

Waste Oil Recycling Using Microwave Pyrolysis Reactors

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Abstract—Waste oil contains a variety of contaminants, including lead, magnesium, copper, zinc, chromium, arsenic, chlorides, cadmium, and chlorinated compounds. One gallon of waste oil can foul a million gallons of drinking water. This work identifies a unique method by which the spent oil is adequately recycled for reuse, the cost of recycling is relatively low compared from its production from crude oil as the numbers of purification stages are reduced. The Waste automotive engine oil was pyrolyzed in a continuous stirred bed reactor using microwave energy as the heat source; the yield and characteristics of the incondensable gaseous products are discussed. Examination of the composition of the gases is carried out and investigated. The experiment was carried out in a microwave reactor over an activated carbon, which acts as a microwave receptor. Operating temperature (from 200 to 1000 °C), controlled by power of microwave reactor, was varied to observe the consequence on the pyrolysis products. The chemical composition and product yield of the pyrolysis products (liquid oil and gas) were analyzed by using gas chromatography/mass spectrometry (GC/MS). The effects of both N₂ and waste oil flow rates on both the aliphatic and aromatic components are carried out and investigated. The effect of the microwave pyrolysis temperature on the pyrolysis products and the microwave pyrolysis energy are obtained.

Index Terms—waste oil, N₂ flow rates, microwave pyrolysis, gas chromatography/mass spectrometry (GC/MS), aliphatic and aromatic components, cracking

I. INTRODUCTION

Due to diminishing supply, crude oil will continue to rise in cost thereby making production of fuels from alternate sources more feasible. Further climate change will be accentuated by the continued use of fossil fuels due to release of environmentally unfriendly gases such as CO₂. [1]–[4] Omer. [5] Showed that people rely upon oil and this will continue for a few more decades. Other conventional sources may be more enduring, but are not without serious disadvantages. Power from natural

resources has always had great appeal. Coal is plentiful, but there is pollution in burning it. Nuclear power not universally welcomed since construction of the plant is energy-intensive and there is concern about the disposal of its long-lived active wastes. Barrels of oil, lumps of coal, even uranium come from nature but the possibilities of almost limitless power from the atmosphere and the oceans seem to have special attraction. Heberlein and Murphy [6] illustrated the urgent need to replace traditional fuels led to emergence of biodiesel and biohydrogen as interesting alternatives, both of which can be obtained via micro alga mediated routes. Singh, Pant, Olsen and Nigam [7] concluded that production of sustainable bio-energy is a challenging task in the promotion of bio-fuels for replacing the fossil based fuels to mitigate challenges of fossil based energy consumption. The commercial-scale production of algae requires careful consideration.

Waste engine oil is a high pollutant material that requires responsible management. Waste engine oil may cause damage to the environment when dumped into the ground or into water streams including sewers.[8] This may result in groundwater and soil contamination.[1] Recycling of such contaminated materials will be beneficial in reducing engine oil costs. In addition, it will have a significant positive impact on the environment [9] [10] The conventional methods of recycling of waste engine oil either requires a high cost technology such as vacuum distillation or the use of toxic materials such as sulfuric acid. Heberlein and Murphy [6] showed that treatment of waste has become one of the central problems of our age, as traditional waste repositories, such as landfills and ocean dumpsites, become less and less available. Lam, Russel, and H. A. Chase. [11] Deduced that the production of waste automotive engine oil (WO) is estimated at 24 million tons each year throughout the world, posing a significant treatment and disposal problem for modern society. Lam, Russell, Lee, Lam and Chase. [12] Represented that waste oil, containing a significant amount of elemental hydrogen (14 wt. %), also it is a potential source of hydrogen

energy. In combination with its high production volume and low cost (10 cents/gallon), waste oil is readily available and presents a cost effective resource for hydrogen production. Ogbeide [13] described an investigation to study the recycling of spent engine oil. From the research, it was discovered that when 25 liters of spent oil was recycled appropriately, 10 liters of lubricating oil was obtained from the process whereas 220 liters of crude oil would be required to produce the same 10 liters of oil. Hamawand, Yusaf and Rafat [8] addressed recycling of waste engine oils treated using acetic acid. Heberlein and Murphy [9] considered the advantages and disadvantages of thermal plasmas in waste treatment applications. Lam, Russell, Lee, and Chase. [14] Concluded that the disposal routes recover only the chemical value of the waste and they are becoming increasingly impracticable as concerns over environmental pollution, and the difficulties and additional costs of sludge disposal are romanticized due to the undesirable contaminants present in waste oil.

Microwave radiation provides a rapid and energy-efficient heating process compared to conventional technologies. The diffuse nature of the electromagnetic field allows microwave heating to evenly heat many substances in bulk, thereby offering an improved uniformity of heat distribution, excellent heat transfer, and providing better control over the heating process, [11]. Fernández, Arenillas and Menéndez [15] explored that microwave technology assist in waste management. Lam, Russell, Lee, Lam and Chase. [12] Illustrated that the use of microwave radiation offer additional advantages over traditional thermal heat sources and the combination of carbon-based material and the novel use of microwave heating in pyrolysis processes is of increasing interest as reflected by considerable recent researches. Microwave systems show a distinct advantage in providing a rapid, energy-efficient, and targeted heating process compared to conventional technologies and thus facilitating increased production rates and decreased production costs.

Fernández, Arenillas and Menéndez [15] presented that one of the thermo-chemical processes which is rapidly gaining importance in this field is pyrolysis. This process not only allows for higher energy recovery from the waste, but it also generates a wide spectrum of products. Although pyrolysis is still under development in the waste industry, this process has received special attention, not only as a primary process of combustion and gasification, but also as independent process leading to the production of energy-dense products with numerous uses. In pyrolysis techniques, the waste material is thermally cracked and decomposed in an inert atmosphere, with the resulting pyrolysis oils and gases able to be used as a fuel or chemical feedstock, and the char produced used as a substitute for activated carbon, though the use of this technology is not widespread as yet. The pyrolysis-oil is of particular interest due to its easy storage and transportation as a liquid fuel or chemical feedstock. [8] Jamjumras and Amornraksa [16] used C6

non-aromatic hydrocarbons obtained from olefins plant as a feedstock in microwave pyrolysis. The chemical composition and product yield of the pyrolysis products (liquid oil and gas) were analyzed by using gas chromatography/mass spectrometry (GC/MS). The results indicated that microwave pyrolysis has the potential to upgrade low value hydrocarbons into high value products. Lam, Russell, and H. A. Chase. [17] used automotive engine oil in a microwave-induced pyrolysis process, with the intention of assessing the suitability of the process in recovering valuable products from this otherwise difficult to dispose of waste. Although several studies have revealed the potential of pyrolysis as a disposal method for waste oil, the use of this technology is not wide spread at the present time. Microwave-induced pyrolysis (MP) is relatively new process that was initially developed. The results indicated that microwave pyrolysis has the potential to upgrade low value hydrocarbons into high value products.

The main objective was to convert the waste oil to petrochemical products suitable for use as a fuel or raw chemical feedstock. This paper explored the effects of varying the feed injection rate of waste oil, and the purge gas flow rate. The investigations of pyrolysis products at different temperatures have been reported. The effects of both waste oil and N₂ flow rates on the pyrolysis oil liquids, solids and gases are carried out and investigated. The variations of the pyrolysis waste oil aliphatic and aromatics with both the waste oil and N₂ flow rates are obtained and investigated.

II. EXPERIMENT

The oil was collected from a Dihatsu Grand Terious car oil change after 4000 km in use. The type of base oil used in this study is GULF MAX API SL (SAE 10W-30), run on unleaded fuel in Al-Baha, KSA. The first experiment was carried out with heating the oils collected to a temperature of over 250 °C for one hour, for the purpose of evaporating the water and the volatile substances in the used oil. The oil was then cooled to room temperature (16 °C, winter) and then equal amounts of this oil (10 mL) were added to a number of beakers. Before pyrolysis, the waste oil samples were filtered such that the size of any remaining particulates (i.e. metal particles, carbon soot's, and other impurities) were less than 100 µm. Microwave-heated pyrolysis of waste oil shown in Fig. 1 was performed in a quartz reactor (200 X 200 X 200 mm) filled with 1 kg of carbon, that stirred and heated by a 6 kW microwave oven at a heating rate of about 50°C/min over a range of pyrolysis temperatures (200–1000°C), feed injection rates (0.5–10 kg/h) and purge gas flows (0.1–1.00 L/min) to understand the influence of these process parameters on the final pyrolysis oils obtained; N₂ purge-gas was vented through the system to maintain the apparatus in an inert nitrogen atmosphere, and the particulate carbon, added initially into the reactor in one batch, was stirred to ensure a uniform temperature distribution throughout the reactor. The sample of waste oil was continuously added to the

reactor at each target feeding rate over a period of 3h as soon as the target pyrolysis temperature was achieved.

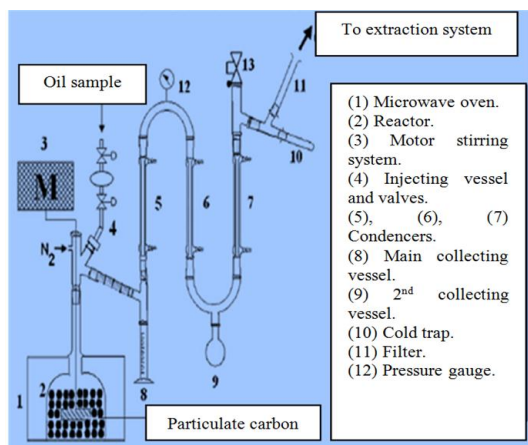


Figure 1. Microwave pyrolysis device.

Products generated in the pyrolysis reaction, consisting of a mixture of hydrocarbon gases, liquids, and suspended solids existing in a vapour phase, leave the reactor and pass through a condensation system, and condense into pyrolysis oil. The weight of residue material was measured. The residue materials are likely to be chars produced from tertiary cracking reactions of the pyrolysis process. These chars, which mostly accumulated on the surface of the carbon bed, were separated from the carbon particles using sieves (100 and 300 ml) and mixed with the metallic residues deposited on the filter installed between the reactor and the condensation system for later analysis. The yield of pyrolysis oil was determined by measuring the weight increase in the collecting vessels and filter. The pyrolysis oil was then transferred into glass bottles until further analysis. The gas yield was determined by mass balance and assumes that whatever mass of added sample that is not accounted for by the residue and pyrolysis oil measurements left the system in gaseous form. All the pyrolysis experiments were repeated several times and the data recorded is the average of the results obtained from three valid repeated runs performed under identical conditions. The virgin oil (FO), the waste oil, and the pyrolysis oils were examined for hydrocarbon composition by gas chromatography coupled with a mass selective spectrometry detector and a flame ionization detector. These oil samples were also analyzed by Fourier Transform Infrared Spectroscopy and elemental analyzer to identify their chemical functional group and elemental content (C, H, N, S, and O). In addition, the fuel properties of these oil samples (i.e. calorific value) was determined. The temperature of the carbon bed in the system was carried out using two thermocouples; one ducted into the middle layer of the carbon bed through the center of the shaft that protrudes from the bottom of the stainless steel stirrer shaft, the other enters the reaction chamber through a side port on the top of the reactor and is positioned at the top of the carbon bed.

III. RESULTS AND DISCUSSIONS

A. Effect of Temperature

Fig. 2 showed that at temperatures between 0 and 300°C, no liquid is produced, but only small amount of gases are produced. Up to 400 °C a large amount of residue that indicates incomplete decomposition. With temperature increment from 400 to 600°C, the amount of residue decreases coupled with liquids and gases increment. At temperature higher than 600 °C, the amount of residue decreases allowing the waste oil to vaporize, leaving the reactor and condensing into a liquid product. Also, the cracking of hydrocarbons into smaller molecules increases resulting in higher amounts of gaseous products. At temperature higher than 800 °C, the cracking occurs more rapidly and the molecules enter the gaseous state causes more rapid flow of gases where there is sufficient energy for causing cracking coupled with the shorter residence time of the oil in the reactor in agreement with Lam *et al.* [18].

It is clear that within the microwave pyrolysis process; at 400 °C, the wt. % of solids will be higher (about 70%) while the wt. % of the pyrolysis gases is 3wt. %. With the pyrolysis temperature increment up to 600°C the amounts of pyrolysis liquids reached its maximum values (about 78 wt. %), with an approximately equal amounts of both pyrolysis solids and gases (12wt% and 10wt% respectively). With temperature increment, the amounts of gases continuously increases (55 wt. %) coupled with the decrement of both the pyrolysis liquids and solids decrement (about 35wt.% and 10 wt. % respectively).

Fig. 3 illustrated the energy recovery estimated in the microwave pyrolysis process according to Lam *et al.* [18]. The results show that microwave-induced pyrolysis of waste automotive engine oil is able to produce significant quantities of commercially valuable products.

Such a slight olefins, gaseous hydrocarbon and liquid hydrocarbon oils containing BTX and benzene derivatives. 600°C seems to be the ideal temperature for waste engine oil pyrolysis based on the highest yield of valuable compounds in the liquid product and lower heating energy requirements at this temperature.

B. Effect of N₂ Flow Rate

Fig. 4 illustrated the effect of N₂ purge rate on the waste oil converted to pyrolysis gases, pyrolysis oils, and char residues. Increasing the N₂ purge rate up to 100 ml/min resulted in an increased yield of pyrolysis oil, gases and residue. It is also shown that the higher rates of N₂ purge gas from 100 to 300 ml/min resulted in increment of pyrolysis oil coupled with a decrement of both residue and gases. At rates more than 300 ml/min, the amounts of both pyrolysis oil and residue decreases while the amount of gases increases. At very high N₂ purge rates (800 ml/min), the yield of pyrolysis oil decreases, suggesting that the installed condensation system is unable to condense the pyrolysis volatiles at this considerably faster vapour flow rate, which leads to a higher corresponding yield of pyrolysis gases. The results show that extended heating of the pyrolysis volatiles in the reactor could promote different product compositions due to secondary reactions of the primary pyrolysis

product; hence it was observed that some waste oil is consumed in the production of pyrolysis gases and char in addition to pyrolysis oil.

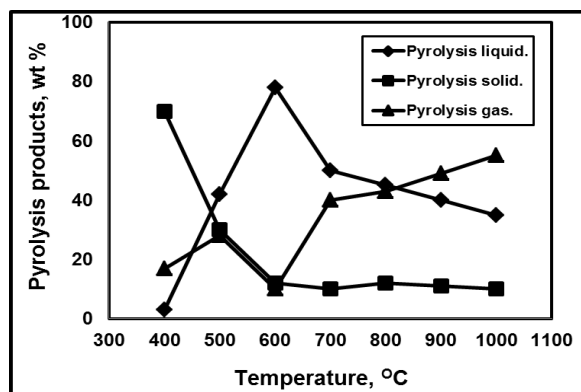


Figure 2. Effect of temperature on the waste oil pyrolysis products.

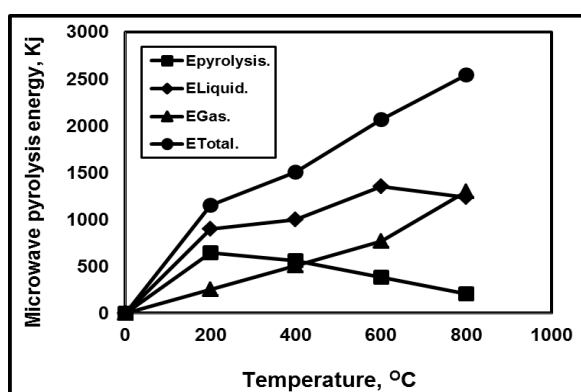


Figure 3. Variation of microwave energy recovery with temperature.

C. Effect of Waste Oil Feed Rate

Fig. 5 showed the effect of waste oil feed rate on the fraction of waste oil converted to pyrolysis gases, pyrolysis oils, and char residues. The increase in waste oil feed rate demonstrated a minor influence on the yield of pyrolysis oil, as the total increase in the yield of pyrolysis oil resulting from the increase in waste oil feed rate up to 2kg/h. With the increment of waste oil feed rate resulted in continuous increment of pyrolysis oil, while the amount of residue increases up to 4kg/hr. followed by continuous decrement, while the amount of gases decreases with the oil feed rate until the temperature increases over 600°C. Then the hydrocarbons in waste oil undergo cracking into shorter molecules, which then composed pyrolysis volatiles, the volume increase accompanying the phase change from liquid to vapour creates an increment of pressure in the reactor that drives the pyrolysis volatiles out of the reactor into the product collection system, decreases with the oil feed rate until the temperature increases over 600°C. Then the hydrocarbons in waste oil undergo cracking into shorter molecules, which then composed pyrolysis volatiles, the volume increase accompanying the phase change from liquid to vapour creates an increment of pressure in the reactor that drives the pyrolysis volatiles out of the reactor into the product collection system.

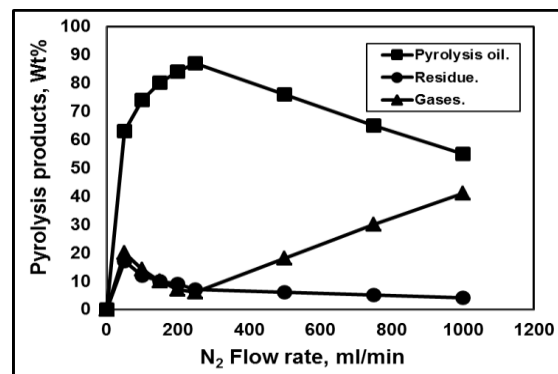


Figure 4. Effect of N_2 flow rate on the pyrolysis products wt%.

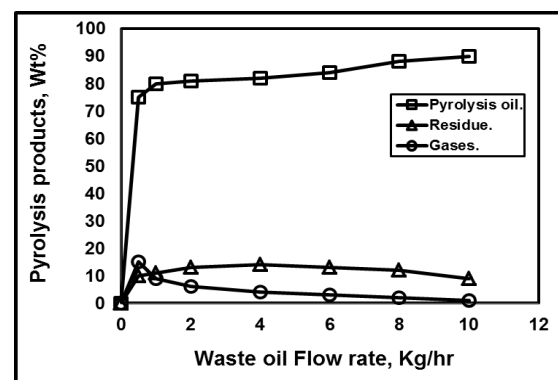


Figure 5. Variation of pyrolysis products wt% with the waste oil flow rate.

D. Effect of N_2 and Waste Oil on the Aliphatics

The aliphatic hydrocarbons were mostly alkanes and alkenes. These aliphatic hydrocarbons represent a potentially high-value chemical feedstock or fuel source. The pyrolysis oils were found to contain many different lengths of aliphatic chains, showing that waste oil was randomly cracked into short fragments of different carbon chain lengths. The wide distribution of aliphatic chains in the pyrolysis oils suggested that the thermal cracking of waste oil in this process predominantly follows the free-radical-induced random scission mechanism. This mechanism may have led to the production of hydrocarbon radicals that were romatizat by capturing the hydrogen atoms from nearby molecules, producing alkanes and alkenes via the free radical and b-scission reactions. Heavy n-alkanes and alkenes were then cracked to form lighter compounds. This accounts for the alkanes, naphthenes, and alkenes observed for each carbon number across all collected samples.

The decrease in purge and feed rates was thought to lead to an increased residence time of the pyrolysis volatiles in the reactor, resulting in a decrease in aliphatic content in the pyrolysis oils, and the aliphatic content improved towards smaller hydrocarbon chains. The amounts of alkanes and alkenes were reduced in favor of aromatic formation. In secondary reactions n-alkenes and dialkenes were found in the pyrolysis oils). Furthermore, the decreased concentrations of alkenes at low purge and feed rates (Fig. 6 and Fig. 7) suggest that it was the increased occurrence of these type secondary reactions

that had combined and converted the n-alkenes and dialkenes into aromatics, leading to increased formation of benzene, toluene, and alkylbenzenes.

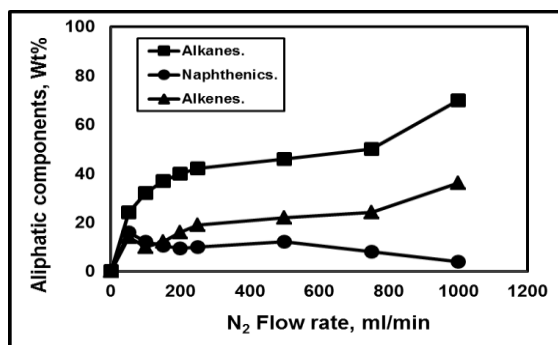


Figure 6. Variation of aliphatic components with N₂ flow rates.

Fig. 6 shows the effect of N₂ flow rate on the wt.% of aliphatics, it is clear that the trends are divided into three regions, at the first region, all the aliphatic components are increased with N₂ flow up to 50 ml/min. Between 50 and 800 ml/min, both the alkanes and alkenes increases while the naphthenics decreases. After 800 ml/min, there is parallel rapid increment of both alkanes and alkenes while the alkanes trend is found higher with a rapid decrement of naphthenics. The waste oil flow rate effect will be similar to the N₂ flow rate as shown in Fig. 7.

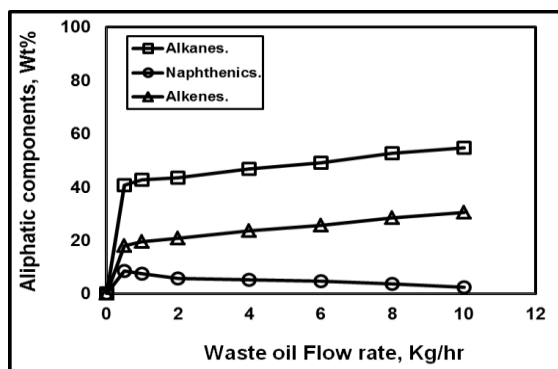


Figure 7. Variation of aliphatic components with waste oil flow rates.

E. Effect of N₂ and Waste Oil on the Aromatics

Valuable light aromatics such as benzene, toluene, and xylene were found in the pyrolysis oils. The aromatics were composed mainly of single ring alkyl aromatics, including benzene derivatives as well as benzene rings with short alkyl groups – mainly toluene, ethyl-benzene, and xylene. The substituent chains attached to the benzene rings. The decrease in N₂ purge and waste oil feed rates was thought to lead to an increased residence time of the pyrolysis volatiles in the reactor, resulting in an increase in aromatic content along with a decrease in aliphatic content in the pyrolysis oils, and the aliphatic and aromatic content improved towards smaller hydrocarbon chains. Depending on the degree of aromatization, condensation reactions of the ring structure of the aromatic compounds may occur subsequently and result in formation of heavier aromatics. Fig. 8 and Fig. 9 illustrate that with the increment of N₂ and waste oil flow

rate, the aromatic components increases followed by a rapid decrement of benzene, toluene and xylene while the alkylbenzene increases rapidly.

Fig. 8 shows the effect of N₂ flow rate on the wt.% of aromatics, it is clear that the trends are also divided into three regions, at the first region, all the aliphatic components are increased with N₂ flow up to 50 ml/min. Between 50 and 800 ml/min, all the aromatic components are decreased but the alkylbenzenes increases. After 800 ml/min, there is parallel rapid decrement of all aromatic components but the alkylbenzenes increases in higher rate. The waste oil flow rate effect will be similar to the N₂ flow rate as shown in Fig. 9.

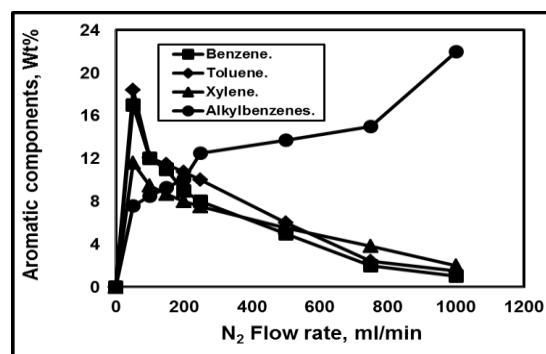


Figure 8. Variation of aromatic components wt% and the N₂ flow rate.

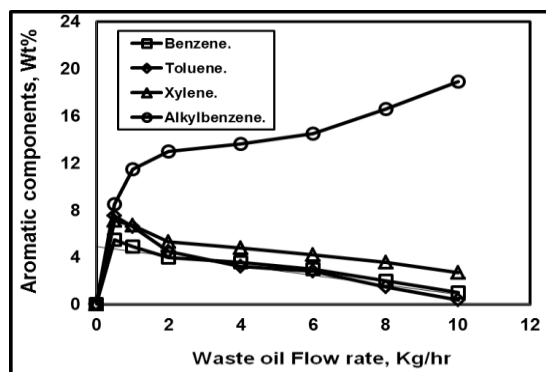


Figure 9. Variation of aromatic components wt% and the waste oil flow rate.

IV. CONCLUSIONS

The following conclusions are obtained.

The microwave pyrolysis process is found successful in the recycling of the waste engine oil.

Microwave-heated pyrolysis offers an exciting green approach to the treatment and recycling of automotive engine oil.

Both N₂ and waste oil flow rates were found to have effects on the fraction of original waste oil converted to pyrolysis gases, pyrolysis oils, and residues..

At the highest rates of N₂ and waste oil flow rates, the amounts of alkanes and alkenes increases while the amounts of naphthenics decreases.

At the highest rates of N₂ and waste oil flow rates, the amounts of alkylbenzene increases while the amounts of benzene, toluene and xylene decreases.

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