.

Table 3

Chemical composition (wt.%) and HHV (MJ/kg) of the feedstocks.

Compound	Formula	WCO	FCO
n-Hexadecanoic acid	C ₁₆ H ₃₂ O ₂	37.43	36.81
Trans 13-Octadecanoic acid	C ₁₈ H ₃₄ O ₂	42.02	44.26
Oleic acid	C ₁₈ H ₃₄ O ₂	18.32	–
Squalene	C ₃₀ H ₅₀	2.23	–
1-Heptatriacotanol	C ₃₇ H ₇₆ O	–	12.25
Z-(13,14-Epoxy/tetradec-11-en-1-ol acetate	C ₁₆ H ₂₈ O ₃	–	6.67
HHV		39.3	39.1

chain of waste cooking oil employed is longer than that of raw materials used in other studies. This proved that the longer the carbon chain in the oil sample, the higher the pyrolysis temperature is needed.

3.3. Chemical composition of the feedstock and bio-oil product

3.3.1. Feedstock composition

Gas chromatography-mass spectrometry (GC-MS) is an analytical method that commonly applied for identification of different substances within a sample in which the sample is heated in order to separate its chemical elements and to identify their molecular level contributions.

The GC-MS chromatograms of the feedstock (WCO and FCO) are presented in Figs. 7 and 8, respectively. Identification of the characteristic peaks was carried out by employing NIST MS 2.0 software to their spectrum. The results showed that response values and peak identification provided a high accuracy of over 99 % (or less than 1 % error) that is within the allowable level of accuracy. The identified molecular compounds for both feedstocks are also provided in Table 3.

In general, both samples mainly composed of palmitic and oleic acids. The data showed that waste cooking oil contained about 37.43 wt. % palmitic acid and 60.34 wt.% oleic acid, while for fresh cooking oil there were 36.81 wt.% and 44.26 wt.%, respectively. This is consistent to a previous study that majority of fatty acid of fresh and waste cooking oils is composed of oleic and palmitic acids [19]. Fresh and waste cooking oils also consisted of high carbon and hydrogen elements. Waste cooking oil had relatively short-chain of hydrocarbons than fresh cooking oil. This is due to the repeated frying process of waste cooking oil causing carbon chain cracking. The high number of hydrocarbons in fresh and waste cooking oils indicated that both of the feedstock could be pyrolyzed and converted into alternative fuel.

3.3.2. Bio-oil product composition

There were different chemical compounds and compositions of bio-

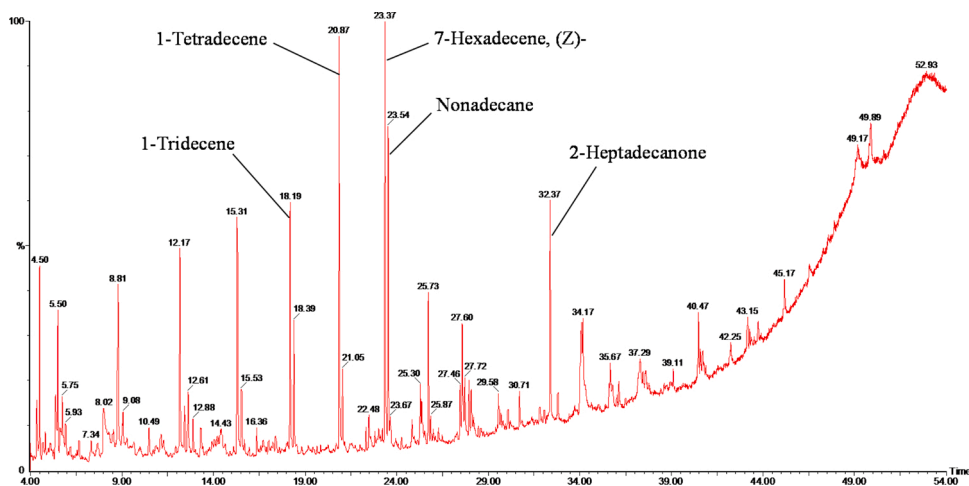


Fig. 9. GC-MS chromatogram of bio-oil from WCO produced at 500 °C.

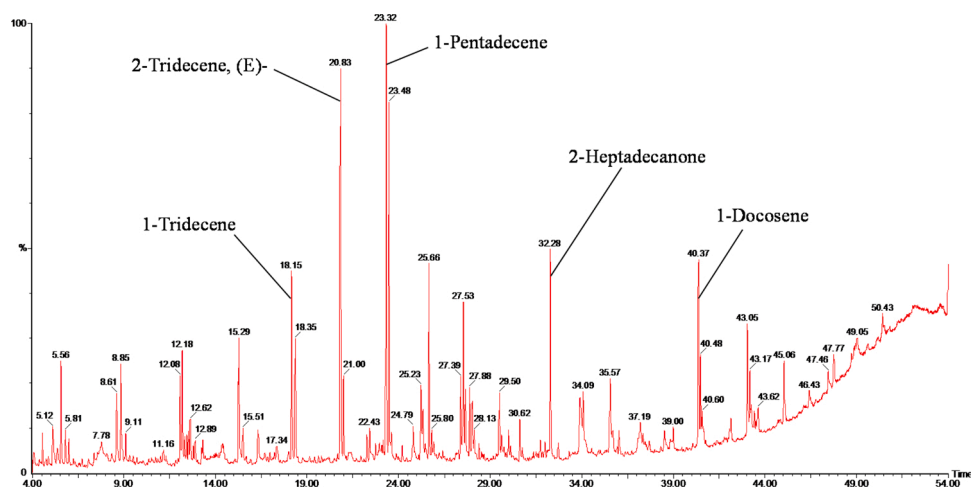


Fig. 10. GC-MS chromatogram of bio-oil from FCO produced at 500 °C.

Table 4

Chemical composition (wt.%), carbon component (wt.%), and HHV (MJ/kg) of the bio-oils.

Compound	WCO				FCO		
	400 °C*	450 °C	500 °C	550 °C	400 °C*	450 °C	500 °C
Aliphatics							
Alkanes	55.50	49.85	31.38	40.14	50.42	71.91	36.91
Cycloalkanes	4.78	5.32	–	5.56	4.13	3.00	4.31
Alkenes	39.72	44.83	58.25	45.05	45.45	24.07	53.34
Alkuna	–	–	–	–	–	–	5.44
Aromatic	–	–	10.37	9.26	–	1.02	–
Sum	100	100	100	100	100	100	100
Carbon component							
C ₅ -C ₉	8.26	8.93	12.91	22.09	7.32	11.26	7.83
C ₁₀ -C ₁₅	43.78	54.39	46.19	34.42	52.41	44.97	44.74
C ₁₆ -C ₂₀	47.96	36.68	39.48	43.50	35.71	40.95	39.54
C ₂₁ -C ₂₅	–	–	1.42	–	2.35	–	5.83
C ₂₆ -C ₃₀	–	–	–	–	2.21	2.82	2.06
Sum	100	100	100	100	100	100	100
HHV	44	46	46	46	–	–	–

* Data source: [20].

oil from waste and fresh cooking oils for each pyrolysis temperature. This is due to they have different feedstock composition and rate of decomposition. Based on the yield of bio-oil discussed previously, the largest yield of bio-oil product was achieved at pyrolysis temperature of 500 °C. So that, this condition was used for bio-oils chromatogram evaluation. The bio-oils GC-MS chromatograms produced at 500 °C for both waste and fresh cooking oils are depicted in Figs. 9 and 10, respectively. As can be observed in Fig. 9, bio-oil product of WCO mainly has domain compounds of C₆-C₂₂ bonds. This is achieved from the cracking of WCO compounds that have longer carbon bonds from C₁₆ to C₃₀.

The longer the carbon bond in the oil sample, the more compounds are produced. It was shown by the number of bio-oil compounds produced from fresh cooking oil sample (hydrocarbon chain ≤ 37) more than that of used cooking oil sample (hydrocarbon chain ≤ 30). For instance, the highest number of compounds produced from waste cooking oil was obtained at a temperature of 500 °C, where there were 25 peaks with 18 identified compounds. Among them, 1-Tetradecene was the most common compound found in bio-oil which occupies 14.20 wt.%. In the meantime, at the same temperature of 500 °C, there were 28 peaks bio-oil compounds from fresh cooking oil sample as shown in Fig. 10. Twenty-five compounds were identified at this temperature, in which the bio-oil compounds were more dominated by 1-Pentadecene which occupies 11.10 wt.%. The high number of

compounds produced at 500 °C also showed that the molecular breakdown process takes place with high intensity.

The bio-oils product of waste and fresh cooking oils pyrolysis contained many hydrocarbon compounds. There were several compounds in bio-oil such as alkanes, alkenes, cycloalkanes, aromatic, and other unknown components. The main compounds of bio-oils generated at different process temperatures for WCO and FCO are tabulated in Table 4.

This study showed that the dominant compounds obtained were aliphatic hydrocarbons (90–100 wt.%) and slightly aromatic hydrocarbons (1–11 wt.%). Alkanes and alkenes were found to be the biggest aliphatic hydrocarbons in each bio-oil product. The aliphatic hydrocarbons in this study also consisted of small amounts of alkuna and cycloalkanes. Alkuna or alkyne of 5.44 wt.% was only found in bio-oil from FCO cracking at a temperature of 500 °C. The formation of Alkuna is very possible to occur at high temperatures. In the presence of heat, saturated hydrocarbons can eliminate hydrogen gas to form unsaturated hydrocarbons. This means that when saturated hydrocarbons of alkanes or alkenes are heated to high temperatures in absence of air, a mixture of saturated and unsaturated hydrocarbons (e.g. alkenes and alkuna) is formed [27–29]. This condition then caused a reduction in the alkane's content at 500 °C as shown in Table 4. Other studies suggest that the formation of alkynes can occur at relatively lower temperatures with the aid of catalysts such as silica, alumina or carbonaceous

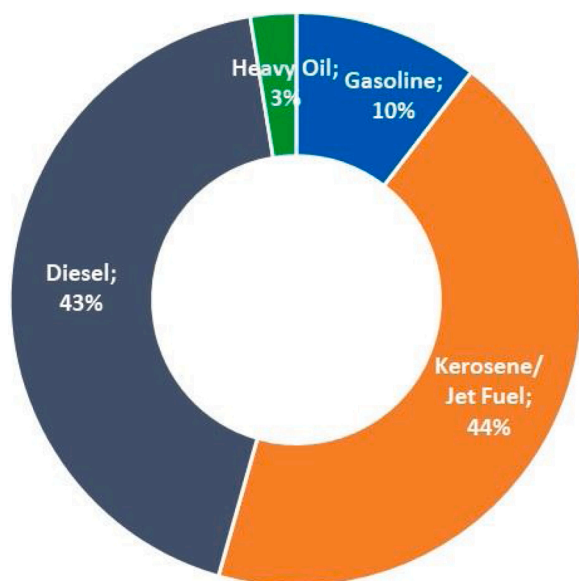


Fig. 11. Average yields of fuel fractions produced from microwave distillation of bio-oil from WCO.

materials [30]. In addition, there was the occurrence of components cracking to generate several aromatic structures, probably originated from the cyclization and aromatization reactions during the pyrolysis process [19].

Alkanes obtained ranged from 30–72 wt.%, while alkene ranged between 24–58 wt.%. Liquid hydrocarbons of alkanes from nonane (C_9H_{20}) to nonadecane ($C_{19}H_{40}$) derived from thermal decomposition of WCO were found to be the compounds with higher concentration in bio-oil. In the meantime, compared to alkanes, alkenes consisted of shorter liquid hydrocarbon chain compounds, especially C_9 – C_{16} . Hexadecene ($C_{16}H_{32}$) and tetradecene ($C_{14}H_{28}$) were the most dominant compounds in alkene with a range of 8–15 wt.%. The results also showed that bio-fuels from FCO raw material were mainly composed of longer carbon chains compounds in comparison to those from WCO. The bio-oils composed of nonane (C_9H_{20}) to octadecane ($C_{26}H_{54}$) compounds for alkanes group and 1-Nonene (C_9H_{18}) to 1-Docosene ($C_{22}H_{44}$) compounds for alkenes group.

The obtained aliphatic hydrocarbon compounds have a high percentage of 90–100 wt.%. At low temperatures of 400 °C, the aliphatic hydrocarbon compounds of each raw material have a percentage of 100 wt.%. Aromatization reaction using WCO raw material occurred at temperatures of 500 °C (10.37 wt.%) and 550 °C (9.26 wt.%), while FCO occurred at a temperature of 450 °C (1.02 wt.%). Bio-oil products with high aliphatic hydrocarbons content have the potential as fuel and chemical raw materials [18]. In addition, the highest content of green-diesel (C_{10} – C_{15}) of bio-oil was achieved at a temperature of 450 °C of 54.39 wt.% for WCO and 400 °C of 52.41 wt.% for FCO raw materials.

Table 5
Comparison of fuel fractions in the bio-oil from different oil feedstocks.

Heating method	Oil feedstock	Gasoline (wt.%)	Kerosene/Jet fuel (wt.%)	Diesel (wt.%)	Heavy oil (wt.%)	Reference
Microwave	Waste cooking oil	10	44	43	3	Present study
Electric resistance	Waste cooking oil	40	–	34	2	[23]
Microwave	Waste automotive engine oil	69	16	15	4	[18]
Electric resistance	Waste machinery oil	38	1	14	53	[31]
Electric resistance	Scrap tire + waste lubricant oil	40	18	13	34	[32]
Electric resistance	Used sunflower oil	23	–	–	45	[33]
Electric resistance	Waste cooking oil	–	–	–	75	[34]

Table 6
Comparison of fuel properties of green-diesel fraction with fossil diesels (Pertamina Dex and Solar).

Parameters	Units	Results				
		Green-Diesel fraction	Pertamina Dex*		Solar**	
			Min	max	min	max
Density @ 15 °C	kg/l	0.8325	0.820	0.860	0.815	0.860
Kinematic viscosity @ 40 °C	mm ² /s	2.620	2.0	4.5	2.0	4.5
Kinematic viscosity @ 100 °C	mm ² /s	1.122	–	–	–	–
Total acid number	mgKOH/g	0.2	–	0.3	–	0.6
Flash point PMCC	°C	54.5	55	–	52	–
Pour point	°C	–9	–	18	–	18
Final boiling point	°C	340	–	370	–	>370
Higher heating value	MJ/kg	41.14	–	56.67	–	43.04
Cetane index	–	50.3	48	–	45	–

* Data source: [35].

** Data source: [36].

3.4. Bio-oil distillation and green-diesel properties

The bio-oil produced from previous microwave-assisted pyrolysis process of cooking oil particularly WCO was further processed under microwave-assisted distillation reactor for obtaining the biofuel fractions. The oil sample for distillation processes was bio-oil produced at 450 °C as it had the highest green-diesel content. The result of fuel fractions obtained from microwave distillation process is presented in Fig. 11.

The distillation results as given in Fig. 11 indicated that from a total of 500 g of bio-oil produced around 52 g of gasoline fraction, 220 g of kerosene or jet fuel fraction, 215 g of green-diesel fraction, and only 13 g of heavy fuel fraction. The results showed that heavy fuel fraction and jet fuel fraction were found to be the lowest and the highest fuel fractions in bio-oil, respectively. Moreover, in terms of percentage of the yield, the average gasoline fraction was around 10 wt.%, jet fuel was 44 wt.%, green-diesel was 43 wt.%, and heavy fuel was about 3 wt.%. The obtained fuel fractions are in accordance with the bio-oil composition results in which the bio-oil content was more dominated by tetradecane, 7-hexadecene, (Z)-, and nonadecane with an amount of 12.35 wt.%, 13.05 wt.%, and 17.68 wt.%, respectively as previously shown in Fig. 9. These compounds are classified as kerosene/jet fuel and diesel fractions.

Table 5 shows that the bio-oil produced under microwave-enhanced pyrolysis exhibited higher quantities of C_{10} – C_{19} light fractions compared to those attained from the oils pyrolyzed under conventional electric heating. Meanwhile, pyrolysis of oil under conventional electric heating tended to have more amounts of heavy hydrocarbon fractions. This showed that microwave irradiation intensity might be contributed in enhancing cracking reactions that could not be attained with

Table 7

Energy consumption and energy recovery evaluation in bio-oil production from pyrolysis of WCO under microwave irradiation.

Process condition	E_{WCO} (kJ/h)	E_{Py} (kJ/h)	E_{BO} (kJ/h)	E_{Py}/E_{WCO} (%)	E_{BO}/E_{WCO} (%)	$E_{P, ratio}$	$E_{P, in}$ (kJ/h)	$E_{P, loss}$ (kJ/h)	$E_{P, recovery}$ (kJ/h)	(%)
Temperature (°C)										
400	4009	1248	2563	31	64	2.1	5257	2694	2563	49
450	4009	1560	2993	39	75	1.9	5569	2575	2993	54
500	4009	1872	3593	47	90	1.9	5881	2287	3593	61
550	4009	2340	3378	58	84	1.4	6349	2971	3378	53
WCO feed rate (kg/h)										
0.051	2004	936	1466	47	73	1.6	2940	1474	1466	50
0.102	4009	1560	2993	39	75	1.9	5569	2575	2993	54
0.204	8017	1755	6015	22	75	3.4	9772	3757	6015	62
0.306	12,026	2574	8713	21	72	3.4	14,600	5887	8713	60

conventional heating [17]. These results indicate that the application of microwave heating for thermochemical decomposition of materials could solve the limitation of conventional heating, in which microwave-heated pyrolysis can enhance cracking reactions to break longer hydrocarbon chains into shorter ones. Moreover, the type of oil raw material used also affects the composition of the bio-fuels fraction as they actually have different hydrocarbon content.

The green-diesel fraction was then analyzed for obtaining its fuel properties such as density, kinematic viscosity, total acid number, flash point, etc. Table 6 shows green-diesel fraction properties and its comparison with two grade of fossil diesel fuels that are commercially available in Indonesia called Pertamina Dex (high grade diesel fuel) and Solar (low grade diesel fuel).

According to Table 6, the green-diesel fraction originated from waste cooking oil showed good fuel properties agreement to that of the fossil diesels. The density, viscosity, and pour point of the green-diesel fraction were found within the prescribed range of diesels given by PT. Pertamina, Indonesia. The flash point of the green-diesel fraction was found to be higher than that of Solar and quite close to that of Pertamina Dex. The relatively high flash point suggests that most of the components with lower boiling point in the bio-oil had been removed, although the final boiling point of the green-diesel fraction was quite lower than that of Pertamina Dex and Solar.

Another important fuel property of the fuel is heating value that is also listed in Table 6 in term of higher heating value (HHV). The HHV of fuels represents the gross energy output of combustion in the engine combustion chamber. As shown in the table, the HHV of green-diesel fraction was relatively lower compared to that of the fossil diesels. The lower HHV of green-diesel fraction might be due to the fuel constituent compounds being dominated by lighter hydrocarbons which have low heating values. This also closely related to the final boiling point of compounds in the green-diesel fraction which is lower than that of fossil diesel fuels.

Total acid number figures the quantity of acidic chemicals in the fuel oils and normally used as an indicator of the quality of fuel oils. The acidity of fuel oils is also represented by the presence of oxygen in fuel oils. Lower acid content is needed to avoid severe corrosion in the fuel supply system and in the combustion chamber. In this work, total acid number of the green-diesel fraction was found to be relatively low which is comparable to that of Pertamina Dex, showing it has good fuel quality of total acid number property. Moreover, cetane index of the green-diesel fraction was higher than that of Pertamina Dex and Solar. The quality of diesel fuels is also highly associated to the cetane index. The cetane index illustrates how fast fuel can be ignited in the compression chamber. This shows that the fuel which has a high cetane index will generally combust faster in the engine combustion chamber. As such, engine working conditions are usually quieter and smoother and produce less harmful emissions.

3.5. Energy consumption and recovery

The assessment of energy consumption and energy recovery provides valuable information that illustrates the energy efficiency of the process, including WCO pyrolysis and bio-oil distillation under microwave irradiation used in this study. In addition, this information can also be used to evaluate whether the process is feasible to be applied on a larger scale. For this purpose, a microwave input power (MP_{in}) of 1.3 kW was used as a basis for calculating electrical energy consumption and only the HHV of bio-oil and green-diesel products were included in the assessment.

Table 7 shows estimates of the energy recovery compared with the energy consumption in bio-oil production through WCO pyrolysis under microwave irradiation both at various temperatures and feed flow rates. It should be noted that the effects of temperature on WCO pyrolysis were performed at a fix feed flow rate of 0.102 kg/h, while the effects of feed flow rate were studied at a constant temperature of 450 °C. Assessment of the energy content of WCO (E_{WCO}), the electrical energy needed for pyrolysis process (E_{Py}), and the energy content of bio-oil (E_{BO}) were adapted from previous studies [18,19] as the following equations:

$$E_{WCO} = HHV_{WCO} \times \text{WCO feed rate} \quad (4)$$

$$E_{Py} = MP_{in} \times \text{"on" duration of magnetron during pyrolysis process} \quad (5)$$

$$E_{BO} = HHV_{BO} \times \text{WCO feed rate} \times \frac{Y_{BO}}{100} \quad (6)$$

Equations above were then used to calculate the energies involved in pyrolysis process as follows:

$$E_{P, ratio} = \frac{E_{BO}}{E_{Py}} \quad (7)$$

$$E_{P, in} = E_{WCO} + E_{Py} \quad (8)$$

$$E_{P, loss} = E_{P, in} - E_{BO} \quad (9)$$

$$E_{P, recovery} = E_{P, in} - E_{P, loss} \quad (10)$$

As shown in Table 7, on the one side, the electrical energy needed for pyrolysis process (E_{Py}) increased with the increase of reaction temperature, ranging from 2563 kJ/h to 3378 kJ/h that is equivalent to 31%–58% of the HHV of WCO. At higher pyrolysis temperature, more electrical energy was consumed as it needed to supply sufficient heat to sustain the reaction temperature. On the other side, the electrical energy usage for pyrolysis process with regard to HHV of WCO decreased with the increase of feed flow rate under the investigated condition. The lower electrical energy usage at higher feed flow rates might be due to the heat generated by the microwave can be more effectively used to heat the feed of WCO to be converted into bio-oil. The result also showed that the energy content of waste cooking oil can be recovered well as indicated by the high energy content of bio-oil which is proportional to the increase in the yield of bio-oil products. In general, the bio-oil products contained about 64%–90% of the energy content of waste

Table 8
Energy consumption and energy recovery evaluation in green-diesel production under microwave irradiation.

WCO feed rate (kg/h)	No of runs	E_{WCO} (kJ/h)	E_{Py} (kJ/h)	E_{BO} (kJ/h)	E_{Dis} (kJ/h)	E_{GD} (kJ/h)	E_{GD}/E_{BO} (%)	$E_{D,ratio}$	$E_{D,in}$ (kJ/h)	$E_{D,loss}$ (kJ/h)	$E_{D,recovery}$ (kJ/h)	(%)
0.051	16	32,069	14,976	23,460	3120	9029	39	2.9	73,625	64,596	9029	12
0.102	8	32,069	12,480	23,948	3120	9217	39	3.0	71,617	62,400	9217	13
0.204	4	32,069	7020	24,061	3120	9260	39	3.0	66,269	57,009	9260	14
0.306	3	36,077	7722	26,139	3120	10,060	39	3.2	73,059	62,999	10,060	14

cooking oil.

Table 7 also shows that the bio-oil products produced from the applied pyrolysis process have much higher energy content than the electrical energy used to carry out the pyrolysis process. This is indicated by a good energy ratio in the range of 1.4–3.4, where the energy ratio increased significantly with the increase of feed flow rate. In addition, the estimation results showed that although in small-scale equipment, the energy recovery was quite high in the range of 49 %–62 % of the input energy involved in the pyrolysis process. Thus, regardless of the assumptions used in this energy estimation, the pyrolysis process of WCO using a microwave reactor appears to be able to produce high energy content of bio-oil and high energy recovery in the system.

Table 8 shows estimates of the energy recovery compared with the energy consumption for green-diesel production. In this work, there were two main processes applied for the production of green-diesel from WCO, namely microwave-assisted pyrolysis of WCO to produce bio-oil, and microwave-assisted distillation of pyrolytic bio-oil to separate green-diesel fraction. So, these two processes are involved in the estimation of energy consumption and energy recovery. It should be noted that each distillation process used about 500 g of bio-oil, so that a minimum of 800 g of WCO feed is needed for bio-oil production in the pyrolysis processes. In this evaluation, the pyrolysis process is assumed to be carried out several times depending on the feed flow rate used. As a consequence, the E_{WCO} , E_{Py} , and E_{BO} values in the table are then multiplied by the number of runs. While the electrical energy needed for distillation process (E_{Dis}), the energy content of green-diesel (E_{GD}) and others are calculated based on the following equations:

$$E_{Dis} = MP_{in} \times \text{"on" duration of magnetron during distillation process} \quad (11)$$

$$E_{GD} = HHV_{GD} \times \text{WCO feed rate} \times \text{no. of runs} \times \left(\frac{Y_{BO} \times \text{GD Fraction}}{100} \right) \quad (12)$$

$$E_{D,ratio} = \frac{E_{GD}}{E_{Dis}} \quad (13)$$

$$E_{D,in} = E_{WCO} + E_{Py} + E_{BO} + E_{Dis} \quad (14)$$

$$E_{D,loss} = E_{D,in} - E_{GD} \quad (15)$$

$$E_{D,recovery} = E_{D,in} - E_{D,loss} \quad (16)$$

As shown in Table 8, the green-diesel fuel produced from the applied distillation process has a much higher energy content than the electrical energy used for distillation process. The energy ratio was relatively high ranging from 2.9 to 3.2, where the energy ratio increased as the feed flow rate increases. Moreover, the estimation results showed that to produce green-diesel in this work, the energy recovery was in the range of 12 %–14 % of all the input energy involved in both pyrolysis and distillation processes. This is apparently low because the energy content of other fuel fractions, especially gasoline and kerosene, is ignored. Inclusion of their energy content in the estimation will give prominent results.

It should be noted that in industrial scale equipment, although the energy used for the collection and transportation of WCO is normally taken into account as the input energy; the utilization of energy content from gas and solid products produced from the pyrolysis process, and

heat integration and recovery are also carried out. This is certainly expected to increase the amount of energy that can be recovered from the pyrolysis and distillation system. Thereby, the pyrolysis and distillation processes of WCO under microwave irradiation appear feasible to be applied in producing high energy content of liquid bio-fuels.

4. Conclusion

Liquid bio-fuels from waste cooking oil were successfully produced under microwave-assisted pyrolysis and distillation. In contrast to the effect of feed flow rate, pyrolysis temperature and type of cooking oils played a prominent role in the yield and composition of bio-oil. Both WCO and FCO required higher pyrolysis temperatures to effectively break the long-carbon chain of the liquid sample. Compared to 500 °C, although pyrolysis temperature of 450 °C provided a relatively lower bio-oil yield, the bio-oil product contained more amounts of jet fuel and green-diesel fractions. The fuel properties of green-diesel fraction showed good agreement to that of the fossil diesels. Based on energy estimates, application of microwave irradiation for pyrolysis and distillation processes showed high benefit for converting waste cooking oil into liquid bio-fuels, in particular for the production of green-diesel.

CRedit authorship contribution statement

Samsudin Anis: Supervision, Conceptualization, Methodology, Writing - review & editing. **Rais Alhakim:** Investigation, Data curation, Writing - original draft. **Wahyudi:** Visualization, Validation. **Ahmad Mustamil Khoiron:** Methodology, Formal analysis. **Adhi Kusumastuti:** Validation, Writing - review & editing.

Declaration of Competing Interest

The authors report no declarations of interest.

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