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ORIGINAL RESEARCH ARTICLE

# Recycling and characterization of used engine lubricating oil

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## ABSTRACT

Used engine lubricating oil contains degradation products and wear-derived metals that require controlled treatment before any recovery option can be considered. This study evaluated five used-oil samples (three gasoline-engine oils and two diesel-engine oils) and examined a staged process comprising settling, magnetic separation, filtration, dewatering, acetic-acid treatment, solvent flocculation, and treatment with two synthesized non-ionic surfactants. Physicochemical properties were measured using standardized methods for density (15 °C), kinematic viscosity (100 °C), flash point, pour point, carbon residue, and ash content, while elemental concentrations were determined by ICP-MS. For surfactant-1-treated samples, gasoline-engine oils showed densities of 0.8714-0.8904 g cm−3, kinematic viscosities of 9.51-13.59 mm2 s−1, and ash contents of 0.010-0.014 wt%, whereas diesel-engine oils showed densities of 0.8934-0.8954 g cm−3, viscosities of 13.16-20.26 mm2 s−1, and ash contents of 0.011-0.012 wt%. Treatment reduced Fe from 40.93 to 28.56 ppm, Zn from 47.26 to 38.83 ppm, and Al from 10.50 to 8.15 ppm; however, sulfur remained high at 407.39 ppm, and no engine, bench, anti-wear, moisture-balance, or long-term stability tests were performed. The results therefore support physicochemical improvement and partial contaminant removal, but they do not establish that the regenerated oil is suitable for direct reuse in engines; operational validation and additive rebalancing are required.

***Keywords:*** used lubricating oil; regeneration; acetic acid; non-ionic surfactant; physicochemical characterization; ICP-MS

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## 1. Introduction

Engine lubricants reduce friction, remove heat, limit corrosion, suspend contaminants, and protect moving surfaces. Maintaining an internal-combustion engine is essential for reliable and efficient operation; torque loss and wear can be reduced by selecting wear-resistant materials and suitable lubricants[1,2]. During service, oxidation, thermal stress, fuel dilution, soot, water, additive depletion, and wear debris progressively alter lubricant performance. In this article, used lubricating oil (ULO) means oil withdrawn after service because its measured or operational properties no longer satisfy the intended service requirement; the term does not imply that every useful base-oil fraction has been destroyed.

Improper disposal of ULO can contaminate soil and water, whereas controlled regeneration can recover hydrocarbon resources. Established routes include acid-clay treatment, solvent extraction/flocculation, adsorption, vacuum distillation, hydrotreating, membrane separation, and catalytic conversion[3-8]. Acid-clay treatment is accessible but can generate acidic sludge and corrode equipment.

Solvent extraction separates oxidized products and suspended matter but requires solvent recovery and careful control of solvent-to-oil ratio. Distillation and hydrotreating can produce higher-quality base stocks, although their energy, pressure, catalyst, and capital requirements limit small-scale deployment. Adsorption is simple but transfers contaminants to a spent solid. Recent reviews therefore identify contaminant removal, additive restoration, product consistency, energy demand, and treatment-residue management as linked technical bottlenecks[22-27].

Rheological and tribological performance cannot be inferred from appearance alone. Density, kinematic viscosity, viscosity index, flash point, pour point, carbon residue, ash, water, acidity, elemental composition, oxidation stability, and wear performance provide different evidence about the recovered fraction[8-12,28-31]. Physicochemical similarity to a fresh oil is necessary but not sufficient for engine reuse because service performance also depends on additive balance, volatility, deposits, corrosion, oxidation resistance, and boundary-lubrication behavior. This distinction is central when evaluating low-cost chemical treatment routes.

The literature contains many studies of acid, solvent, and adsorbent recovery, but fewer studies combine staged physical pretreatment, acetic-acid separation, newly prepared surfactants, standardized bulk-property measurements, and ICP-MS screening across multiple gasoline- and diesel-engine oil origins. The present work addresses this gap by determining how the proposed route changes selected physicochemical properties and elemental concentrations, identifying the acid and surfactant conditions associated with the closest measured values to the fresh-oil comparators, and defining the evidence still required before operational reuse. The study does not claim to certify the regenerated oil for engines. The Experimental section describes materials, synthesis, sample treatment, and measurements; Results and Discussion compare fresh, used, and treated oils; and the Conclusions summarize quantitative outcomes and limitations.

# 2. Experimental

## 2.1. Materials

The chemicals used in this study are listed in **Table 1**.

**Table 1.** Chemicals used in this research.

| no | Name of compound | Formula | Molar mass g.mol$^{-1}$ | Purity% | supplier |
|---|---|---|---|---|---|
| 1 | Acetic acid | $CH_3COOH$ | 60.05 | 99.5 | VWR International (formerly BDH) |
| 2 | Sulfuric acid | $H_2SO_4$ | 98.08 | 99.9 | VWR International (formerly BDH) |
| 3 | Sodium hydroxide | NaOH | 40.00 | 96.0 | VWR International (formerly BDH) |
| 4 | Potassium hydroxide | KOH | 56.11 | 99.9 | VWR International (formerly BDH) |
| 5 | Methanol | $CH_3OH$ | 32.04 | 99.9 | VWR International (formerly BDH) |
| 6 | n-hexane | $C_6H_{14}$ | 86.17 | 99.5 | VWR International (formerly BDH) |
| 7 | Acetone | $C_3H_6O$ | 58.08 | 99.9 | VWR International (formerly BDH) |
| 8 | 1,2-Dichloro ethane | $C_2H_4Cl_2$ | 98.95 | 98.8 | VWR International (formerly BDH) |
| 9 | Pentaethylene hexamine | $C_{10}H_{28}N_6$ | 232.37 | 98.0 | VWR International (formerly BDH) |
| 10 | Tetraethylene pentamine | $C_8H_{23}N_5$ | 189.307 | 98.0 | VWR International (formerly BDH) |
| 11 | Octyl phenol formaldehyde resins | $C_{12}H_{24}O_2$ | 236.349 | 98.0 | SAM Chemical Supplier, Iran |
| 12 | Novolac | $C_9H_{14}O_2$ | 154.205 | 98.0 | SAM Chemical Supplier, Iran |

## 2.2. Instruments

The following equipment was used. FTIR spectra were recorded with a PerkinElmer Spectrum Two spectrometer (PerkinElmer, USA). 1H and 13C NMR spectra were recorded with a Bruker Avance 300 MHz spectrometer (Bruker BioSpin, Germany) using deuterated dimethyl sulfoxide (DMSO-d6) as solvent. Elemental concentrations were measured with an Agilent 7900 ICP-MS system (Agilent Technologies, USA). Pour point was measured according to ASTM D97 using an ISL automatic pour-point analyzer (model information supplied by the laboratory; ISL is a PAC company, France). Flash point was measured according to ASTM D92 with a Koehler K13990 Cleveland open-cup apparatus (Koehler Instrument Company, USA). Kinematic viscosity was measured at 40 and 100 °C according to ASTM D445 using a Koehler K23790 bath (USA). Density at 15 °C was measured according to ASTM D1298 using a C070 hydrometer set (Normalab, France). Sample heating used an IKA integrated magnetic hotplate stirrer, model 1.613.01.001 (Germany). Ash content was measured according to ASTM D482, and surfactant melting points were measured with an HMPD-100 apparatus. Manufacturer and model details not available in the original laboratory record are explicitly identified rather than inferred.

## 2.3. Synthesis of Surfactants

### 2.3.1. Synthesis of Surfactant (1)

Octylphenol-formaldehyde resin (0.01 mmol, based on the reported average resin formula) was contacted with an excess of 1,2-dichloroethane (0.04 mmol), used as reactant and reaction medium, and refluxed at 84 °C for 2 h. After cooling, the intermediate was isolated and washed repeatedly with acetone. The originally reported value of 0.02 mmol for this isolated intermediate was a transcription error; the synthesis was based on 0.01 mmol of starting resin. Because the resin is polydisperse, this stoichiometry is nominal and should be interpreted on the reported average formula basis.

The isolated intermediate was dissolved in dichloromethane and cooled in an ice bath. Aqueous NaOH (0.02 mmol) was added dropwise to pentaethylenehexamine with stirring at 0-5 °C, followed by the resin-derived intermediate. The mixture was stirred for 2 h at 0-5 °C, filtered, washed with acetone and methanol, and vacuum-dried. The proposed terminal substitution shown in Scheme 1 is a simplified structural representation, not proof of exclusive terminal-amine reaction. Internal amines can also participate; definitive site selectivity would require additional two-dimensional NMR or mass-spectrometric evidence. **Table 2** summarizes the isolated product.

**Table 2.** Color, nominal formula, melting point, and isolated yield of surfactant 1.

| Surfactant | Formula | Substances | Color | m.p. (°C) | Yield (%) |
|---|---|---|---|---|---|
| **(1)** | $C_{46}H_{88}O_5N_{12}$ | Octyl phenol formaldehyde resin<br>1,2-Dichloro ethane<br>Pentaethylene hexanmine<br>Sodium hydroxide | Yellow | 132 | 95% |

### 2.3.2. Synthesis of Surfactant (2)

1. Octylphenol-formaldehyde resin (0.01 mmol, nominal average-formula basis) was contacted with 1,2-dichloroethane (0.04 mmol) and refluxed at 84 °C for 2 h. The isolated intermediate was washed repeatedly with acetone. As for surfactant 1, the previously stated 0.02 mmol intermediate value was a transcription error.
2. The isolated intermediate was dissolved in dichloromethane and cooled in an ice bath. Aqueous NaOH (0.02 mmol) was added dropwise to tetraethylenepentamine with stirring at 0-5 °C, followed by the resin-derived intermediate. After 2 h, the mixture was filtered, washed with acetone and methanol, and vacuum-dried. The proposed structure is a representative product distribution because polyamine substitution may involve terminal and internal nitrogen sites. **Table 3** summarizes the isolated product.

**Table 3.** The color, yield, and melting point for the synthesis of surfactant (2).

| Surfactant | Formula | Substances | Color | m.p (°C) | Yield (%) |
|---|---|---|---|---|---|
| **(2)** | $C_{41}H_{68}O_5N_8$ | Phenol formaldehyde resin<br>1,2-Dichloro ethane<br>Tetra ethylene pentamine<br>Sodium hydroxide | Yellow | 113 | 93% |

## 2.4. Filtration Process

The old lubricating oil was filtered to eliminate any contaminants that may have been present in the base oil, including metal chips, sand, dust, particles, and micro impurities. A funnel with a piece of filter paper inside was used for this purpose.

## 2.5. Dewatering Stage

To remove the free water and emulsified water, 500 mL sections of the sample were heated to 180 °C in an open beaker using a magnetic stirrer for 1 hour.

## 2.6. Desludging (extraction) Stage Using Acetic Acid

Acetic acid was evaluated at acid-to-oil ratios of 5, 10, 15, and 20 vol%. For each run, 100 mL of the selected local-origin gasoline ULO was mixed at 60 °C for 2 h, then settled for 6 h in a separatory funnel. The lower acid/sludge phase was drained completely, and the upper oil phase was recovered and measured. No water wash or total-acid-number measurement was included in the original protocol; consequently, trace residual acid cannot be excluded and direct engine reuse cannot be inferred. Among the tested ratios, 5 vol% was selected for subsequent work because its measured viscosity, density, flash point, pour point, carbon residue, and ash values collectively remained closest to the fresh-oil comparator (**Table 4**), while higher ratios provided no consistent improvement.

## 2.7. Experimental Procedure

For the surfactant stage, 200 mL of the selected pretreated ULO was heated to 60 °C and mixed for 20 min. Surfactant 1 or 2 was then added at 5, 10, or 15 g per 200 mL of oil, and mixing continued before 24 h settling. The clarified upper oil phase was decanted from the aqueous/residual phase. The 10 g dose (50 g $L^{-1}$ oil) was selected because it gave the closest kinematic-viscosity value to the fresh-oil comparator among the three tested doses (**Table 5**). No four-ball wear test was performed; the surfactants are therefore evaluated here as treatment/flocculation agents, not validated anti-wear additives.

## 2.8. Recycling Preparation

For solvent-assisted flocculation, 200 mL of ULO was combined with 100 mL of a 70:30 (v/v) methanol:n-hexane mixture and 3.0 g of solid KOH. The suspension was stirred at 600 rpm and 60 °C for 30 min and then settled for 24 h. The lower sludge/KOH-containing phase was separated, and the recovered oil/solvent phase was heated to 120 °C to remove volatile solvent. KOH was not assumed to remain dissolved in the recovered oil; it was removed predominantly with the settled polar/sludge phase. Residual potassium and alkalinity were not independently quantified, which is a limitation of the study.

## 2.9. Analyses of Samples

### 2.9.1. Carbon residue Test (micro-method IP 398)

The vial was filled with half a gram of the sample and placed in the vial holder. After that, the vial holder was placed in the oven and the lid was sealed. For 15 minutes, the sample was heated to 500 °C. After that, it was cooled under 600 cm3/min of pure nitrogen. The vial holder was taken out and placed in a desiccator to cool down after the oven temperature reached precisely 250 °C. We measured the carbon residual after removing and weighing the residue.

### 2.9.2. Ash Content %

Ash content indicates the amount of inorganic residue remaining after combustion and may reflect oil-soluble metallic additives, wear metals, dirt, rust, or other inorganic contamination. It was determined according to ASTM D482.

### 2.9.3. Flash Point Test

To provide a precise measurement, the Cleveland open cup tester was filled to the line where the meniscus sits on top. Prior to applying heat to the heating plate, the diameter reading of the test flame light was set to 4.8 mm. A temperature value was promptly collected and recorded using a thermometer while the flash point was being monitored. The flash point was indicated by this thermometer measurement. A fume cupboard was used to conduct the flash point test.

### 2.9.4. Pour Points Test

Oil loses its fluidity and cannot flow or circulate below its pour point, which is the temperature below which it is no longer effective as a lubricant. Oils for lubrication that have a low pour point and a high specific gravity at 15 °C are of high quality.

Density was measured at 15 °C. It is defined as the sample mass divided by its volume:

$$\rho = m/V \quad (1)$$

where ρ is density (g cm−3), m is sample mass (g), and V is volume (cm3). Kinematic viscosity was measured at 40 and 100 °C according to ASTM D445 and calculated from the calibrated viscometer constant:

$$\nu = C \times t \quad (2)$$

Results and Discussion

This section first evaluates the synthesized surfactants by the available spectroscopic evidence and then compares fresh, used, and treated oils. Each sample is identified by engine type and origin. The discussion is restricted to the measured endpoints; it does not treat property matching as proof of engine-service performance.

$$\text{Recovery yield (\%)} = (V_{recovered}/V_{initial}) \times 100 \quad (3)$$

The temperature dependence of viscosity may be represented by the Walther relation, $\log10[\log10(\nu + 0.7)] = A - B \log10(T)$, where T is absolute temperature and A and B are fitted constants. The present dataset reports viscosity at fixed test temperatures and was not used to fit A and B.

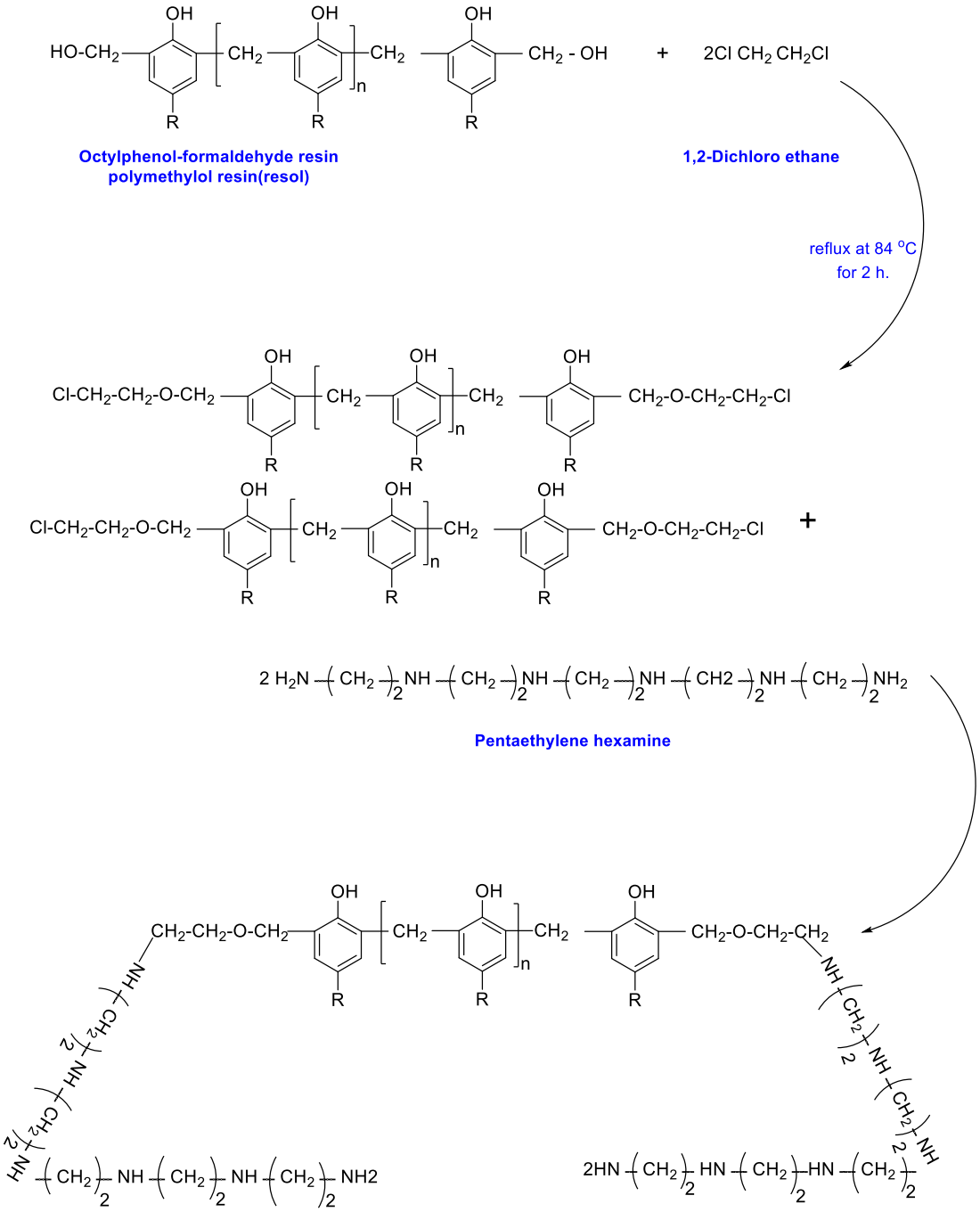
### 2.9.5. Viscosity

Lubricant viscosity is a crucial grading attribute. This metric, which measures the resistance of a fluid to flow, is very sensitive to changes in temperature. Engine oil that has lost some of its viscosity is likely polluted. A lubricating oil's SAE number is a unique identifier in the field of automotive engineering. Most manufacturers of automotive equipment use the SAE viscosity numbers to define the oil viscosity they suggest for their products. A higher SAE viscosity value indicates that the lubricating oil is thicker and heavier.

## 3. Results and discussion

### 3.1. Mechanism of Synthesis for Surfactant (1)

Surfactant (1) based on octylphenol-formaldehyde resins and pentaethylene hexamine was synthesized as illustrated in Scheme 1.




**Scheme 1.** Synthesis of surfactant (1).

### 3.2. FTIR Spectrum of Surfactant (1)

Because of their structural similarities, surfactants have very similar FTIR spectra. **Figure 1** shows the FTIR spectrum of surfactant (1), which shows that at 3270 cm−1, the alcoholic hydroxyl and secondary amine groups of the surfactant were visible as a wide band. The connection between the OH group of octylylphenol-formaldehyde resin and the two major amine groups of pentaethylene hexamine was verified by the elimination of the two NH2 stretching bands at 3400 cm−1. The C−aliphatic stretching bands may be found at 2949 cm−1 and 2818 cm−1. Referring to the aromatic C=C bonds, the stretching band at 1602 cm−1 is significant. The stretching vibration of the C-O bond on phenol rings is associated with the band at 1213 cm−1. Connecting the phenol rings and hydroxymethyl was the stretching vibration of C-C, which was shown by the band at 1117 cm−1. This particular bond occurred at 1047 cm−1[13-15].

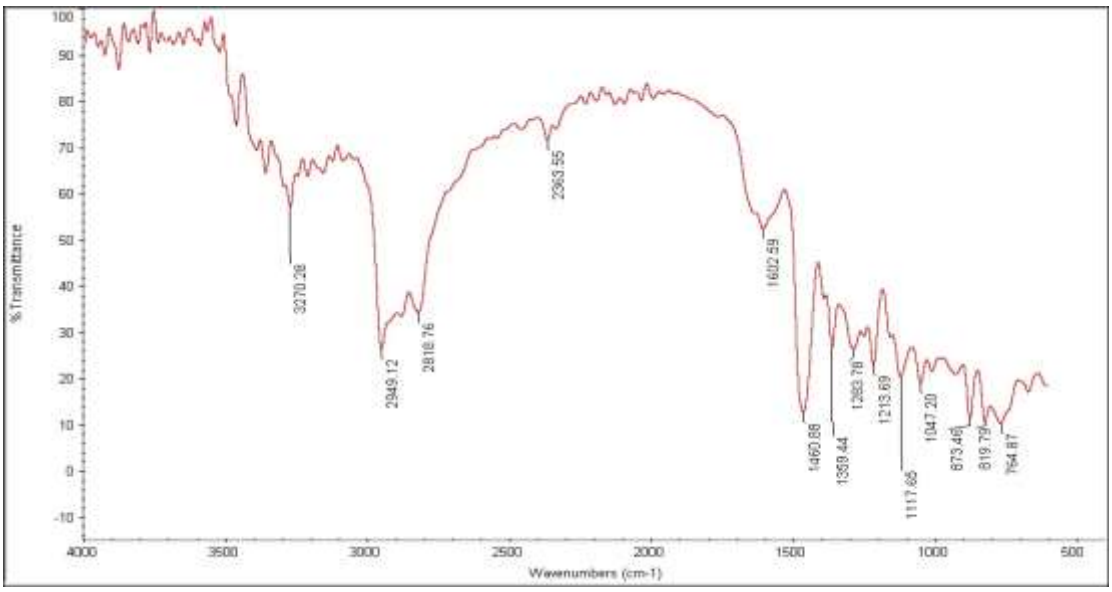



**Figure 1.** FTIR Spectrum of Surfactant (1).

### 3.3. 1HNMR Spectrum of Surfactant (1)

**Figure 2** indicates that the methylene groups are confirmed by the 1HNMR spectra of surfactant (1), which exhibits chemical shifts at 0.4 and 1.2−1.5 ppm. An further set of multiplet peaks appearing at 2.5 ppm verifies that the surfactant's ethylene amine units have really formed. Two new peaks, one at 3.1 ppm and the other at 3.4 ppm, corresponding to CH2O (methylene oxide) and CH linked to OH, have emerged. A multiplet of aromatic protons was detected at 6.6−7.3 ppm, whereas the proton of OH was detected at 8.4 ppm. At 4.3 ppm, the NH2 proton was detected[13-15].

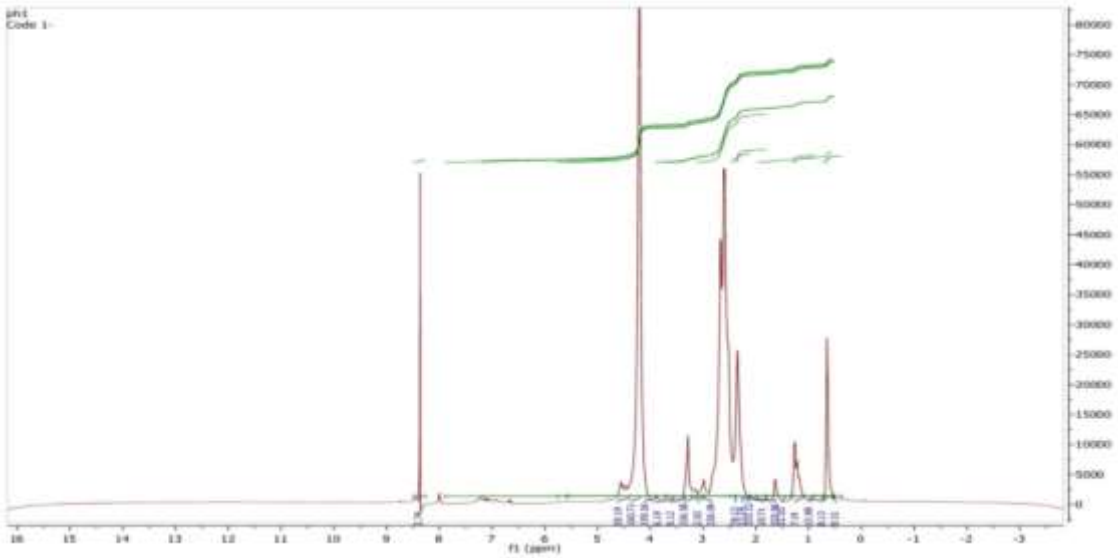


**Figure 2.** [1]HNMR spectrum of surfactant (1).

### 3.4. 13CNMR Spectrum of Surfactant (1)

As shown in **Figure 3**, the chemical structures of the produced surfactant may also be confirmed using the 13CNMR spectrum. The interaction between pentaethylene hexamine and octylylphenol-formaldehyde resin is confirmed by the peaks at 49 and 52 ppm that are attributable to CH2NH. The presence of CH2O is shown by the peaks at 54–58 ppm. In the third area, at 126 ppm, aromatic and olefinic carbon signals emerge, with a solitary signal connected to C-N at 61 ppm[13-15].

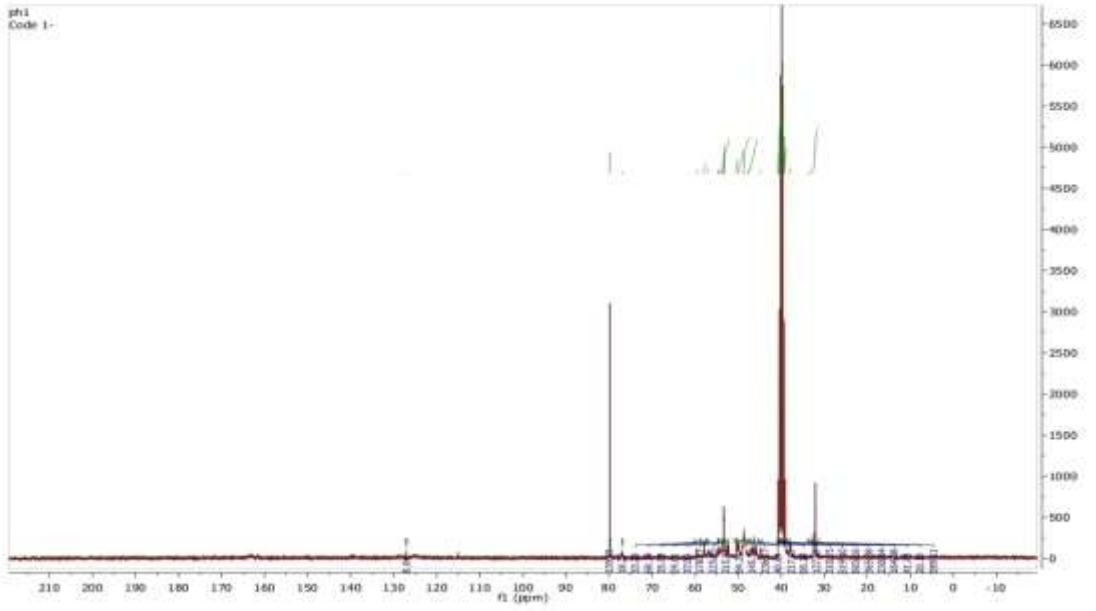
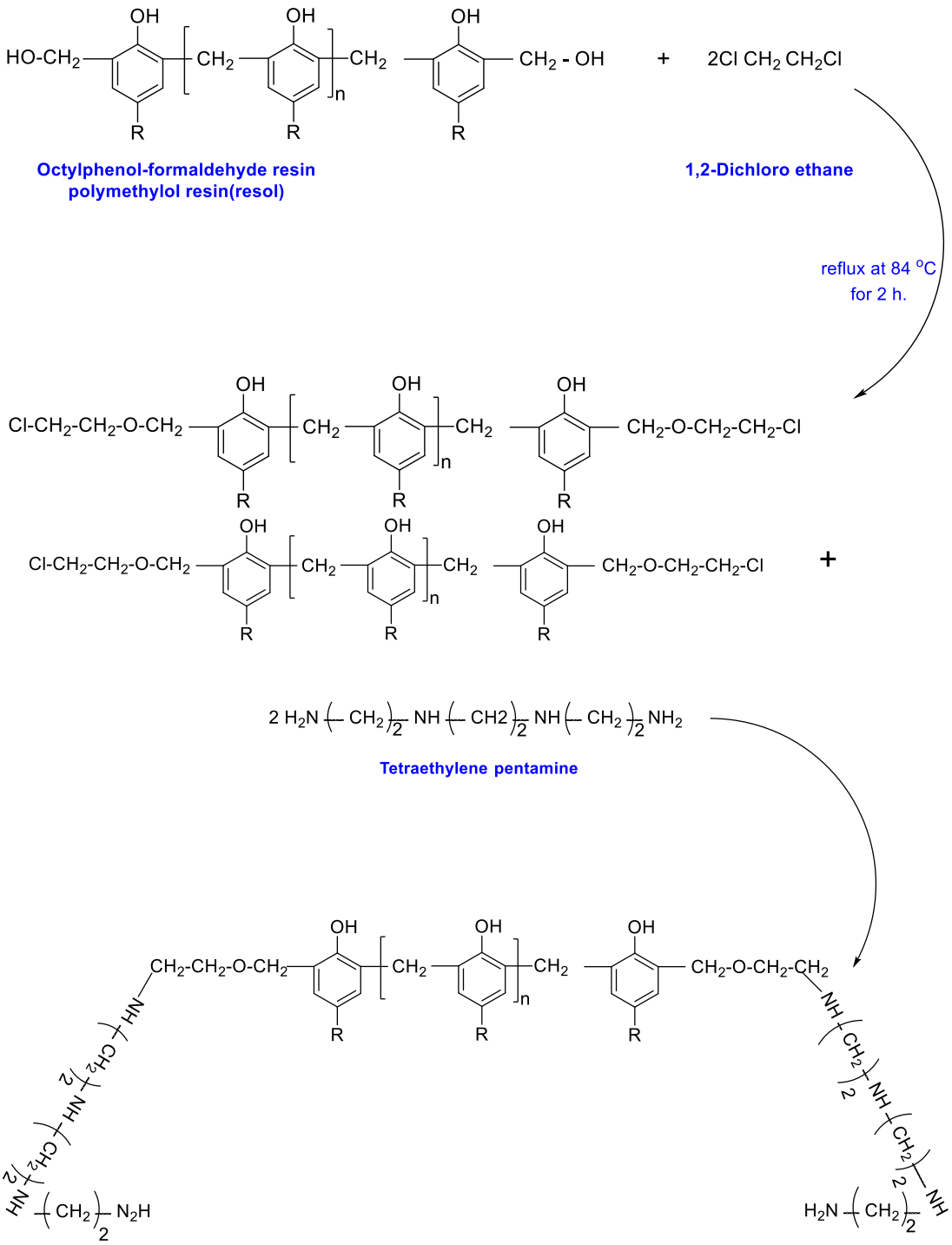


**Figure 3.** [13]CNMR spectrum of surfactant (1).

### 3.5. Mechanism of Synthesis for Surfactant (2)

Surfactant (2) based on octylphenol-formaldehyde resins and triethylenetetramine was synthesized as illustrated in Scheme 2.




**Scheme 2.** Synthesis of surfactant (2).

### 3.5.1. FTIR Spectrum of Surfactant (2)

Because of their structural similarities, surfactants have very similar FTIR spectra. **Figure 4** shows that the alcoholic hydroxyl and secondary amine groups of surfactant (2) showed as a wide band at 3260 cm−1 in the FTIR spectra of surfactant (2). The connection between the OH group of octylylphenol-formaldehyde resin and the two major amine groups of pentaethylene hexamine was verified by the elimination of the two NH2 stretching bands at 3400 cm−1. The aliphatic C-H bonds are shown by the stretching bands at 2918 cm−1 and 2841 cm−1. The aromatic C=C bonds are shown by the stretching band at 1650 cm−1. Phenomenon rings had a C-O stretching vibration peak at 1358 cm−1. Connecting phenol rings with hydroxymethyl, the stretching vibration peak of C-C was observed at 1113 cm−1[13-15].

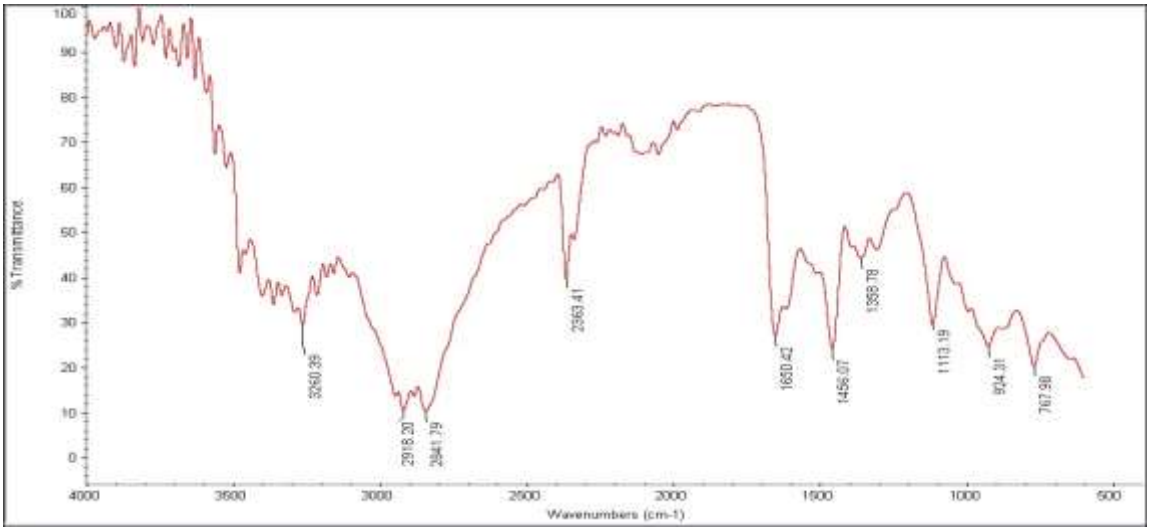



**Figure 4.** FTIR spectrum of surfactant (2).

### 3.5.2. 1HNMR Spectrum of Surfactant (2)

**Figure 5** shows surfactant (2)'s 1HNMR spectra, which verifies the presence of a multiplet of methylene groups at 0.44 and 1.29 ppm. An further set of multiplet peaks appearing at 2.5 ppm verifies that the surfactant's ethylene amine units have really formed. Two new peaks, one at 3.1 ppm and the other at 3.4 ppm, corresponding to CH2O (methylene oxide) and CH linked to OH, have emerged. At 8.0 ppm, the aromatic protons showed up as a multiplet, but at 8.4 ppm, the OH proton showed up. The NH2 proton was detected at 5.0 ppm[13-15].

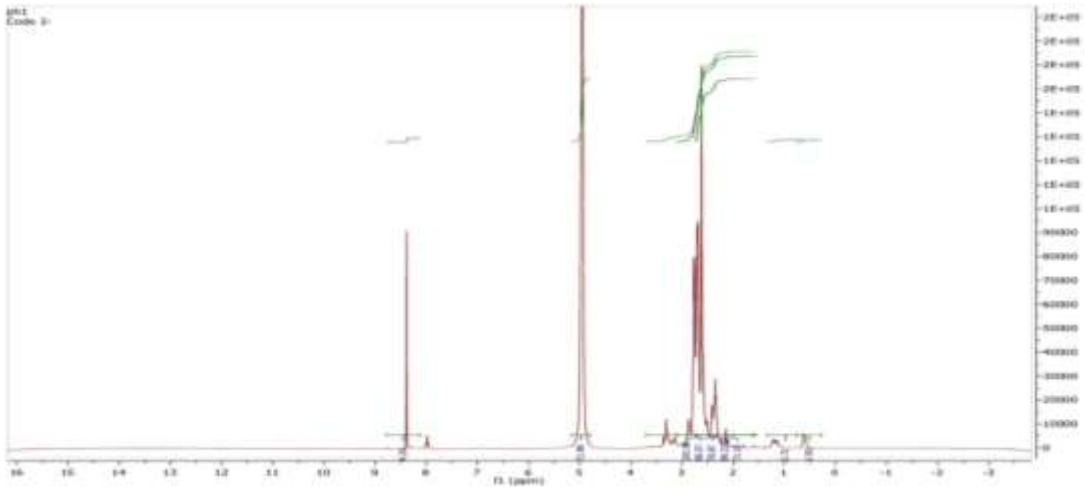


**Figure 5.** [1]HNMR spectrum of surfactant (2).

### 3.5.3. 13CNMR Spectrum of Surfactant (2)

**Figure 6** shows the 13CNMR spectrum that was utilized to confirm the chemical composition of the surfactant that was made. Pentaethylene hexamine reacts with octylylphenol-formaldehyde resin, as seen by the peaks at 49 and 53 ppm that are attributable to CH2NH. Chlorohydrin is responsible for the peaks seen at 54–58 ppm. In the third area, at 126 ppm, the measured value can see signals from aromatic and olefinic carbons, while at 61 ppm, the measured value can see signals from C-N. The CH-N peaks are located at 78 to 80 ppm[13-15].

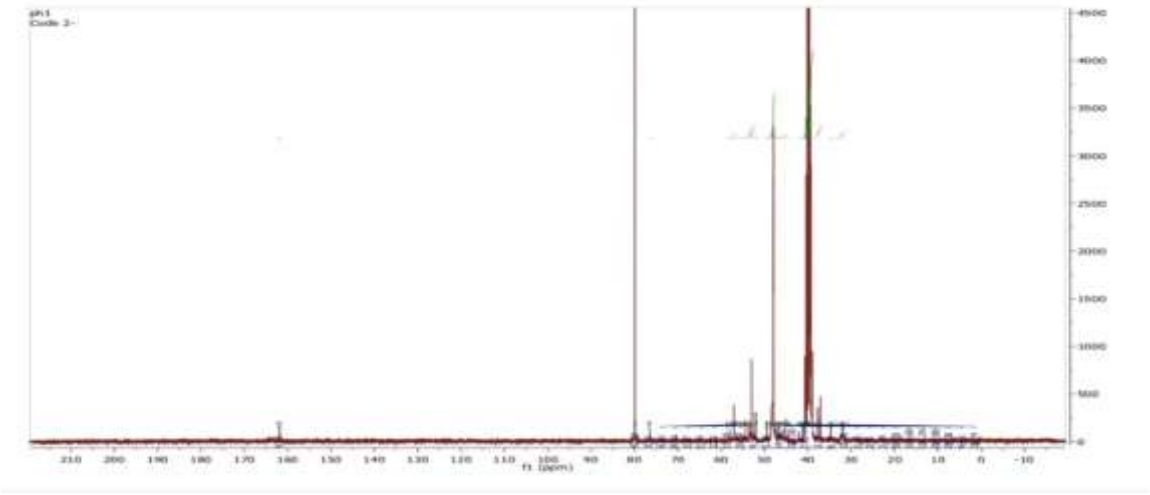


**Figure 6.** $^{13}$CNMR spectrum of surfactant (1).

### 3.6. Role of Surfactant in the Treatment of Used Engine Oil

Used motor oil treatment relies heavily on surfactants, which emulsify or demulsify contaminants (like water, solid particles, and old additives) from the valuable base oil and improve the efficacy of washing or bioremediation procedures. Here are the main responsibilities:

Demulsification: It may be somewhat challenging to separate stable water-in-oil (W/O) emulsions that are present in used oil. By lowering interfacial tension and weakening the protective coating surrounding water droplets, surfactant-based demulsifiers undermine this stability and enable the droplets to coalesce into bigger ones and separate by gravity.

Processes such as acid treatment and thermal washing use surfactants to aid in the removal of inorganic compounds, metals, and specific additives, as well as in the demineralization and washing of contaminants. Thanks to the surfactant, these pollutants end up in a water-based phase that can be easily separated from the oil phase and drained off as a slurry.

When it comes to dissolving and dispersing in water, surfactants are unique in that they contain a hydrophobic tail that attracts oil and a hydrophilic head that attracts water. Through the use of this structure, micelles may encapsulate oil molecules, making it simpler to extract and recover the oil by either "solubilizing" it in water or assisting in its separation from solid particles, such as in the case of oily sludge or contaminated soil remediation.

Modifying Wettability: Surfactants have the ability to change the wettability of particles in solid-surface processes, such as soil remediation or filtration. This makes the particles more hydrophilic, or water-wet, and encourages the oil to separate from the solid.

Enhanced Biodegradation: Certain treatment approaches include the application of targeted surfactants to boost the biodegradation rate and capacity by making oil pollutants more mobile and accessible to microorganisms.

An improved fuel product or a feedstock amenable to further refining into new base oils may be obtained in the end thanks to surfactants, which efficiently separate and remove contaminants.

### 3.7. Determining The Optimal Ratio of Acetic Acid Used

The acetic-acid screening used 5, 10, 15, and 20 vol% for the local-origin gasoline ULO (**Table 4**). Five vol% was retained because it produced the closest overall set of measured bulk properties to the fresh-oil comparator. This selection is specific to the tested sample and endpoints and does not establish direct reusability.

**Table 4.** Screening of acetic-acid dose for the local-origin gasoline oil: fresh, used, and regenerated samples.

| Test | | | 20 vol% acid | | | |
|---|---|---|---|---|---|---|
| | Fresh oil | Used oil | 5 vol% acid | 10 vol% acid | 15 vol% acid | 20 vol% acid |
| Viscosity | 13.52 | 14.59 | 13.59 | 13.50 | 12.01 | 11.50 |
| Density | 0.882 | 0.890 | 0.880 | 0.881 | 0.885 | 0.889 |
| Flash point | 210 | 200 | 205 | 205 | 195 | 200 |
| Pour point | -36 | -31 | -33 | -36 | -32 | -32 |
| Carbon residue | 0.910 | 1.520 | 1.000 | 0.990 | 1.301 | 1.120 |
| Ash % | 0.611 | 0.793 | 0.630 | 0.608 | 0.655 | 0.676 |

## 3.8. Determining The Optimal Amount of Surfactant Used

Surfactant doses of 5, 10, and 15 g per 200 mL oil were compared using kinematic viscosity as the available dose-screening endpoint (**Table 5**). The 10 g dose produced the closest viscosity to the fresh-oil comparator and was selected for subsequent treatment. No direct water-content, anti-wear, wax-deposition, or additive-restoration test was conducted; therefore, these functions are not claimed.

**Table 5.** Screening of surfactant dose (5, 10, and 15 g per 200 mL) for the local-origin gasoline oil.

| No. | Test | | | 15 g/200 mL | | |
|---|---|---|---|---|---|---|
| | | Fresh oil | Used oil | 5 g/200 mL | 10 g/200 mL | 15 g/200 mL |
| 1 | Viscosity | 13.52 | 14.59 | 12.21 | 13.4613 | 12.97 |

## 3.9. Determination of metals in used engine lubricating oil, recycled used engine lubricating oil, and new lubricating oil

Elemental analysis was used to quantify changes in selected metals and sulfur after treatment. The results are reported in ppm by mass and are interpreted only as contaminant-screening data, not as a human-health risk assessment.

**Table 6** reports ICP-MS concentrations in ppm by mass, not percentages, for a composite aliquot prepared from equal volumes of the five ULO samples before and after the same treatment sequence. Treatment reduced the measured concentrations of all listed elements: Fe decreased by 30.2%, Al by 22.4%, Zn by 17.8%, and Pb by 19.0%, while S decreased by 7.9%. Fresh-oil elemental concentrations were not measured in this study, so the post-treatment values cannot be claimed to match virgin oils.

**Table 6.** Elemental concentrations in the composite used-oil sample before and after treatment (ppm by mass).

| Metal | Composite used-oil sample before treatment (ppm) | Composite oil sample after treatment (ppm) |
|---|---|---|
| Al | 10.50 | 8.15 |
| B | 8.07 | 2.57 |
| Ba | 1.65 | 1.26 |
| Ca | 58.57 | 48.46 |
| Cr | 0.22 | 0.20 |
| Cu | 1.30 | 1.23 |
| Fe | 40.93 | 28.56 |
| Mn | 0.61 | 0.47 |
| Mg | 7.51 | 5.77 |
| Zn | 47.26 | 38.83 |
| Pb | 0.42 | 0.34 |
| S | 442.29 | 407.39 |

The decreases in **Table 6** indicate partial removal of the measured elements. Because fresh-oil elemental concentrations, replicate uncertainty, additive chemistry, and species-specific toxicity were not determined, the data cannot establish compliance with lubricant specifications or quantify health risk.

## 3.10. Comparison Between the Used Oil and the New Oil

**Table 7** compares each fresh oil with the corresponding used oil before treatment. The five pairs comprise three gasoline-engine oils (local, Japanese, and German origins) and two diesel-engine oils (local and German origins). The observed property changes differed among origins, supporting separate sample reporting rather than pooled claims.

**Table 7.** Baseline physicochemical properties of the five paired fresh and used oil samples.

| Origin | | Type of lubricating oil | Test | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | | Density (g/cm3) at 15 °C | Viscosity (mm2 s−1) at 100 °C | Flash point (°C) | Pour point (°C) | Carbon residue (% wt) | Ash content (% wt) |
| Gasoline | Local origin | New | 0.8934 | 13.52 | 210 | -33 | 1.710 | 0.011 |
| | | Used | 0.8964 | 14.59 | 214 | -31 | 1.940 | 0.061 |
| | Japan | New | 0.8666 | 11.41 | 229 | -33 | 1.101 | 0.015 |
| | | Used | 0.8754 | 10.70 | 240 | -31 | 2.401 | 0.062 |
| | German | New | 0.8704 | 9.35 | 220 | -36 | 1.915 | 0.011 |
| | | Used | 0.8844 | 8.39 | 230 | -31 | 2.514 | 0.210 |
| Diesel | Local origin | New | 0.8964 | 13.08 | 212 | -30 | 1.441 | 0.012 |
| | | Used | 0.9044 | 13.75 | 222 | -31 | 1.512 | 0.112 |
| | German | New | 0.8934 | 19.03 | 220 | -26 | 1.244 | 0.011 |
| | | Used | 0.8994 | 21.45 | 238 | -24 | 1.908 | 0.061 |

## 3.11. Physical and Chemical Properties

Several physical characteristics of the sample were chosen for measurement in this section. The convenience of the properties' test facilities was a deciding factor in their selection. The chosen physical characteristics include color, density, ash content, pour point, viscosity, and flash point. The findings were contrasted with the utilized oil sample's and the base oil standards', as well as with comparable results.

Visually, this describes how light and transparent petroleum products are. According to the findings of the appearance tests conducted on fresh, worn, and treated lubricant oil, the former has a clear and brilliant hue, while the latter is dark. Because it was heated and oxidized at such high temperatures by the combustion engine for so long, the used lubricating oil became a black color. Treatment with sulfuric acid in the presence of surfactants yields base oil, whereas acetic acid has shown a significant reactivity with the pollutants in the used oil. The base oil treated with acetic acid clearly has a more vibrant hue and a more transparent yellowish hue.

Engine oil has a flash point that is the lowest temperature at which it can spontaneously ignite when heated to a certain degree under controlled circumstances, releasing enough vapour to combine with air. The contamination level of engine oil is indicated by its flash point. If engine oil has a very low flash point, it means it has been polluted with gasoline and other volatile substances. An additional tool for determining the nature of a certain petroleum product is its flash point. A higher molecular weight of oil results in a higher flash point. The production of volatile components during oxidation would cause the flash point to fall. These numbers demonstrate without a doubt that a rise in temperature has a significant impact on the flash point of the base oil[17]. **Tables 8** and **9** represent the flash point test results for gasoline and diesel engine lubricating oils, respectively.

**Table 8.** The flash point for the lubricant oil sample of the gasoline engine.

| Origin | Type of sample (of gasoline ) | Flash point (°C) |
|---|---|---|
| Local origin | New lubricating oil | 210 |
| | Used lubricating oil | 202 |
| | Recycle lubricating oil by surfactant (1) | 207 |
| | Recycle lubricating oil by surfactant (2) | 210 |
| Japan | New lubricating oil | 229 |
| | Used lubricating oil | 211 |
| | Recycle lubricating oil by surfactant (1) | 230 |
| | Recycle lubricating oil by surfactant (2) | 230 |
| German | New lubricating oil | 220 |
| | Used lubricating oil | 210 |
| | Recycle lubricating oil by surfactant (1) | 222 |
| | Recycle lubricating oil by surfactant (2) | 219 |

**Table 9.** Flash points for the lubricant oil samples of the diesel engine.

| Origin | Type of sample | Flash point (°C) |
|---|---|---|
| Local origin | New lubricating oil | 212 |
| | Used lubricating oil | 202 |
| | Recycled lubricating oil by surfactant (1) | 210 |
| | Recycled lubricating oil by surfactant (2) | 211 |
| German | New lubricating oil | 220 |
| | Used lubricating oil | 207 |
| | Recycled lubricating oil by surfactant (1) | 218 |
| | Recycled lubricating oil by surfactant (2) | 220 |

Pour point: The lowest temperature at which engine oil will continue to flow is known as its pour point. The waxes and paraffins included in most engine base oils harden when exposed to cold temperatures. The pour point of engine oil is directly proportional to the amount of wax and paraffin it contains. Engine oils with a high viscosity tend to have high pour points, since the viscosity of the oil has a significant impact on the pour point. When cold weather starts the engine, the pour point of the oil becomes an essential variable. Oil must be able to enter the oil pump and be transported to the engine's different components, even when temperatures are low [18]. The refining process for lubricating oil determines whether the pour point is reduced or raised. The treated oil has a pour point of -33 to -36 ºC, whereas the used engine oil has a pour point of -31 ºC, according to the data in **Tables 10** and **11**, as well as observed improvement in the low-temperature flow properties of the treated oil.

**Table 10.** Pour points for the lubricant oil sample of a gasoline engine.

| Origin | Type of sample (of gasoline ) | pour point (°C) |
|---|---|---|
| Local origin | New lubricating oil | -33 |
| | Used lubricating oil | -31 |
| | Recycled lubricating oil by surfactant (1) | -33 |
| | Recycled lubricating oil by surfactant (2) | -33 |
| Japan | New lubricating oil | -33 |
| | Used lubricating oil | -31 |
| | Recycled lubricating oil by surfactant (1) | -33 |
| | Recycled lubricating oil by surfactant (2) | -33 |
| German | New lubricating oil | -36 |
| | Used lubricating oil | -31 |
| | Recycled lubricating oil by surfactant (1) | -36 |
| | Recycled lubricating oil by surfactant (2) | -33 |

**Table 11.** Pour points for the lubricating oil samples of the diesel engine.

| Origin | Type of sample | pour point (°C) |
|---|---|---|
| **Local origin** | New lubricating oil | -30 |
| | Used lubricating oil | -31 |
| | Recycled lubricating oil by surfactant (1) | -33 |
| | Recycled lubricating oil by surfactant (2) | -27 |
| **German** | New lubricating oil | -26 |
| | Used lubricating oil | -24 |
| | Recycled lubricating oil by surfactant (1) | -26 |
| | Recycled lubricating oil by surfactant (2) | -27 |

Kinematic viscosities: Vapor density, pressure, and temperature are the three variables that determine viscosity. The connection between temperature and viscosity is inverse. The viscosity of motor oil rises as its temperature falls and falls as its temperature rises. Used engine oil may have contaminants detected by viscosity testing. Engine oils with a reduced viscosity suggest gasoline pollution, whereas oils with an increased viscosity may have dissolved and suspended oxidized and polymerized compounds. When used in an engine, base oils oxidize, creating varnishes, deposits, and corrosive oxidized compounds that raise the viscosity of the oil. **Tables 12** and **13** show the kinematic viscosity of used and treated oil at two different temperatures. The oxidized products, deposits, and varnishes may be successfully removed from the used oil using the aforementioned processes. The additives used to increase the viscosity may also have to be removed because of this. Depending on the kind and extent of pollution, it can be more or less than treated engine oil[18].

**Table 12.** Viscosity values for lubricating oil samples of the gasoline engine.

| Origin | Type of sample (of gasoline ) | Viscosity (mm2 s−1) |
|---|---|---|
| Local origin | New lubricating oil | 13.52 |
| | Used lubricating oil | 14.59 |
| | Recycled lubricating oil by surfactant (1) | 13.59 |
| | Recycled lubricating oil by surfactant (2) | 13.12 |
| Japan | New lubricating oil | 11.41 |
| | Used lubricating oil | 10.70 |
| | Recycled lubricating oil by surfactant (1) | 11.57 |
| | Recycled lubricating oil with surfactant (2) | 11.41 |
| German | New lubricating oil | 9.35 |
| | Used lubricating oil | 8.39 |
| | Recycled lubricating oil by surfactant (1) | 9.51 |
| | Recycled lubricating oil with surfactant (2) | 9.51 |

**Table 13.** Viscosity for lubricant oil samples of a diesel engine.

| Origin | Type of sample (of diesel ) | Viscosity (mm2 s−1) |
|---|---|---|
| Local origin | New lubricating oil | 13.08 |
| | Used lubricating oil | 13.75 |
| | Recycled lubricating oil by surfactant (1) | 13.16 |
| | Recycled lubricating oil by surfactant (2) | 13.20 |
| German | New lubricating oil | 19.03 |
| | Used lubricating oil | 21.45 |
| | Recycled lubricating oil by surfactant (1) | 20.26 |
| | Recycled lubricating oil by surfactant (2) | 19.70 |

Density: Previous findings indicate that refined lube oil has a lower density than used lube oil. The treated lubricating oils had a value of 0.8904 whereas the used ones had a score of 0.8964. Compared to refined oil, the density of used oil was greater. The specific gravity of old lubricating oil will be lower than that of new or refined oil if it has been polluted by fuel dilution, water from fuel burning in the engine, or unintentional contamination by rain. Two temperatures are used to determine the specific gravity of a material, which is defined as the mass of the material divided by the mass of water in a given volume. The oil's chemical make-up affects its density. The specific gravity of an oil is proportional to its aromatic and saturated chemical concentrations; as the former rises, the latter falls. According to[19], there is a roughly linear relationship between density, sulfur content, carbon residues, viscosity, and nitrogen content. There is a direct correlation between the quantity of solids in old motor oil and its density. A density increase of 0.00739 is possible for every 1% increase in the mass of solids in the sample. Old motor oil contains carbon-rich oxidized and condensed byproducts. Compared to the used engine oil, which has a density of 0.8964, 0.8754, and 0.8844 for the gasoline engine and 0.9044 and 0.8994 for the diesel engine, the treated oil has a low density of 0.8904, 0.8944, 0.8714, 0.8704, 0.8754, and 0.8754 for the gasoline engine and 0.8954, 0.8934, and 0.8904 for the diesel engine. This information is shown in **Tables 14** and **15**. The presence of oxidation products, metals, and contaminants causes old engine oil to have a high specific gravity. When applied to the base oil, both acids have about the same impact on the specific gravity[17].

**Table 14.** Density values for lubricant oil samples of the gasoline engine.

| Origin | Type of sample (of gasoline ) | Density |
|---|---|---|
| Local origin | New lubricating oil | 0.8934 |
| | Used lubricating oil | 0.8964 |
| | Recycle lubricating oil by surfactant (1) | 0.8904 |
| | Recycled lubricating oi lby surfactant (2) | 0.8944 |
| Japan | New lubricating oil | 0.8666 |
| | Used lubricating oil | 0.8754 |
| | Recycled lubricating oilby surfactant (1) | 0.8714 |
| | Recycled lubricating oilby surfactant (2) | 0.8704 |
| German | New lubricating oil | 0.8704 |
| | Used lubricating oil | 0.8844 |
| | Recycled lubricating oil by surfactant (1) | 0.8754 |
| | Recycled lubricating oil by surfactant (2) | 0.8754 |

**Table 15.** Density values for lubricant oil samples of the diesel engine.

| Origin | Type of sample | Density |
|---|---|---|
| Local origin | New lubricating oil | 0.8964 |
| | Used lubricating oil | 0.9044 |
| | Recycled lubricating oil by surfactant (1) | 0.8954 |
| | Recycled lubricating oil by surfactant (2) | 0.8954 |
| German | New lubricating oil | 0.8934 |
| | Used lubricating oil | 0.8994 |
| | Recycled lubricating oil by surfactant (1) | 0.8934 |
| | Recycled lubricating oil by surfactant (2) | 0.8904 |

Carbon Residue: Coke, or the carbon forming propensity, refers to the quantity of carbonaceous residue that remains following the thermal breakdown of motor oil in a restricted quantity of air. Simultaneously assessing the carbonaceous depositing properties of engine oils used in internal combustion engines is possible with the carbon residue test. One way to estimate how much carbonaceous deposits engine oil might produce in an engine's combustion chamber is to look at its carbon residue value. Because many oils include additives, its relevance is currently questioned. For instance, a detergent ingredient that forms ash may raise the carbon

residue value of engine oil while typically decreasing its deposit-forming propensity[20]. According to **Tables 16** and **17** which include the value of carbon residue for gasoline and diesel engine lubricant oil samples, the carbon residue in base oil made from raw petroleum sources is 0.011 wt%, which is 0.014 wt% lower than the amount made from recycled oil using the sulfuric acid-clay process. Potentially increasing the oil's sulfur content, this might be the result of the intricate chemical interactions between the oil's constituents and sulfuric acid. There is a more direct correlation between carbon residue and the atomic ratio of hydrogen to carbon, nitrogen, and sulfur[21]. Reusing old oil using a mixture of acetic acid and surfactant seems to work better. This oil has a carbon residue that is 0.014 weight percent more than the base oil. The table shows that the carbon residue rose by just 0.004 wt% after adding acetic acid and sulfuric acid to the base oil. It is evident that components are being formed that directly impact the oil's carbon residue.

**Table 16.** The value of carbon residue for lubricant oil samples of gasoline engine.

| Origin | Type of sample | Carbon residue (wt%) |
|---|---|---|
| Local origin | New lubricating oil | 1.710 |
| | Used lubricating oil | 1.940 |
| | Recycled lubricating oil by surfactant (1) | 1.900 |
| | Recycled lubricating oil by surfactant (2) | 1.710 |
| Japan | New lubricating oil | 1.101 |
| | Used lubricating oil | 2.401 |
| | Recycled lubricating oil by surfactant (1) | 1.815 |
| | Recycled lubricating oil by surfactant (2) | 1.559 |
| German | New lubricating oil | 1.915 |
| | Used lubricating oil | 2.514 |
| | Recycled lubricating oil by surfactant (1) | 1.995 |
| | Recycled lubricating oil by surfactant (2) | 2.012 |

**Table 17.** The value of carbon residue for lubricant oil samples of the diesel engine.

| Origin | Type of sample | Carbon residue (wt%) |
|---|---|---|
| Local origin | New lubricating oil | 1.441 |
| | Used lubricating oil | 1.512 |
| | Recycled lubricating oil by surfactant (1) | 1.570 |
| | Recycled lubricating oil by surfactant (2) | 0.828 |
| German | New lubricating oil | 1.244 |
| | Used lubricating oil | 1.908 |
| | Recycled lubricating oil by surfactant (1) | 1.708 |
| | Recycled lubricating oil by surfactant (2) | 1.244 |

Ash content is the inorganic residue remaining after combustion. **Tables 18** and **19** show that treatment generally reduced ash relative to the used oils, but the values reflect both contaminant removal and changes in metal-containing additive species. Accordingly, ash reduction alone cannot establish lubricant service quality.

**Table 18.** The ash content for lubricant oil samples of a gasoline engine.

| Origin | Type of sample | Ash content (%) |
|---|---|---|
| Local origin | New lubricating oil | 0.011 |
| | Used lubricating oil | 0.061 |
| | Recycled lubricating oil by surfactant (1) | 0.014 |
| | Recycled lubricating oil with surfactant (2) | 0.011 |
| Japan | New lubricating oil | 0.015 |
| | Used lubricating oil | 0.062 |
| | Recycled lubricating oil by surfactant (1) | 0.010 |
| | Recycled lubricating oil with surfactant (2) | 0.009 |
| German | New lubricating oil | 0.011 |
| | Used lubricating oil | 0.210 |
| | Recycled lubricating oil by surfactant (1) | 0.010 |
| | Recycled lubricating oil with surfactant (2) | 0.012 |

**Table 19.** The value of ash content for lubricant oil samples of a diesel engine.

| Origin | Type of sample | Ash content (%) |
|---|---|---|
| Local origin | New lubricating oil | 0.012 |
| | Used lubricating oil | 0.112 |
| | Recycled lubricating oil by surfactant (1) | 0.012 |
| | Recycled lubricating oil by surfactant (2) | 0.010 |
| German | New lubricating oil | 0.011 |
| | Used lubricating oil | 0.061 |
| | Recycled lubricating oil by surfactant (1) | 0.011 |
| | Recycled lubricating oil with surfactant (2) | 0.006 |

### 3.12. Comparison of Chemical and Physical properties of the Used Oil sample

The chemical and physical properties of the used oil sample were compared with those of the same new oil. As shown in **Table 20**, it was observed that the used lube oil contains water, soot, lead, and sodium, which are not contained in the new oil sample.

**Table 20.** Summary of measured physicochemical properties of used and surfactant-regenerated oils.

| Type of Lubricating Oil | | Test | Density ($g/cm^3$) at 15 °C | Viscosity (mm2 s−1) at 100 °C | Flash point °C | Pour point °C | Carbon residue % wt | Ash content % wt |
|---|---|---|---|---|---|---|---|---|
| Gasoline | Local origin | Used lubricating oil | 0.8964 | 14.59 | 214 | -31 | 1.940 | 0.061 |
| | | R. by surfactant 1 | 0.8904 | 13.59 | 202 | -33 | 1.900 | 0.014 |
| | | R. by surfactant 2 | 0.8944 | 13.12 | 210 | -33 | 1.710 | 0.011 |
| | Japan | Used lubricating oil | 0.8754 | 10.70 | 240 | -31 | 2.401 | 0.062 |
| | | R. by surfactant 1 | 0.8715 | 11.57 | 230 | -33 | 1.815 | 0.010 |
| | | R. by surfactant 2 | 0.8704 | 11.41 | 230 | -33 | 1.559 | 0.009 |
| | German | Used lubricating oil | 0.8844 | 8.39 | 230 | -31 | 2.514 | 0.210 |
| | | R. by surfactant 1 | 0.8754 | 9.51 | 222 | -36 | 1.995 | 0.010 |
| | | R. by surfactant 2 | 0.8754 | 9.51 | 224 | -33 | 2.012 | 0.012 |
| Diesel | Local origin | Used lubricating oil | 0.9044 | 13.75 | 222 | -31 | 1.512 | 0.112 |
| | | R. by surfactant 1 | 0.8954 | 13.16 | 218 | -33 | 1.570 | 0.012 |
| | | R. by surfactant 2 | 0.8954 | 13.20 | 214 | -27 | 0.828 | 0.010 |
| | German | Used lubricating oil | 0.8994 | 21.45 | 238 | -24 | 1.908 | 0.061 |
| | | R. by surfactant 1 | 0.8934 | 20.26 | 228 | -26 | 1.708 | 0.011 |
| | | R. by surfactant 2 | 0.8904 | 19.70 | 224 | -27 | 1.244 | 0.006 |

## 4. Novelty Statement

The combined route yielded approximately 58% recovered oil under the reported laboratory conditions. The preliminary reagent and operating estimate was approximately USD 79 per liter of recovered oil, compared with USD 7.5-10.5 per liter for new oil at the time of the study. The present laboratory-scale route is therefore not economically competitive. Its contribution is methodological: it evaluates acetic-acid separation and two resin/polyamine-derived surfactants across five sample origins and quantifies changes in bulk properties and selected elements. Scale-up would require reagent recovery, lower surfactant consumption, residue

management, full mass and energy balances, total-acid-number and water measurements, additive analysis, and bench/engine validation.

## 5. Conclusion

The staged physical, acetic-acid, solvent, and surfactant treatment changed the measured physicochemical properties of five used engine oils toward their fresh-oil comparators. For surfactant-1-treated gasoline oils, density was 0.8714-0.8904 g cm−3, kinematic viscosity at 100 °C was 9.51-13.59 mm2 s−1, and ash was 0.010-0.014 wt%; the corresponding diesel ranges were 0.8934-0.8954 g cm−3, 13.16-20.26 mm2 s−1, and 0.011-0.012 wt%. ICP-MS analysis of the composite sample showed reductions of 30.2% for Fe, 22.4% for Al, 17.8% for Zn, and 19.0% for Pb, while sulfur decreased by only 7.9%.

These findings confirm the expectation that the treatment can remove part of the suspended/inorganic contaminant burden and restore selected bulk properties. They do not confirm that the product can be used directly in machinery. Residual acid and potassium, water content, total acid number, oxidation stability, additive depletion, corrosion, volatility, deposits, and anti-wear performance were not measured, and no bench or engine test was conducted. The 58% laboratory recovery and estimated USD 79 L−1 cost also indicate that the present formulation is not commercially viable. Future work should optimize reagent recovery and dosage and then perform replicated moisture, acidity, elemental, FTIR/NMR, tribological, oxidation, bench, and engine tests against applicable lubricant specifications.

## Conflict of interest

The authors declare no conflict of interest.

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