



Pyrolysis of waste oil in the presence of a spent catalyst



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ABSTRACT

A spent catalyst has great potential to convert waste oil into diesel oil. This study investigated the pyrolysis of waste oils in the presence of a spent catalyst, as well as the regeneration properties of the spent catalyst, such as the type of regeneration (in situ and ex situ) and time and temperature for spent catalyst regeneration. In addition, the effect of the spent catalyst, type of waste oil, weight ratio of the spent catalyst to the waste oil, and stability of the spent catalyst were evaluated. The yield of diesel oil was higher than 60% when waste oil was pyrolyzed at 370 °C.

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1. Introduction

Currently, more than 80% of energy sources are fossil fuels, particularly crude oil, natural gas, and coal [1]. Because known fossil energy sources are being rapidly exhausted, fossil fuel sources are predicted to be depleted in the near future. Crude oil, gaseous fuels, and coal are estimated to last only for approximately next 80, 150, and 230 years, respectively. The burning of fossil fuels have resulted in a reduction in the fossil fuel sources, increase in the demand and cost of petroleum-based fuels, and environmental hazards. Therefore, many studies focused on finding alternative new energy resources and utilizing them [1,2].

The treatment of waste has become one of the most crucial concerns of modern society in protecting the environment. Treating waste has several benefits that include utilizing waste energy sources, preserving valuable petroleum resources, protecting the environment from toxic and hazardous chemicals, reducing petroleum imports and thus improving foreign exchange, reducing greenhouse gas emissions, and promoting regional development and social structure, particularly in developing countries [3].

In recent years, millions of tons of waste oil have been disposed through dumping on the ground or in water, land filling, or nonenergy recovery [4]. Because global reserves of fossil fuels are limited, great efforts are being made to find alternate carbon sources for producing fuels [5]. Used or waste oil can be refined and treated to produce fuels or lubricating oil base stock. In addition,

waste oils pose an environmental hazard because of both their metal content and other contaminants [4]. Therefore, recycling waste oils is crucial [3].

Wastes oil include automotive engine oil, machinery oil, and lubricant oil. The waste automotive engine oil produced worldwide is estimated at 24 million tons per year, posing a considerable treatment and disposal problem for modern society [6,7].

The composition of waste machinery oil changes with its use because some contaminants, such as sulfur and oxidized compounds, hydrocarbons, and metals (chromium or lead), are present in the virgin oil. All waste oils of various origins are often mixed during collection and storage, producing a final contaminated residue that is often called waste machinery oil [7]. Waste oils can be reconstructed chemically by heating in an oxygen-free environment. This process is called pyrolysis, which is defined as chemical decomposition by the action of heat and generally refers to the chemical decomposition of organic materials heated in an environment with an insufficient supply of oxygen for combustion. Pyrolysis has certain advantages over other treatment methods for waste disposal [4]. The most crucial advantage of pyrolysis is that it does not pollute the environment when performed appropriately, because pyrolysis products such as gases, oils, and carbonaceous residues can be used as fuels. However, pyrolyzed oil can be polymerized again because it consists of many unsaturated hydrocarbons [4].

Pyrolysis is an option for converting waste oils into a more useful product. It has received considerable attention because of the multiphase product variation of yields in solid, liquid, and gas forms depending on process conditions [7]. In addition, using solid catalysts to facilitate pyrolysis has been reported, and the product

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selectivity is strongly affected by the presence and nature of heterogeneous catalysts and the presence of water steam and gas feed [8]. Some literature results are listed in Table 1. Applying a catalyst or catalyst-like mineral affected the heat required for this process, the yields, and composition of products [9]. Although this represents the total destruction of organic matter, a high-efficiency method from an energy perspective involves using waste oils as engine fuels. Pyrolysis can be valuable and requires further investigation [10]. Although several studies have revealed the potential of pyrolysis as a disposal method for waste oils, the use of this technology is not widespread currently [11].

Hydrotreating, hydrocracking, and fluid catalytic cracking (FCC) are the major steps in refining petroleum. Hydrotreating and hydrocracking catalysts are used to increase the yield of high-quality light oil fractions obtained from heavy crude oil and petroleum feedstock that contain high levels of impurities. FCC catalysts increase the yield of high octane gasoline obtained from crude oil [13]. Most catalysts used in refining or petrochemical applications deactivate relatively quickly with time. In addition, catalysts are typically composed of rare and expensive precious metals. Hence, using a simple, cost-effective, and efficient regeneration procedure to improve catalyst performance is essential [14].

A catalyst plays a crucial role in heterogeneous chemical reactions. However, catalysts have a limited life time. Catalysts can be deactivated by a long duration of reaction, thermal degradation, poisoning, and sintering. Hence, the reuse technology of spent catalysts is a worth consideration before becoming useless waste. In this study, spent catalysts from FCC process were examined. Spent catalysts are expected to reduce the operating temperature of waste oil degradation and to keep the stability to produce the diesel oil. Various types of waste oils were pyrolyzed to form diesel oil in the presence of a spent catalyst.

2. Experimental

2.1. Materials

Waste oils from waste motor (WM) oil (S: 0.31 wt%, Cu: 15.4 mg/kg, Ni: 0.874 mg/kg, Cr: 0.874 mg/kg, Cd: 0.485 mg/kg, Zn: 403 mg/kg, Pb: 5.63 mg/kg, Hg: 0.083 mg/kg, and As: 0.821 mg/kg.), waste machinery engine (WME) oil (S: 0.27 wt%), and waste bottom product (WBP) acquired after extracting the waste oil by distillation (S: 0.77 wt%) were obtained from Taichin Global Co., Taiwan. The waste oils were not purified before use. A spent

catalyst for residue oil cracking (Zeolite-like form) was provided by the CPC Corporation, Taiwan. Acetone (98%, Echo Chemical, Taiwan) and other reagents were analytical-grade chemicals.

2.2. Regeneration of the spent catalyst

Catalyst regeneration is the reactivation of a used catalyst. Three regeneration techniques are used for regeneration: acetone leaching, thermal regeneration, and a combination of both. (i) Acetone leaching: a spent catalyst and acetone (1:2 w/w) are mixed, and the solution is shaken for 1 h and then filtered to separate the solid catalyst from the acetone. (ii) Thermal regeneration: a spent catalyst is heated in two ways, ex situ (the spent catalyst regeneration and oil reaction are performed in separate reactors) and in situ (the regeneration of the spent catalyst and oil reaction are performed in the same reactor). A known amount of a spent catalyst (0–28 g) is introduced into the reactor and heated to a fixed temperature (300–370 °C) for a fixed duration (30–60 min) at atmospheric pressure. (iii) The combination method involves acetone leaching and heating of the spent catalyst.

2.3. Testing procedure

A schematic of the experimental apparatus is shown in Fig. 1. The apparatus consisted of a preheater, magnetic stirrer, reactor (conical flask), condenser 1, cold trap 1, condenser 2, cold trap 2, collecting vessel, and vacuum pump. The main functions of these components are explained briefly as follows.

The preheater and magnetic stirrer were used to provide heat and stirring, respectively. The operation was set for a semi-continuous process, in which 175 ml (approximately 140 g) of waste oil was introduced in the reactor at a rate of 25 ml/h. The waste oil was mixed with a spent catalyst (0–28 g) in a reactor, and the solution was then heated (300–370 °C) and stirred (0–200 rpm) using the magnetic stirrer for 1–7 h for the reactants to react and produce diesel oil in vapor form.

Diesel oil in vapor form was condensed using a vacuum pump in ethanol-cooled condenser 1 at –2 °C, and the condensed diesel oil accumulated in cold trap 1 at –2 °C. Diesel oil vapors that escaped condenser 1 and cold trap 1 were condensed again at –2 °C in ethanol-cooled condenser 2, and the condensed diesel oil accumulated in cold trap 2. The elemental concentrations of the final diesel oil product were analyzed. The results are as follows: S: 0.19%, Cu: 0.698 mg/kg, Ni: 0.0 mg/kg, Cr: 0.1 mg/kg, Cd: 0.0 mg/kg,

Table 1
Summary of diesel oil production through catalytic pyrolysis of various types of waste oils.

No	Oil source	W_o (g)	Catalyst	W_c (g)	T (°C)	t	Method	Yield (wt%)	References
1	Waste engine oil	–	Na ₂ CO ₃	10 ^a	360	–	Pyrolytic distillation	60	[2]
2	Waste engine oil	–	Zeolite	10 ^a	360	–	Pyrolytic distillation	60	[2]
3	Waste engine oil	–	Lime (CaO)	10 ^a	360	–	Pyrolytic distillation	60	[2]
4	Waste automotive engine oil	0.4–5 ^e	carbon	1 ^b	550	2	Continue microwave pyrolysis (5 kW)	88	[6]
5	Waste machinery oil	3.75	HZSM5	0.5 ^a	400	–	Batch thermal pyrolysis	65	[7]
6	Vegetable oil	2 ^d	HZSM5	2 ^c	400	–	Bench home-made 5 L stainless steel	60	[8]
7	Malaysian refuse derived oil	–	Zeolite	20 ^a	450	4	Batch thermal pyrolysis	17.8	[12]
8	Waste automotive engine oil	0.4–5 ^e	Carbon	1 ^d	550	1	Continue microwave pyrolysis (5 kW)	69	[11]
9	Waste machinery oil	20	Spent catalyst	12	370	1	Batch thermal pyrolysis	45	This study
10	Waste machinery oil	20	Spent catalyst	28	370	1	Batch thermal pyrolysis	73	This study
11	Waste machinery oil	20	Spent catalyst	12	300	1	Batch thermal pyrolysis	18.4	This study

t : reaction time, T : operation temperature, W_o : weight of the waste oil, W_c : weight of the spent catalyst.

^a wt%.

^b kg.

^c cm.

^d liter.

^e kg/h.

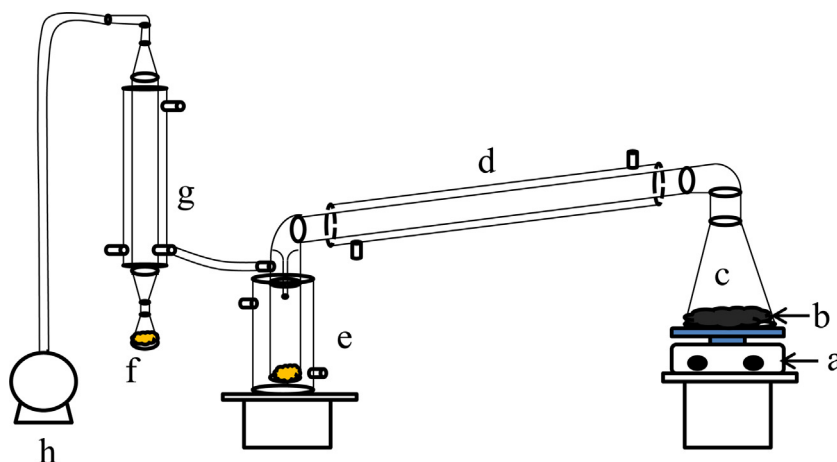


Fig. 1. Schematic of the experimental apparatus. (a) Magnetic stirrer, (b) position of thermocouple, (c) conical flask (500 ml), (d) condenser 1 (-2°C), (e) cold trap 1 (-2°C), (f) cold trap 2, (g) condenser 2 (-2°C), and (h) aspirator.

Zn: 9.57 mg/kg, Pb: 1.8 mg/kg, Hg: 0.02 mg/kg, and As: 0.838 mg/kg.

2.4. Analysis of the product

Diesel oil was drawn from cold traps 1 and 2 every hour and analyzed for its weight, absolute viscosity, and kinematic viscosity. Absolute viscosity (cP) was measured using a viscometer (Brookfield LVDV-II pro, USA) with the ASTM D4624 method. The viscosity of the waste oils (WM oil, WME oil, and WBP) was measured using a viscometer fitted with spindles S62, the viscosity of the diesel oil was measured using the viscometer fitted with spindles S61, and the spindle rotational speed was 60 rpm for all samples. The viscosity reading, torque percentage, and degree of settlement were recorded at 60 s intervals for each rotational speed setting. Absolute viscosity was measured in triplicate.

Kinematic viscosity (m^2/s) was measured using a Cannon-Fenske viscometer (300/PmT, $C=0.299$ min, temperature = -25°C), which was calibrated with a known compound (2-propanol, chloroform, ethanol, 1-butanol, or 1-pentanol) at 20°C . The flow time of a specific volume of the known compound flowing through the pipe capillary was measured. An equation showing the relationship between flow time and kinematic viscosity was then derived for use as a standard curve in measuring the kinematic viscosity of waste oil and diesel oil.

2.5. Equation used for measuring the catalyst activity

Based on diesel oil production from waste oil (Fig. 1), three types of products were collected: P1 from Cold trap 1, P2 from Cold trap 2, and P3 from a low-boiling gas from the aspirator.

For measuring the catalyst activity, there was one concern about P1 and P2. The catalyst activity was measured using the yield of diesel oil (%):

$$\text{Yield}(\%) = \frac{(P1 + P2)}{W_o} \times 100, \quad (1)$$

where W_o is the total weight of waste oil (g).

3. Results and discussion

3.1. Effect of the spent catalyst

The enhanced optimal use of waste engine oil would help reduce environmental liability, save waste oil disposal costs, and reduce the burden of fuel imports [15]. The technologies for

converting waste oil to diesel oil are well established and involve using an alkali, acid, or no catalytic reaction in the presence of supercritical methanol. Therefore, this study investigated the use of a spent catalyst for the degradation of waste machine oil to form diesel oil. To understand the effect of a spent catalyst on the degradation of waste oil, an experiment was conducted by comparing the yields of the products formed with and without the presence of a spent catalyst.

The WM oil (25 ml) was reacted with 12 g of a spent catalyst at 370°C and 200 rpm for 1 h without regeneration. The temperature of 370°C chosen is the best regeneration temperature, and the yield of diesel oil for 370°C is larger than those for below it. The yields of diesel oil formed in the presence and absence of the spent catalyst were 26% and 15%, respectively. This result showed that the spent catalyst increased the yield of diesel oil.

3.2. Regeneration of the spent catalyst

The spent catalyst used in this study was obtained from a refining petroleum oil company in Taiwan. This catalyst was unused (or its effectiveness is low). Therefore, it can be used to produce diesel oil from waste oil before its disposal in the environment. Before a catalyst is used for diesel oil production, it is regenerated. The catalyst in this study was regenerated using acetone leaching, thermal regeneration, and a combination of both.

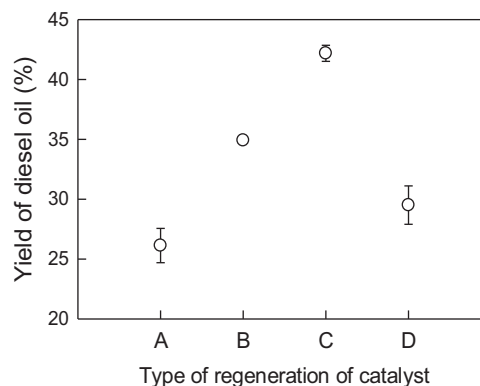


Fig. 2. Effect of the type of regeneration catalyst on the yield of diesel oil. WM oil = 25 ml, spent catalyst = 12 g, regeneration temperature = 370°C , regeneration time = 0.5 h, agitation = 200 rpm, reaction temperature = 370°C , and reaction time = 1 h. A: without regeneration, B: leaching with acetone, C: in situ thermal regeneration, D: combination of B and C.

According to the experimental results (Fig. 2), thermal regeneration was the optimal method for regenerating the spent catalyst because it led to the production of diesel oil in a high yield. Until the mid-1970s, hydroprocessing catalysts were regenerated in situ in unit reactors; however, ex situ regeneration gradually became the industry standard in Western countries. Other parts of the world are now frequently using the ex situ regeneration method, which is preferred for many reasons including corrosion concerns, safety, time considerations, and improved recovery of catalytic activity. As mentioned, a high degree of sophistication for regenerating most catalysts, for hydrotreating or hydrocracking, is obtained using ex situ thermal regeneration. In addition, high performance of the catalyst is obtained for the second run of the catalyst. Other examples include the alkylation of olefins with benzene for producing ethyl benzene, in which the zeolite catalyst can be regenerated safely only ex situ [16].

The milder reaction conditions chosen for the in situ and ex situ regeneration of the spent catalyst were regeneration temperature = 370 °C, regeneration time = 0.5 h, reaction temperature = 340 °C, and reaction time = 1 h. The yields of diesel oil obtained using in situ and ex situ regenerations of the catalyst were 42.5% and 32.5%, respectively. Therefore, in situ regeneration is favorable for regenerating spent catalysts. This is because in situ regeneration reduced the air contact of the catalyst in this study, thus reducing the possibility of oxidation of the catalyst.

Furthermore, the regeneration time of the catalyst affected the reaction between the catalyst and waste oil. The diesel oil yields at the regeneration times of 0.5 and 1 h were 42.5% and 45.4%, respectively, and not increased for larger than the generation time of 1 h. This result is similar to that reported by Komvokis et al. [17], who claimed that a regeneration time of 1 h is more favorable than 0.5 h. Fig. 3 shows the effect of regeneration temperature on the yield of diesel oil. A high regeneration temperature of the spent catalyst does not always result in a high product yield, because the high temperature of the calcination of the spent catalyst can damage the texture of the catalyst, thus reducing the number of active sites. This leads to a warning that the temperature is high.

3.3. Effect of agitation

The reaction between a solid catalyst and the liquid of waste oil is greatly influenced by agitation, which substantially affects the uniformity of the solution in the vessel (solid, liquid, gas concentration, temperature, and pH) and increases the solid-liquid interfacial mass transfer. To obtain a high reaction yield, an optimal stirring speed is required. Fig. 4 shows that a stirring speed of 200 rpm is optimal for producing diesel oil in a high yield. This

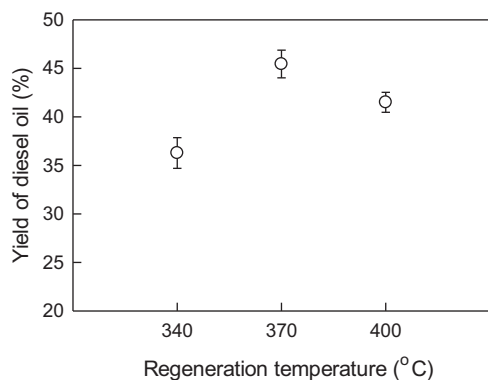


Fig. 3. Effect of regeneration temperature of the spent catalyst on the yield of diesel oil. WM oil = 25 ml, spent catalyst = 12 g, regeneration time = 0.5 h, agitation = 200 rpm, reaction temperature = 370 °C, and reaction time = 1 h.

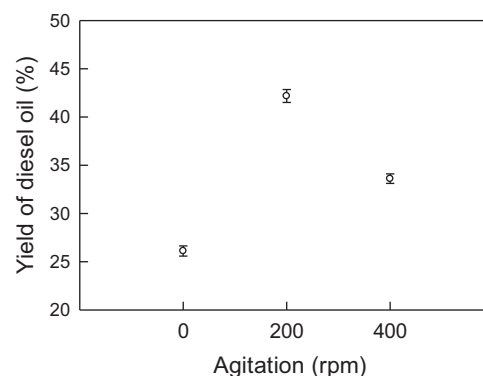


Fig. 4. Effect of agitation on the yield of diesel oil. WM oil = 25 ml, spent catalyst = 12 g, in situ regeneration temperature = 370 °C, regeneration time = 0.5 h, reaction temperature = 300 °C, and reaction time = 1 h.

Table 2

Kinematic viscosities of WBP, WM oil, WME oil, and diesel oil.

	Kinematic viscosity (m ² /s)	
	Before reaction	After reaction ^a
WBP	0.00024 (109)	0.0000595
WM	0.00036 (150)	0.0000598
WME	0.00072 (280)	0.0000601 (20)

The values in the parenthesis are dynamic viscosities (cP).

^a It is diesel oil.

revealed that an increase in agitation can increase the reaction rate. However, a further increase in agitation can reduce the reaction rate because some catalysts can splatter on the reactor wall.

3.4. Effect of the type of waste oil

The order of the kinematic viscosities of various types of waste oils and diesel oils was WME > WM > WBP (Table 2). Fig. 5 shows the effect of the type of waste oil on the yield of diesel oil. The results are WBP > WM > WME. The diesel oil yield was the maximum (60%) when WBP was used as the reactant. This is due to the viscosity of WBP being lower than those of WM and WME oils (Table 2). The kinematic viscosity of the diesel oil was approximately 0.00006 m²/s (60 cSt). The low-boiling gas (P3) obtained for WM and WME oils and WBP was 18%, 10%, and 16%, respectively. Fig. 6 shows the difference in the color of diesel oil produced from various types of waste oils. The WM and WME oils and WBP are homogeneous dark brown oils. The color of product is

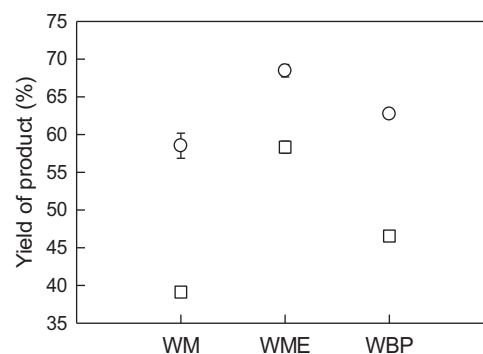


Fig. 5. Effect of the type of waste oil on the yield of diesel oil. Waste oil = 25 ml, spent catalyst = 12 g, in situ regeneration temperature = 370 °C, regeneration time = 0.5 h, agitation = 200 rpm, reaction temperature = 370 °C, and reaction time = 1 h (□): P1 + P2 (diesel oil) and (○): P1 + P2 + P3.

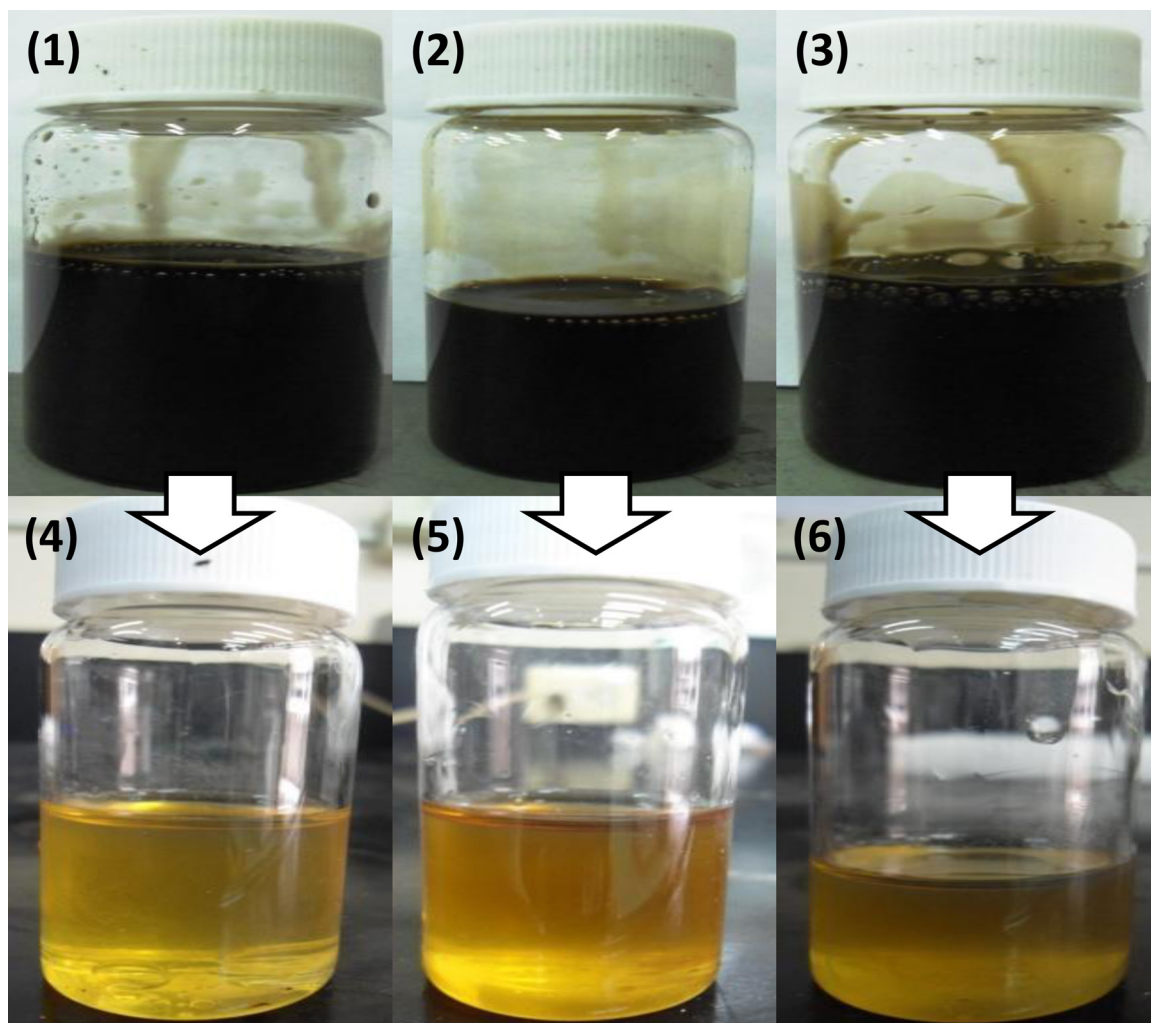


Fig. 6. Color image of the diesel oil produced from various types of waste oils. (1) WBP, (2) WM oil, (3) WME oil, (4) diesel oil produced from WBP, (5) diesel oil produced from WM oil, and (6) diesel oil produced from WME oil.

dark yellowish-gold. Lam et al. [11] reported that the pyrolysis oil obtained at 550 °C was a fairly pale yellowish-gold hydrocarbon liquid containing a small amount of dark solids. It corresponds to our result. The color of the diesel oil produced from the WME oil

was darker than those produced from WM oil and WBP. Fig. 7 shows the lifecycle of a spent catalyst after its regeneration with various types of waste oils. 25 ml of waste oil was introduced into the reactor every hour semicontinuously. After seven cycles, the final volume of the waste oil was 175 ml. The variations of the yield

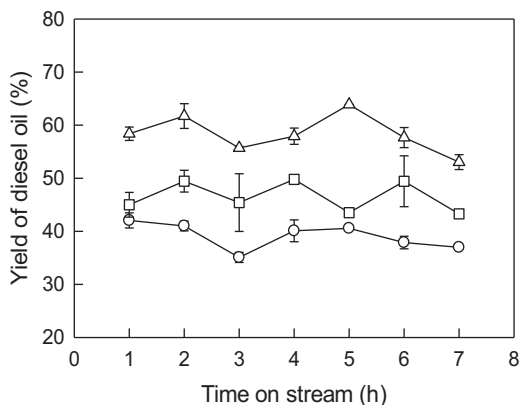


Fig. 7. Effect of the lifecycle of the spent catalyst on the yield of diesel oil produced from various types of waste oils. Waste oil = 175 ml at a rate of 25 ml/h, spent catalyst = 12 g, in situ regeneration temperature = 370 °C, regeneration time = 0.5 h, agitation = 200 rpm, reaction temperature = 370 °C, and reaction time = 7 h (○): WM oil, (□): WME oil, and (Δ): WBP.

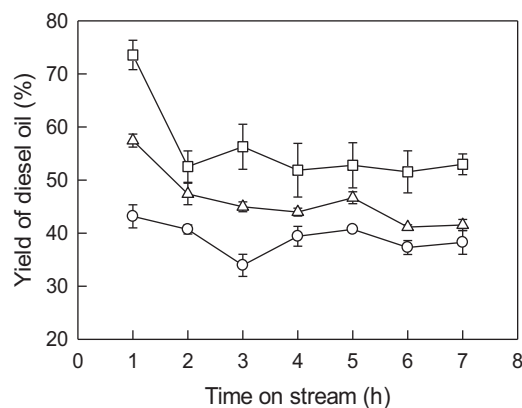


Fig. 8. Effect of the weight of the catalyst on the yield of diesel oil. WM oil = 175 ml at a rate of 25 ml/h, regeneration temperature = 370 °C, regeneration time = 0.5 h, agitation = 200 rpm, reaction temperature = 370 °C, and reaction time = 7 h. Spent catalyst = (○): 12 g, (Δ): 20 g, and (□): 28 g.

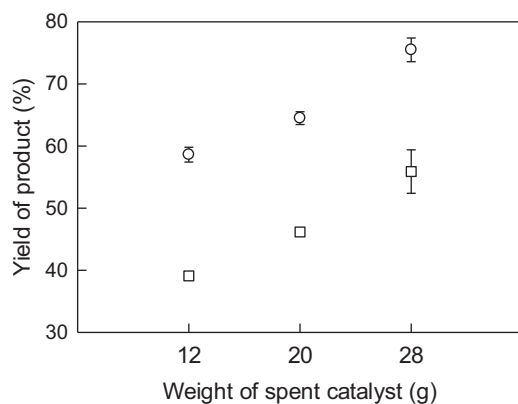


Fig. 9. Plot of the weight of the spent catalyst versus the yield of diesel oil. The reaction conditions are the same as those described in Fig. 8. Data were acquired after a 7 h reaction. (□): P1 + P2 (diesel oil) and (○): P1 + P2 + P3.

of diesel oil for three waste oils were within 10%. Hence, the stability of the spent catalyst for the three waste oils was high. For 7 h of reaction, the yield of the diesel oil produced from WBP was higher than those produced from WM and WME oils. The amount of waste oil was treated more than 11.7 g/g catalyst (140 g of waste oil/12 g spent catalyst used). However, the color of diesel oil is gradually dark with increasing the number of reaction cycle.

3.5. Effect of the weight of the spent catalyst

Fig. 8 shows the effect of the weight of the spent catalyst on the yield of diesel oil. The increase in the weight of the spent catalyst increased the diesel oil yield. This result corresponds to that of Meng et al. [18], who, reported that increasing the weight ratio of the spent catalyst to oil results in a higher reaction degree of pyrolysis. For the first run, when 28, 20, and 12 g of the spent catalyst were used, the diesel oil yields were 73%, 57%, and 43%, respectively. If P3 for 28 g (weight ratio of the spent catalyst to waste oil = 1.4) was considered, the diesel oil yield was higher than 90%. The stability of the spent catalyst was still high after seven cycles. The products (P1 + P2 + P3) for all cases (various weights of the spent catalyst) were more than 60%. Fig. 9 shows that the diesel oil yield was proportional to the weight of the catalyst. The diesel oil yield was calculated using the slope of the linear curve; 0.4 g of diesel oil/h g of spent catalyst was produced at 370 °C, and 0.32 g of diesel oil/h g of spent catalyst was produced at 300 °C. The apparent activation energy calculated using the Arrhenius equation was 4.91 kcal/mol. The yields of diesel oil produced at 300 and 370 °C in the absence of the spent catalyst were approximately 0% and 15%, respectively.

4. Conclusions

The spent catalyst was effectively used to treat the waste oil. The spent catalyst was more stable at low temperatures (370 °C). The diesel oil yield was higher than 90% when a high weight ratio of

catalyst to waste oil was used. According to the results in Table 1, this method is suitable for industrial applications.

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