



## 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 <sub>2</sub> CO <sub>3</sub>	10 <sup>a</sup>	360	–	Pyrolytic distillation	60	[2]
2	Waste engine oil	–	Zeolite	10 <sup>a</sup>	360	–	Pyrolytic distillation	60	[2]
3	Waste engine oil	–	Lime (CaO)	10 <sup>a</sup>	360	–	Pyrolytic distillation	60	[2]
4	Waste automotive engine oil	0.4–5 <sup>e</sup>	carbon	1 <sup>b</sup>	550	2	Continue microwave pyrolysis (5 kW)	88	[6]
5	Waste machinery oil	3.75	HZSM5	0.5 <sup>a</sup>	400	–	Batch thermal pyrolysis	65	[7]
6	Vegetable oil	2 <sup>d</sup>	HZSM5	2 <sup>c</sup>	400	–	Bench home-made 5 L stainless steel	60	[8]
7	Malaysian refuse derived oil	–	Zeolite	20 <sup>a</sup>	450	4	Batch thermal pyrolysis	17.8	[12]
8	Waste automotive engine oil	0.4–5 <sup>e</sup>	Carbon	1 <sup>d</sup>	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.

<sup>a</sup> wt%.

<sup>b</sup> kg.

<sup>c</sup> cm.

<sup>d</sup> liter.

<sup>e</sup> kg/h.

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