Recovery of Waste Engine Oil Using Vacuum Distillation: Effect of Solvent Pre-Treatment

Hendriyana a,1,*

- ^a Department of Chemical Engineering, Faculty of Engineering, Universitas Jenderal Achmad Yani, Cimahi, Indonesia
- 1 hendriyana@lecture.unjani.ac.id *
- * corresponding author

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ABSTRACT

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The purification process of used engine oil, which involves vacuum distillation preceded by pretreatment using chemical solvents such as acetic acid and sodium hydroxide (NaOH), has been studied to improve the quality and efficiency of base oil recovery. Pretreatment was performed using varying solvent concentrations of 5%, 10%, and 15% (v/v) and incubation times of 24, 48, and 96 hours. The primary objective of pretreatment is to decompose polar contaminants and break down complex compounds, making them easier to separate during the distillation stage. The vacuum distillation process was carried out at a pressure of -70 cmHg and a temperature of 230°C, allowing separation of fractions based on differences in boiling points. Experimental results showed that the average density of the base oil distilled from motor oil was 833 kg/m³ and from car oil was 840 kg/m³, approaching the characteristics of new oil. Optimum conditions were achieved by pretreating with 15% v/v acetic acid for 48 hours, resulting in the highest base oil yield of 45.0%. However, NaOH solvent produced more precise visual results, indicating a better ability to bind polar impurities. Chemical component analysis revealed that the main compounds of the new oil, such as octanoic acid, 1,2,3-propanetriol, and glyceryl tridecanoate, were not present in the distillate because they underwent thermal degradation to alkanes and cyclic compounds. On the other additives such as pentatriacontane, hexacosane, tetrapentacontane-1,54-dibromo were still detected, proving their thermal stability.

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1. Introduction

Waste engine oil is categorized as a hazardous and toxic material. Due to the high level of dangerous contaminants in waste engine oil, it can cause environmental impacts that are harmful to the ecosystem. For human health, it can cause respiratory disorders, nerve damage, and increase the risk of cancer [1-4]. Contaminants that can be found in waste engine oil consist of polycyclic aromatic hydrocarbons originating from imperfect combustion, polychlorination originating from lubricant additive compounds, heavy metals originating from the wear process of metal components in the engine, and water originating from combustion and intake air [5],[6]. With the proper method, contaminants can be removed from waste engine oil to produce lube base oil. Thus, the cost of making new lube oil can be reduced. In addition, this purification will have a significant positive impact on the environment [7-9].

Various processing and recovery methods have been utilized in the recycling process of waste engine oil. In the acid and clay-based recycling method, asphaltene compounds are removed by using high-concentration sulfuric acid. The use of this method has been discontinued because it produces tar acid waste, which is toxic and heavily polluted, and is difficult to manage [10-13].





The non-acid clay-based recycling process begins with the pretreatment of waste engine oil using a polymer-based material to remove carbon residue. It is further processed through vacuum distillation and adsorption using clay media. While effective, this process produces a product with high metal content and requires significant amounts of clay to achieve the desired color clarity [14].

Another method used to recycle waste engine oil is through a solvent extraction process. Various types of solvents have been used in the solvent extraction process to recycle used lubricating oil, including 2-propanol, 1-butanol, and methyl ethyl ketone (MEK). Of the three solvents, MEK has proven to be the most effective, exhibiting the best performance in terms of oil recovery efficiency. MEK produces the lowest percentage of oil loss during the extraction process, indicating its high solubility and better compatibility with the base oil components [15],[16]. The use of liquefied petroleum gas (LPG) as a solvent has been studied by [17]. Although it has shown effectiveness in the extraction process, reliance on LPG presents challenges due to its volatile and flammable characteristics, requiring handling and storage that meet high safety standards. Furthermore, the availability and relatively high cost of LPG increase the complexity and economic burden of this process. Riyanto carried out the process of extracting base oil from used lubricating oil using polar solvents combined with the addition of potassium hydroxide [18],[19].

Nabil et al. utilized various adsorbent materials, including date palm kernel powder, activated bentonite, and eggshell powder, to recover waste engine oil through an adsorption method. These materials proved effective in significantly reducing asphaltene and heavy metal contaminant levels [20]. Propane, butane, ethane, and carbon dioxide, under supercritical conditions, have been utilized in waste engine oil processing and have been proven to support environmentally friendly and sustainable processing practices [21-23].

Membrane technology is also used in the reprocessing of waste engine oil. This technique utilizes a hollow polymer membrane to separate impurities and metal particles from used oil. This filtration process is usually carried out continuously. As a result, the physical characteristics of the regenerated oil, including clarity, viscosity, and flash point, are improved. However, the main challenge with this technology is the potential for membrane clogging and damage from large particles, given the membrane's high cost and high maintenance costs [24],[25].

Among the various methods proposed, each exhibits significant limitations. The acid-clay method produces toxic waste that is difficult to manage sustainably. The adsorption method requires large amounts of material, making it an economically inefficient process. The hydrocarbon-based solvent extraction method poses significant risks due to its flammability and potential environmental hazards. Meanwhile, membrane technology, while effective, is relatively expensive and highly susceptible to fouling, which degrades its performance.

Vacuum distillation has been identified as a promising method for the recycling of used engine oil [26]. This method works effectively in removing most contaminants from waste engine oil. The process begins with atmospheric distillation to separate light fractions such as water and volatile hydrocarbons, followed by distillation under reduced pressure at a temperature of around 300°C. The processing results show that the resulting base oil has high quality and its characteristics are comparable to the standard base oil specifications. The method is very efficient because it produces high yields. This confirms that vacuum distillation is a feasible and sustainable technology for converting used lubricating oil into high-value products, while supporting the principles of a circular economy and environmental conservation.

This research utilizes a combination of solvent-based pretreatment and vacuum distillation methods in the recycling of waste engine oil. While solvent extraction is well-established, the optimal combination of solvent type, concentration, and incubation time for pre-treatment before vacuum distillation remains unclear, particularly for common solvents such as acetic acid and NaOH.

2. Research Methodology

2.1. Materials

In this study, experiments were conducted on two types of waste engine oil samples obtained from different sources: motorcycle and car oil change service stations. The average motorcycle oil used was 10W-40, and the car oil was 15W-40. The lubricating oil used for comparison is sourced from Shell

for cars and motorcycles from AHM. The chemical used consists of glacial acetic acid with a concentration of 98% wt and sodium hydroxide with a concentration of 98% wt. These chemicals were obtained from an online chemical store.

2.2. Procedures

A total of 100 mL of waste engine oil was placed into a 250 mL beaker. Then, a solvent was added with a ratio of solvent to waste engine oil of 5%, 10%, and 15% (v/v). The solution was stirred at 150 rpm following [27] for 30 minutes and then incubated for 24, 48, and 96 hours in a closed condition using aluminum foil. After the incubation process, the waste oil was centrifuged for 10 minutes at 1000 rpm, following the method described in [28], to separate the sample from the sludge.

The schematic diagram of the laboratory-scale vacuum distillation is presented in Fig. 1. Key components include: (1) Electrical heater, (2) Distillation tank made of stainless steel with an inner diameter of 3.0 cm and a height of 16 cm, (3) Doble pipe condenser, (4) Oil/product trap, (5) Pressure gage, (6) Vacuum pump, (7) Chiller, (8) Tempea Temperature control panel. A 20 mL sample of waste engine lubricating oil that has been centrifuged (free of sludge) is placed in the vacuum distillation unit tank and then sealed tightly. The unit is placed inside a cylindrical electric furnace, where the outlet pipe of the vacuum distillation unit is connected to a condenser, oil trap, and vacuum pump. Oil traps are used to collect condensed base oil. Batch mode operation begins by turning on the vacuum pump to reduce the pressure inside the distillation unit to -70 cmHg. The selection of vacuum conditions in the distillation process is due to the physicochemical characteristics of heavy hydrocarbon fractions contained in oil, which have a high boiling point at atmospheric pressure, exceeding 400 °C. This can cause thermal cracking, oxidation, and degradation of base oil quality. Vacuum distillation can significantly lower the boiling point of oil fractions, allowing separation to take place at lower temperatures (230-240°C), thereby minimizing thermal decomposition and preserving the base oil compounds. Saleem and Karim also performed vacuum distillation of used oil at a pressure of approximately -70 cmHg [29]. After that, the operating temperature was set at 230°C and continued for 150 minutes.

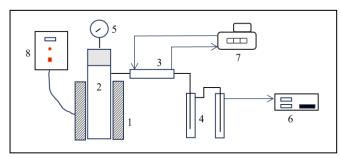


Fig. 1. The schematic diagram of the vacuum distillation equipment: 1. Electrical heater, 2. Distillation tank, 3. Condenser, 4. Trap, 5. Pressure gauge, 6. Vacuum pump, 7. Chiller, 8. Control panel

3. Results and Discussion

3.1. Density

The base oil obtained from the vacuum distillation process is then subjected to physical analysis. The physical property analyzed is density, which is obtained by dividing the mass of a substance by its volume. It is essential to know the temperature at which the density measurement is performed, as density can change with temperature.

Density = Mass of oil/Volume of oil
$$(1)$$

Fig. 2 shows a comparison between the density of fresh engine oil and base oil obtained from the research results. The density changed, with the base oil having a lower density than the fresh engine oil. The density of new motorcycle oil is approximately 860 kg/m³, and new car oil is 904 kg/m³ [30,31], while the base oil density is 833 kg/m³ for motor and 840 kg/m³ for car. This is in line with [26] and [32], which show that vacuum distillation can produce lubricating oil fractions with a lower density than fresh engine oil. Similarly, Saleem and Karim [29] demonstrate that vacuum distillation reduces density due to the predominance of lighter paraffinic fractions over aromatic fractions. The consistency of these findings confirms that the decrease in density in vacuum-distilled base oil is a

direct consequence of the shift in the composition of long-chain molecules towards shorter chains with lower molecular masses. This is evident in the GC-MS analysis results presented in Tables 3-6.

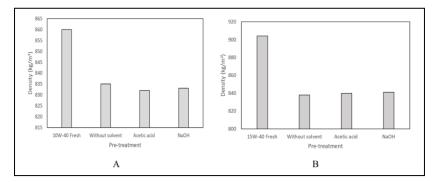


Fig. 2. Density of fresh oil and base oil with solvent concentration. 15% and incubation 96 h
A. motorcycle and B. car

From Fig. 2, it can also be seen that the density of the base oil obtained with the pre-treatment without solvent is not much different from that obtained using a solvent. The density of the base oil obtained is still below the API Group N150I base oil standard of 872.5 kg/m³ [33]. Base oils that do not meet these standards cannot be used as base materials for lubricant formulations due to their lack of protective properties and short service life [31][,34].

3.2. Yield

In the first experiment, waste engine oil was directly vacuum distilled without any solvent pretreatment. The motorcycle and car base oil yields were 10% v/v and 17.5% v/v, respectively. The very low yields were due to the high asphaltene content in the waste engine oil. Fig. 3 shows the appearance of base oil obtained without solvent pretreatment: (A) from motorcycle oil, (B) from car oil. The reddish-orange color indicates a high content of oxidized compounds and impurities, which is in line with the research results [26].

In the second experiment, waste engine oil was pretreated before vacuum distillation. This treatment was intended to reduce asphaltene content, thus increasing base oil yield. The results of the pretreatment using acetic acid solvent are presented in Table 1. The results showed that the higher the acetic acid concentration, the higher the base oil yield. This is due to the increasing amount of asphaltene precipitated after undergoing the pretreatment and centrifugation process. This is in line with research by [7], which shows that the use of glacial acetic acid as a solvent can separate asphaltenes and heavy metals through a precipitation and centrifugation process, producing a cleaner and clearer base oil fraction with physico-chemical qualities similar to those of new oil. Fig. 4 shows an example of precipitate asphaltene: (A) after pretreatment with solvent and centrifugation, (B) the asphaltene after drying. Incubation time also has a significant effect on yield. A longer incubation time can increase the yield of base oil. However, at an incubation time of 96 hours, the base oil yield decreased dramatically. This could be due to the polymerization reaction of aromatic compounds with acetic acid [29].

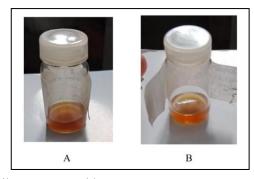


Fig. 3. Base oil appearance without pre-treatment: A. motorcycle and B. car

Table 1. Pre-treatment using acetic acid solvent

Waste engine oil from	Time of incubation (h)	Solvent Concentration (%-v/v)	Yield (%-v/v)
	24	5	20.0
	24	10	36.0
	24	15	41.5
Motorcycle	48	5	20.5
	48	10	37.5
	48	15	42.5
	96	15	20.0
	24	5	28.5
	24	10	40.0
	24	15	45.0
Car	48	5	30.0
	48	10	41.0
	48	15	45.0
	96	15	32.5

In the third experiment, waste engine oil was pretreated with a NaOH solution. The solvent concentration used in this experiment was 15% (v/v) with an incubation time of 96 hours. The results are presented in Table 2. The base oil obtained for each motorcycle and car was 25% v/v and 42.5% v/v, respectively. This yield is higher than that obtained when using an acetic acid solvent at the same concentration and incubation time. This is because NaOH is capable of removing chloride compounds, metals, additives, and acidic compounds. These impurities can be bound to asphaltene by NaOH, allowing them to be easily separated from the oil [35].

Table 2. Pre-treatment using NaOH solvent

Waste engine oil from	Time of incubation (h)	Solvent Concentration (%-v/v)	Yield (%-v/v)
Motorcycle	96	15	25.0
Car	96	15	42.5

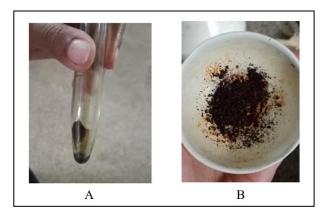


Fig. 4. Appearance of asphaltene residue: A. after centrifugation and B. after drying

Fig. 5 shows the appearance of base oil obtained from the acetic acid pre-treatment process with an incubation time of 48 hours for motorcycle oil (A: 5%, B: 10%, C: 15%) and for car oil (D: 5%, E: 10%, F: 15%). Meanwhile, for the NaOH solution pre-treatment, the incubation time was 96 hours for both motorcycle oil (F) and car oil (G), at a concentration of 15%. Base oil obtained through acid pre-treatment tends to be of lower quality than that obtained through alkaline treatment. In addition, the base oil produced is dark in color and tends to have an unpleasant odor [35]. This is in line with NaOH's ability to remove impurities and ease of separation as described above.

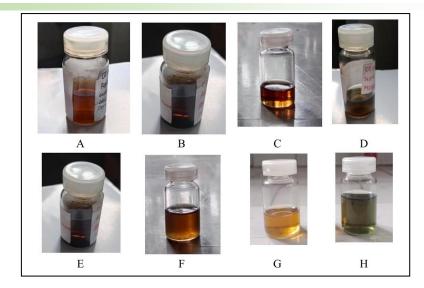


Fig. 5. Base oil appearance of motorcycle with pre-treatment (using acetic acid A.5%, B.10%, C.15% incubation 48h and using NaOH 15% incubation 96h(G)) and car with pre-treatment (using acetic acid D.5%, E.10%, F.15% incubation 48h and using NaOH 15% incubation 96h (H)).

Testing of pH levels revealed that all distillate products, including both motor oil and car oil, had a pH of 6. This indicates that the pH of the distillate is lower than that of new oil, which is 6.5 for both motor oil and car oil. This can be caused by oxidation in the oil during the combustion process, making the used oil more acidic.

3.3. Identification of Compounds in Base Oil

Identification of compounds contained in fresh engine oil and base oil from vacuum distillation using Gas Chromatography–Mass Spectrometry (GC-MS) instrument. Fresh engine oil for a motorcycle with a 10W-40 specification has been analyzed and presented in Table 3. The analysis results show that fresh engine oil consists of heptacosane and titriacontane compounds as the most significant components. In fresh engine oil of a car with specification 15W-40, the most essential element and titriacontane, as presented in Table 4, even the concentration of this component is greater than that in fresh engine oil of a motorcycle. In both fresh engine oils, there is a compound, tetrapentacontan-1,54-dibromo, which functions as an additive to protect metal surfaces from wear under high-pressure conditions. Bromine can react with metal surfaces, forming a protective layer (film boundary) that reduces wear and tear.

•	•	
Compounds	Formula	%-mass
Heptacosane	C ₂₇ H ₅₆	69.79
Pentatriacontane	C35H72	0.14
Hexacosane	$C_{26}H_{54}$	0.22
Tetrapentacontan1,54-dibromo	$C_{54}H_{108}Br_2$	0.04
Tetracontane	$C_{40}H_{82}$	0.09
Titriacontane	C33H68	29.73

Table 3. Fresh Engine Oil for Motorcycles 10W-40

The contents of motorcycle and car base oils produced by vacuum distillation with pretreatment using NaOH solution have been analyzed and presented in Tables 5 and 6. The resulting motorcycle and car base oils contain many long-chain and cyclic alkane compounds. Heptacosane and titriacontane, the main compounds in fresh engine oil, were not found in the base oil. This is most likely due to thermal decomposition that occurs in the engines of motorcycles or cars. Pentatriacontane is found in fresh engine oil and base oil. This compound does not appear to decompose, as pentatriacontane exhibits high oxidation stability. This compound also helps make the lubricant more resistant to oxidation and degradation at high temperatures. Similar to pentatriacontane, hexacosane also does not undergo thermal decomposition.

Table 4. Fresh Engine Oil of Car 15W-40

Compounds	Formula	%-mass
Pentadecane	C ₁₅ H ₃₂	2.17
Heptadecene	$C_{18}H_{36}$	0.65
Tetracontane	$C_{40}H_{82}$	0.36
Tetrapentacontan1,54-dibromo	$C_{54}H_{108}Br_2$	3.28
Titriacontane	$C_{33}H_{68}$	88.41
Tetracosane	$C_{24}H_{50}$	0.17
Pentatriacontane	C35H72	1.41
Hexacosane	$C_{26}H_{54}$	0.46
Docosane	C22H46	1.81
Dotriacontane	$C_{32}H_{66}$	0.83
Tetracontane	$C_{40}H_{84}$	0.43

Hexacosane is a paraffin waxy hydrocarbon that is naturally found in mineral base oils. It plays a role in increasing the saturated hydrocarbon content, thereby improving the oxidation stability and thermal resistance of the oil. It helps increase the kinematic viscosity of oil, especially at room temperature. It also allows the oil to maintain a lubricating film on metal surfaces.

Table 5. Base Oil of Motorcycle with NaOH Solvent

Compounds	Formula	%-mass
Benzene, 1,2,3-trimethyl	C9H12	2.03
Benzene, 1,2,4-trimethyl	C_9H_{12}	0.47
Benzene, 1-methyl-4-(1-methylethyl)	$C_{10}H_{14}$	2.40
Undecane	$C_{11}H_{24}$	1.56
Benzene, 1,2,3,4-tetramethyl	$C_{10}H_{14}$	2.09
Hexadecane	$C_{16}H_{34}$	0.46
5,9,9-Trimethylspiro[3.5]nona-5,7-dien-1-one	$C_{12}H_{16}O$	1.72
Undecane, 3-methyl	$C_{12}H_{26}$	0.57
Dodecane	$C_{12}H_{26}$	3.95
Benzene, 1,3-dimethyl-5-(1-methylethyl)	$C_{11}H_{16}$	0.40
1H-Indene, 2,3-dihydro-4,7-dimethyl	$C_{12}H_{16}$	0.41
Undecane, 2,5-dimethyl	$C_{13}H_{28}$	0.61
1H-Indene, 1-ethyl-2,3-dihydro-1-methyl	$C_{12}H_{16}$	0.93
Dodecane, 2,7,10-trimethyl	$C_{15}H_{32}$	0.61
Tridecane	$C_{13}H_{28}$	2.68
Naphthalene, 2-methyl	$C_{11}H_{10}$	0.59
Tetradecane	$C_{14}H_{30}$	1.14
Pentadecane	$C_{15}H_{32}$	0.49
Nonadecane	$C_{19}H_{40}$	3.66
Pentadecane, 2,6,10,14-tetramethyl	$C_{19}H_{40}$	0.81
Eicosane	$C_{20}H_{42}$	12.14
Heneicosane	$C_{21}H_{44}$	2.34
Tetracontane	$C_{40}H_{82}$	13.58
Pentadecane, 8-hexyl	$C_{21}H_{44}$	1.47
Tetratetracontane	C44H90	2.63
Hexacosane	$C_{26}H_{54}$	13.21
Docosane	$C_{22}H_{46}$	2.09
2-Propenoic acid, 2-methyl-, octyl ester	$C_{12}H_{22}O$	0.40
Docosane, 6-methyl	$C_{23}H_{48}$	0.86
Octadecane, 3-methyl	C ₁₉ H ₄₀	0.67
Tetrapentacontan1,54-dibromo	$C_{54}H_{10}Br_2$	2.57
Heneicosane, 11-(1-ethylpropyl)	$C_{26}H_{54}$	1.59
Hexadecane, 2,6,10,14-tetramethyl	$C_{20}H_{42}$	0.75
Pentadecane, 8-hepttyl	$C_{21}H_{44}$	5,34
Pentacosane	$C_{25}H_{52}$	0.48
1,2-Benzenedicarboxylic acid, bis(2-ethylhexyl)	C ₂₄ H ₃₈ O ₄	12.11
Tetracontane, 3,5,24-trimethyl	$C_{43}H_{88}$	0.37
Pentatriacontane	C35H72	6.31

As an additive, tetrapentacontan-1,54-dibromo also does not undergo thermal decomposition. Analysis shows that this component is concentrated because the volatile base oil fraction is distilled off, leaving the non-volatile additives in the residue. The relative percentage in the distillate appears higher in motorcycle and automotive base oils. The presence of additives such as tetrapentacontane-1,54-dibromo in distillates, even at low levels, indicates that some high-molecular-weight and thermally stable compounds may be carried over during the distillation process. This highlights the potential limitations of the process in producing a completely additive-free base oil, which may require additional purification steps (e.g., adsorption) for complete re-purification.

If the compounds in the base oil are grouped, motor base oil consists of 80.37%-mass paraffin and 21.43%-mass aromatic compounds, while car base oil consists of 77.18%-mass paraffin and 0.92%-mass aromatic compounds. This result is slightly different from the study [36], where the base oil contains > 90.0% paraffin and 5%< aromatic compounds.

Table 6. Base Oil of Car with NaOH Solvent

Compounds	Formula	%-mass
Undecane	C ₁₁ H ₂₄	1.00
Benzene, 1,2,3,4-tetramethyl	$C_{10}H_{14}$	0.38
Hexadecane, 1-chloro	$C_{16}H_{33}$	0.75
Dodecane	$C_{12}H_{26}$	2.78
Undecane,2,5-dimethyl	$C_{13}H_{28}$	0.47
1H-Indene, 1-ethyl-2,3-dihydro-1-methyl	$C_{12}H_{16}$	0.54
Octane, 3,6-dimethyl	$C_{10}H_{22}$	0.67
Tridecane	$C_{13}H_{28}$	2.18
Pentadecane	$C_{15}H_{32}$	1.32
Nonadecan	C ₁₉ H ₄₀	4.58
Pentadecane 2,6,10,14-tetramethyl	$C_{19}H_{40}$	0.43
Hexadecenoic acid, methyl ester	$C_{17}H_{34}O_2$	0.37
Eicosane	$C_{20}H_{42}$	3.16
Docosane	C ₂₂ H ₄₆	6.99
Tetracosane	$C_{24}H_{50}$	9.84
Octadecane, 3-ethyl-5-(2-ethylbutyl)	$C_{26}H_{54}$	0.41
Tetrapentacontan1,54-dibromo	$C_{54}H_{10}Br_2$	6.69
Hexacosane	$C_{26}H_{54}$	4.83
Docosane, 6-methyl	$C_{23}H_{48}$	1.09
Heneicosane, 11-(1-Ethylpropyl)	$C_{26}H_{54}$	3.42
Octadecane, 3-methyl	C ₁₉ H ₄₀	1.35
Hexadecane, 2,6,10,14-tetramethyl	$C_{20}H_{42}$	0.91
Tetratetracontane	$C_{44}H_{90}$	4.95
2-Octadecyloxy-1.1.2.2-Tetradeuteroethanol	$C_{20}H_{42}O_2$	2.68
octyl-diphenylamine	$C_{20}H_{27}N$	1.56
Pentatriacontane	$C_{35}H_{72}$	13.51
1,2-Benzenedicarboxylic acid, bis(2-ethylhexyl) ester	C ₂₄ H ₃₈ O ₄	8.95
Tetracosane	$C_{24}H_{50}$	2.35
Pentacosane	$C_{25}H_{52}$	2.62
Tertracontane,3,5,24-trimethyl	$C_{43}H_{88}$	0.40
Tetrapentacosan	$C_{54}H_{11}O$	5.79
Octadecane	$C_{13}H_{38}$	0.93
Nanocosane	$C_{29}H_{60}$	0.61
Dotriacontane	C32H66	0.59

4. Conclusion

Waste engine oil was processed using a vacuum distillation method combined with solvent pretreatment. In addition to varying solvent types, solvent concentrations and incubation times were also studied. Acetic acid with a concentration of 15% and incubation for 48 hours produced the highest

yield (45%). However, at the same concentration and incubation time, the yield using NaOH was much higher and produced visually more apparent oil. The main compounds in fresh engine motor and car oil are heptacosane and titriacontane. These two compounds are not found in base oil. However, additives such as pentatriacontane, hexacosane, and tetrapentacontan-1,54-dibromo have been found in base oil. These additives do not degrade. This combination of pretreatment and vacuum distillation has proven effective in producing high-quality and environmentally friendly base oils. For industrial applications, NaOH pretreatment is more recommended to maximize yield with high purity. Further research should focus on optimizing the two-stage pretreatment process and analyzing its economic feasibility.

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