

## RECOVERY OF WASTE LUBRICATING OIL BY EXTRACTION USING *n*-BUTANOL AND DEA

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### ABSTRACT

Waste lubricating oil (WLO) contains highly hazardous components that require proper management from storage to disposal. Toxic compounds in discharged lubricating oil can pose huge environmental and human health risks. This study provided a relevant method for recovering fresh base oil from waste lubricating oil. This regeneration process was carried out by using *n*-butanol and DEA (flocculant agent) to extract the base oil, and the solvent layer containing the base oil was isolated. The solvent was recovered by vacuum rotary evaporation, and the base oil fraction obtained reached 85.27% compared to the original waste oil. The regenerated oil was transparent, yellow in color, and had a flash point of 238 °C, indicating high potential for re-blending into commercial lubricants. This study would contribute to a feasible method for recycling waste lubricants, reusing solvents, minimizing environmental impacts, and being reusable after regeneration.

**Keywords:** Waste lubricating oil (WLO), *n*-butanol, DEA (diethanolamine), regenerated oil, quality.

### 1. INTRODUCTION

Lubricating oil has vital functions that assist in maintaining the stability and operation of the engine, such as cooling the internal combustion engine, preventing rust and metal corrosion, lubricating the engine, and sealing holes, etc [1-3]. Lubricant comprises base oil (a liquid mixture of paraffin, naphthene, asphaltene, and aromatics with high molecular weight hydrocarbons), and additives [4, 5]. The boiling point is between 300 °C and 565 °C, including hydrocarbons from C18 to C40 [6, 7]. Vehicles using internal combustion engines require lubricants to ensure the smooth operation of machine parts. In many developing countries, transportation has not met the demand for large motor vehicles, resulting in motorbikes remaining the popular means of transportation. Almost all used lubricants will be discarded and discharged directly into the environment without proper treatment, leading to negative impacts on the ecosystem [8, 9]. Therefore, the demand for recycling lubricants is an inevitable factor contributing to increasing profits and improving environmental quality [8, 10].

To address the impact of waste lubricants on the environment, various studies on waste lubricant recycling have been published such as recycling waste lubricants by chemical and adsorption process using Butanol and Kaolin [11], recycling of waste engine oils using different acids as washing agents [12], recycling of waste lubricating oil by vacuum distillation

[8], re-refining of waste engine oil using membrane: hydrophobic [12, 13] and ultrafiltration [14], pyrolysis of waste oil by a spent catalyst [15], etc. The most common method today is to treat lubricating oil with acid and use vacuum distillation [16], and high commercial potential and is easy to apply in practice [17]. The acid/clay process uses sulfuric acid, and clay is added to remove certain impurities. The residue in the process of separating lubricants has the main component of acidic sludge, which is a hazardous waste material according to the USEPA list [18]. Another widely used method in industrial sectors is vacuum distillation. Although the process is an environmentally friendly process with good quality lubricating oil, it requires high capital investment and sophisticated equipment, leading to the operator's need for high skills [19]. The solvent extraction is a predominant method for enhancing lubricating oil properties that are reported as easy to implement, efficient, and high yield [20-24]. In this work, we carried out the regeneration of waste engine oil by using a *n*-butanol-solvent mixture combined with DEA (flocculant agent) to extract the base oil and then recover the solvent by vacuum evaporation. This is an environmentally friendly solution and very effective in regeneration lubricating oil.

## 2. EXPERIMENTAL METHOD

### 2.1. Materials

This waste lubricating oil investigation is SUZUKI ECSTAR R7000 10W40 on a Honda Suzuki 150i scooter that was run approximately 1200 to 1500 kilometers. Table 1 provides the physicochemical properties of engine oil, provided by the manufacturer.

Table 1. Properties of engine oil SUZUKI ECSTAR R7000 10W40

Property	Unit	Results	Test Method
Flash point temperature by opening the cup	°C	≥ 180	ASTM D92
Dynamic temperature at 100 °C	cSt	12.5 ÷ 16.3	ASTM D445

### 2.2. Experimental procedure

After being recovered from used vehicles, the oil was preheated at 200 °C - 250 °C for 2 hours to remove water and light hydrocarbons in the oil. The preheated oil was cooled to room temperature and was called WLO. Following the appropriate conditions, the experiment was initially utilized with a mixture of 30g WLO, 150 mL *n*-butanol, 60 µL DEA in beaker (mass/volume ratio WLO/*n*-butanol = 1:5; volume/mass ratio DEA/WLO = 1:500). The mixture was heated and stirred at 60 °C for 1 hours under air atmosphere. Then the resulting mixture was cooled down to ambient temperature and settled for 24 hours to separate into 2 layers. The residue layer was discharged. The above layer was evaporated by rotary evaporation for solvent recovery, and the remaining oil was adsorbed with activated bentonite to obtain regenerated oil.

## 3. RESULTS AND DISCUSSION

### 3.1. Effect of flocculant agents

Organic flocculants have a mechanism involving complexation, forming chemical bonds or physical bonds that increase molecular size, facilitating their sedimentation. In this work, the organic flocculants (DEA, MEA, TEOA) are used due to having functional groups -NH<sub>2</sub> and -OH that can form hydrogen bonds and interact with negatively charged particles in waste

lubricating oil. Moreover, these flocculants reduce the content of dispersants and detergents. The mechanism of action of organic flocculants is illustrated in Fig.1.

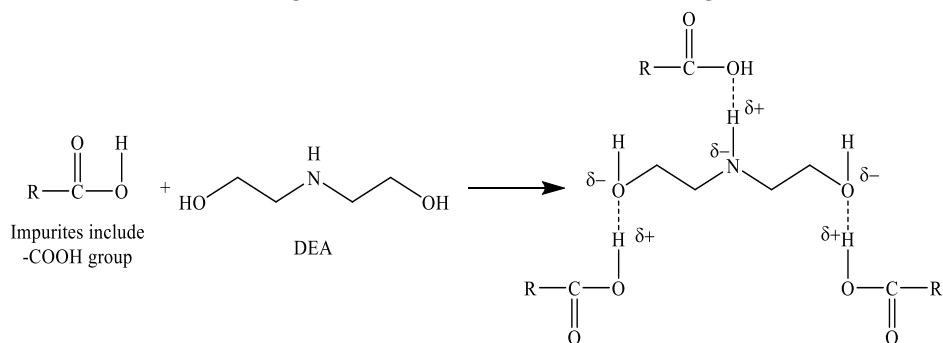


Figure 1. The proposal mechanism of action of organic flocculants in waste oil

When a specific amount of DEA was added to waste lubricating oil, DEA would neutralize the charges of dispersants and detergents to release the suspended flocculant particles that are retained. In suspended organic particles containing groups -OH or -COOH, it would be retained by -COOH by hydrogen bonds, forming larger colloidal particles that were capable of settling. Furthermore, the flocculant was also an intermediate that created a bridge between contaminants, like polymer flocculants. If the suspended particles are metal ions, organic compounds can form complexes with those metal ions, forming large flocs that settle [23].

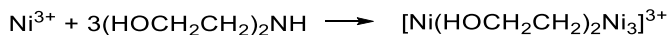


Figure 2. The combination of metal ions and a flocculant agent.

To find out the relevant conditions when flocculant agents appear, we surveyed flocculant agents such as TEOA, DEA, MEA, NaOH,  $\text{Al}_2(\text{SO}_4)_3$ , and  $\text{FeCl}_3$ . The results are as follows in Table 2. As a result, DEA had the highest yield efficiency (85,27%) with a low required dosage. Moreover, the colour of regeneration oil from using DEA and MEA was compared to visual sensory, which showed that regeneration oil from using DEA has a better colour (yellow colour) than regeneration oil from using MEA (red-yellow colour). Other inorganic flocculant agents, NaOH,  $\text{Al}_2(\text{SO}_4)_3$ , and  $\text{FeCl}_3$ , were also included, but the yield was not better than organic flocculant agents, and recovered oils were black. Therefore, DEA was selected as the preferred flocculant because of its consistent performance and effective separation abilities.

Table 2. The influence of flocculant type on recovery efficiency<sup>a</sup>

Entry	Flocculant agent	Flocculant/WLO (g/mL)	Yield <sup>b</sup> (%)
1	NA	1:500	65.17 <sup>d</sup>
2	TEOA	1:500	76.03 <sup>c</sup>
3	DEA	1:500	85.27 <sup>c</sup>
4	MEA	1:500	85 <sup>c</sup>
5	NaOH	1:500	59 <sup>c</sup>
6	$\text{Al}_2(\text{SO}_4)_3$	1:500	65.77 <sup>d</sup>
7	$\text{FeCl}_3$	1:500	63.86 <sup>d</sup>

<sup>a</sup>Reaction conditions: Preheated WLO (30 g), solvent (*n*-butanol 150 mL).

Abbreviation: MEA = monoethanolamine; TEOA = triethanolamine. <sup>b</sup>Isolated yield.

<sup>c</sup>Colour: orange-yellow. <sup>d</sup>Colour: black.

### 3.2. Effect of solvent

The solvent is the key factor that directly determines the quality of regenerated oil. The solvent ought to be closely polar with lubricating oil, miscible with oil, reject impurities, environment-friendly, low-cost, and easy to recover [25]. In this work, we surveyed common solvents: *n*-hexane, *n*-butanol, butanone (MEK), and ethanol with different ratios. The survey results are presented in Table 3.

Table 3. Recovery yield of lubricating oil with various solvents

Entry	Solvent	Oil/solvent ratio (g/mL)	Recovery yield (%)
1	<i>n</i> -butanol	1:5	85.27
2	<i>n</i> -hexane : <i>n</i> -butanol (2:3)	1:5	13.84
5	ethanol : <i>n</i> -butanol (2:3)	1:5	20.81
6	ethanol : <i>n</i> -butanol (3:2)	1:5	9.47
7	ethanol : <i>n</i> -butanol (4:1)	1:5	19.79
8	MEK	1:5	76.64
9	<i>n</i> -butanol: MEK (2:3)	1:5	80.76
10	<i>n</i> -butanol: MEK (3:2)	1:5	38.57
11	<i>n</i> -butanol: MEK (4:1)	1:5	60

The survey results showed that *n*-butanol solvent gave the highest yield efficiency. These results were consistent with previous studies on using solvents for the recovery of waste lubricating oil [25].

### 3.3. Effect of DEA concentration

These experiments were performed in ideal conditions, oil/*n*-butanol (g/mL) was 1:5. The DEA contents were investigated in g/mL ratio with WLO as 1:50, 1:100, 1:200, 1:300, 1:400; 1:500, 1:600, 1:700, and 1:800. The regeneration yields are illustrated in Table 4.

Table 4. Effects of DEA concentrations on recovery yield

Entry	Solvent	DEA/WLO ratio (g/mL)	Recovery yield (%)
1	<i>n</i> -butanol	1:50	34.11
2	<i>n</i> -butanol	1:100	39.91
3	<i>n</i> -butanol	1:200	50.63
4	<i>n</i> -butanol	1:300	60.27
5	<i>n</i> -butanol	1:400	70.67
6	<i>n</i> -butanol	1:500	85.27
7	<i>n</i> -butanol	1:600	82.29
8	<i>n</i> -butanol	1:800	72.59

As shown in Table 4, DEA only needed a small amount, with a DEA/WLO ratio of 1:500 being the most suitable. At this time, the recovery efficiency reached 85.27%. When the concentration of DEA increased, a suspension mixture was formed, making it difficult to

separate into two layers, reducing efficiency. Therefore, under this condition, the DEA/WLO ratio was chosen as 1:500 to investigate the following conditions.

### 3.4. Effect of solvent/WLO (volume/mass ratio)

These experiments were performed in ideal conditions DEA/WLO ratio of 1:500 (g/mL). The solvent (*n*-butanol)/WLO ratio was investigated in mL/g ratio, which were 3:1, 4:1, 5:1, 6:1, 8:1, and 10:1. The yield of regenerated oils is represented in Table 5.

Table 5. Effects of solvent ratio on the regenerated oil quality

Entry	Solvent	Solvent/WLO ratio (mL/g)	Recovery yield (%)
1	<i>n</i> -butanol	3:1	20.06
2	<i>n</i> -butanol	4:1	28.94
3	<i>n</i> -butanol	5:1	85.27
4	<i>n</i> -butanol	6:1	74.44
5	<i>n</i> -butanol	8:1	60.07
6	<i>n</i> -butanol	10:1	47.87

Based on Table 5, the suitable *n*-butanol/WLO ratio for this process was 5:1, the regenerated oil was yellow, and the recovery yield had reached 85.27%. The solvent ratio greatly affects the recovery efficiency. Fixing the DEA/WLO ratio as 1:500, changing the *n*-butanol/WLO ratio gradually from 3:1 to 1:10, the results showed that the ratio of 5:1 was the best (efficiency 85.27%); the ratios around this ratio all gave lower efficiencies. From this, it could be concluded that the relevant *n*-butanol/WLO ratio under this condition was 5:1.

### 3.5. Quality of regenerated oil

Using the above relevant conditions, we proceeded to regenerate the oil according to that process and made a preliminary assessment of the quality of the regenerated oil according to the standards for evaluating the quality of lubricating oils. The results are shown in Fig. 3.

In terms of color, the regenerated oil was yellow, transparent, and similar to many types of engine oil on the market.

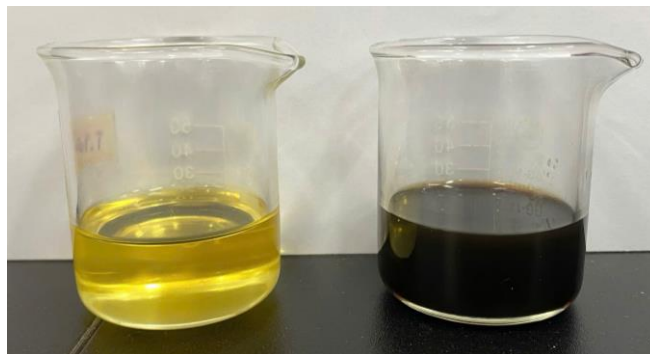


Figure 3. Comparison of the appearance of regenerated oil and waste lubricating oil

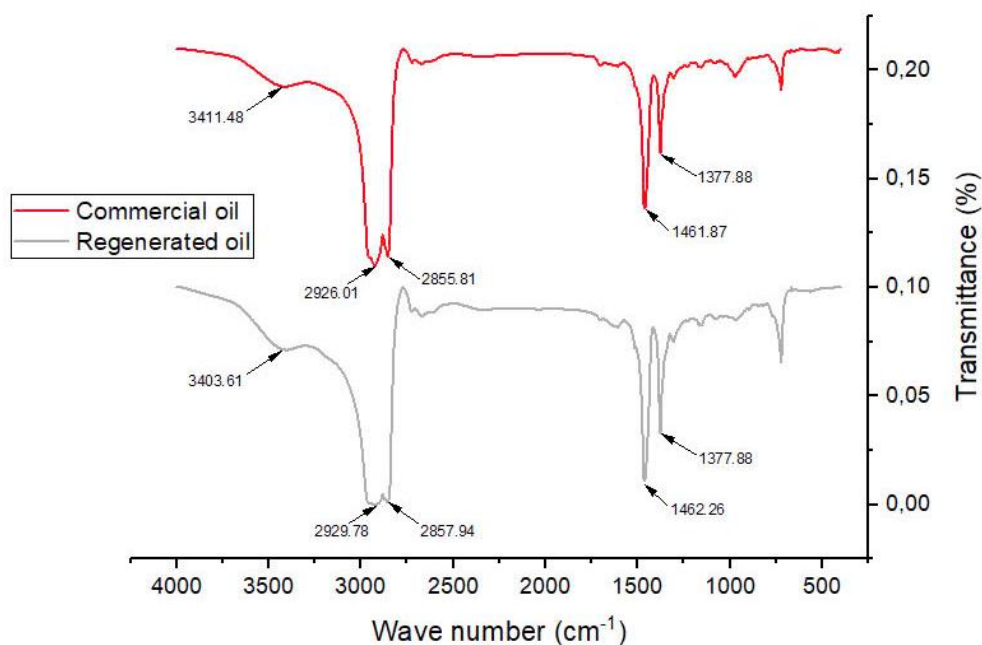


Figure 4. FTIR spectrum comparison between commercial and regenerated oil

In order to compare with commercial oil, we proceed to evaluate the quality of regenerated oil by the FTIR method. The results are shown in Figure 4 and Table 6.

Table 6. Functional groups from the FTIR spectra of commercial oil and regenerated oil

Entry	Frequency (cm <sup>-1</sup> )		Bond and functional group
	Commercial oil	Regenerated oil	
1	3411.48	3403.61	Water
2	2954.79	2929.78	C-H of alkane
3	2926.01	2857.94	
4	2855.81	2724.65	
5	1461.87	1462.26	
6	1377.88	1378.19	C-H of CH <sub>3</sub>

Regarding to metal ingredient factor, the heavy metals content of the recycled oil sample was determined by ashing the sample at 750 °C and dissolved with HNO<sub>3</sub> 1%, measured by ICP-AES spectroscopy, the results were illustrated in Table 7.

According to the above results, metals (Cr, Pb, Hg, Cd) had low concentrations (less than 1 ppb), and other environmentally hazardous metals such as As, Se, Sb also had very low concentrations <1 ppb). These were metals that needed to be controlled during the production of lubricants to ensure environmental safety [26].

Table 7. Metal content in regenerated oil

Element	Metal Concentration (ppm)	Metal Content (%)
B	0.00253	0.00503
Na	1.098	2.18277
Al	0.039	0.07753
Cr	0.00052	0.00103
Mn	0.00284	0.00565
As	0.00018	0.00036
Se	0.00008	0.00016
Cd	0.00001	0.00002
Sb	0.000005	0.00001
Ba	0.00263	0.00523
Hg	0.00004	0.00008
Pb	0.000207	0.00041

In addition to the above evaluation criterias, to evaluate the reusability of regenerated oil, we further evaluated the following factors: flash point temperature by opening the cup, kinematic viscosity at 100 °C, carbon residue content, sulfated ash content, pour point, copper strip corrosion at 100 °C, copper strip corrosion at 150 °C, and density [27]. These factors were analyzed based on the technical standards applied to current commercial lubricants. The results are shown in Table 8.

Table 8. Physicochemical properties of regenerated lubricating oil

Property	Unit	Test Method	Commercial lubricant requirement	Result
Flash points by cleveland open cup tester	°C	ASTM D92 - 18	180	238
Kinematic viscosity at 100 °C	cSt	ASTM D445	9.3 – 12,5	20.4
Pour point	°C	ASTM-5853 – 17a	-	< -250C
Copper Strip Corrosion at 100 °C	-	ASTM-D130-19	1a – light tarnish	1a – light tarnish
Copper Strip Corrosion at 150 °C	-	ASTM-D130-19	1a – light tarnish	1a – light tarnish
Sulfated ash content	% weight	ASTM D874-06	0.18	0.15
Density	kg/m <sup>3</sup>	ASTM D5002-22	800 - 950	858.6

In the above factors, most of them meet the standards of commercial oil, except for the kinematic viscosity of regenerated oil, which was higher than commercial oil. This was explained by the fact that during use, the oil was oxidized when operating at high temperatures.

#### 4. CONCLUSIONS

In summary, we developed a method for regenerating waste lubricating oil by investigating factors such as flocculant agents, flocculant/WLO ratios, solvents, and solvent/WLO ratios; the quality of the regenerated oil was preliminarily evaluated by commercial oil quality evaluation standards. Changing the flocculant agents, flocculant/WLO ratios, solvents, and solvent/WLO ratios significantly affected the recovery efficiency of the regenerated oil. The relevant conditions for recovering waste lubricating oil by the extraction method with *n*-butanol were *n*-butanol/WLO (volume/mass ratio) = 1:5, DEA/WLO (volume/mass ratio) = 1:500. The regenerated lubricating oil has achieved the criteria corresponding to the quality of commercial oil. Importantly, the recovery efficiency achieved 85.27%, and the quality of the regenerated oil was within the allowable limits, except for the dynamic viscosity exceeding the standard of commercial engine oil. This extraction procedure is simple to perform, feasible for industrial-scale regeneration.

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## TÓM TẮT

### THU HỒI DẦU NHỚT THẢI BẰNG PHƯƠNG PHÁP CHIẾT XUẤT SỬ DỤNG n-BUTANOL VÀ DEA

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Dầu nhớt thải (WLO) chứa thành phần gây ô nhiễm môi trường, cần được quản lý đúng cách từ khâu lưu trữ đến khâu thải bỏ. Các hợp chất trong dầu nhớt thải ra có thể gây ra những rủi ro rất lớn cho môi trường và sức khỏe con người. Trong nghiên cứu này, chúng tôi cung cấp một phương pháp phù hợp để thu hồi dầu gốc từ dầu nhớt thải. Quá trình tái sinh này được thực hiện bằng cách sử dụng n-butanol và DEA (chất keo tụ) để chiết xuất dầu gốc. Dung môi chiết xuất được thu hồi bằng phương pháp cô quay chân không, hiệu suất thu hồi dầu gốc đạt 85,27% so với dầu thải ban đầu. Dầu thu hồi trong suốt, có màu vàng và có điểm chớp cháy là 238 °C, cho thấy tiềm năng cao để pha trộn thành dầu bôi trơn thương mại. Nghiên cứu này sẽ là một phương pháp khả thi để tái sinh dầu nhớt thải, tái sử dụng dung môi, giảm thiểu tác động đến môi trường.

*Từ khóa:* Dầu bôi trơn thải (WLO), n-butanol, DEA (diethanolamine), dầu tái sinh, chất lượng dầu thu hồi.