Influence of Solvents on the Extraction of Oil from Waste Lubricating Grease: A Comparative Study

Motshumi Diphare, and Edison Muzenda

Abstract—In this study, the performance of six extracting solvents (n-hexane, toluene, heptane, butane, 1-hexanol, and acetone) was evaluated. The recovery of base oils from waste lithium based lubricating grease was investigated using thermal degradation in an aqueous caustic solution and solvent extraction. Waste grease was initially dissolved in a basic solution at 80°C while being agitated. During this stage most of the oil was recovered. The remaining sludge was cooled to 45°C followed by addition of a solvent to extract the remaining oil. This research further optimised the process by studying the effect of various degrading agents (LiOH, NaOH and KOH) and various solvents (n-hexane, toluene, heptane, butane, hexanol and acetone) on oil recovery. KOH and Hexane produced the highest recovery of 39% and 74.39% respectively.

Keywords—Base, Extraction, Oil Recovery, Solvent Extraction, Waste Grease

I. INTRODUCTION

Solvent extraction is the ability of a solute to distribute itself between an aqueous solution and an immiscible organic solvent. The organic solvent separates and purifies the solutes by extracting into the organic phase, leaving undesirable substances in the aqueous phase [1]. Over the years, fossil fuels have been the cheapest source for petroleum and this led to the development of millions of petroleum based products. However, fuel price and demand have reached record high in recent times triggered by factors such as depletion of easily accessible deposits and ever increasing demand for energy by emerging economies [2].

Steady decline in valuable petroleum commodity against ever-increasing demand has drove researchers to search for alternative resources for energy and chemicals. This includes recycling these hydrocarbons using sustainable methods.

Used lubricant contains a large proportion of valuable base oil that may be used to formulate new lubricants if undesired pollutants are removed [3].

Many processes [3] for recycling used lubricating oil involve the use of vacuum distillation followed by a polishing or decolorizing treatment. However, they present serious problems related to coking and column fouling during distillation and, therefore, some form of pre-treatment to remove additives and contaminants from the oil is required. Furthermore these types of processes are energy demanding due to the high overpressures, deep vacuums and high temperatures applied [4]. The use of extraction has been studied as a low energy consuming alternative by a number of researchers.

A solvent is used selectively to extract the base oil component from the waste lubricant oil. The additives and carbonaceous impurities that are normally present in used oil should be rejected by the extracting solvent. These impurities settle and flocculate based on gravity [5]. The solvent is then recovered by distillation for reuse. However, a portion of this solvent is lost due evaporation during treatment.

Hexane is the solvent of choice for oil extraction mainly due to its efficiency and ease of recovery [4]. Reference [6] reported that hexane is obtained from a non-renewable source and is being consumed faster to the emergence of new sources. It has become necessary to find alternative solvents for oil extraction.

The extractability of oil depends on the nature of the solvent and oil, extraction temperature, contact time between solvent and the feed, pre-treatment conditions of the oil bearing resource [7]. Key properties such as solubility are necessary to evaluate the solvent performance [8]. Reference [9] reported that an increase in solubility parameter difference further enhances the solvent capability to extract the additives and impurities from used oil.

Reference [10] compared the performance of several solvents to extract fuel oil from oil sludge at room temperature. They first used methyl ethyl ketone (MEK) and then LPG condensate at solvent-to-sludge mass ratios ranging from 1:1 to 6:1. The optimum ratio was found to be 4:1. Additional solvents included heptane, hexane, iso-propanol, and iso-butanol. Oil recovery ranged from 8% for the iso-butanol to 39% for the MEK.

Reference [11] used supercritical ethane and dichloromethane to extract hydrocarbons from petroleum sludge using Soxhlet extraction. Recovery ranged from 16% to 55% for supercritical ethane and dichloromethane respectively.
The objective of this study was to evaluate the oil recovery through degradation-flocculation and solvent extraction. The influence of various solvents and degrading agents on oil recovery was investigated.

II. MATERIALS AND METHODS

A. Materials

Technical grade n-hexane, toluene, heptane, butane, hexanol and acetone (95%), and analytical grade potassium hydroxide (KOH), sodium hydroxide pellets and lithium hydroxide (99.9%) were supplied by Laboratory Equipment Supplies (Pty) Ltd. (Germiston, South Africa). The contaminated high temperature metal bearing grease was supplied by the Engine Petroleum (Pty) Ltd.

B. Method

A basic solution was heated to 80°C, followed by a slow addition of 100g of waste grease while agitating at 1000 rpm. After 12min of agitation, brownish oil started to float. The oil was decanted and labelled stage 1 oil. The remaining sludge was then left cool to 45°C. Subsequently a solvent was introduced to extract the remaining oil while agitating. A detailed description of the process was previously discussed by the authors [12].

III. RESULTS AND DISCUSSION

A solvent should dissolve base oil and precipitate other substances to form sludge. n-hexane, toluene, heptane, butane, hexanol and acetone were selected due to their good solubility parameters for base oil and good anti-solvent effect for nonpolar or slightly polar polymeric additives, metals and carbonaceous particles.

Fig. 1 shows variation of oil recovery with the use of various solvents. n-Hexane gave the highest recovery of 74.39% while acetone gave the lowest of 7.43%. The difference between them is related to the solubility of the base oil in these solvents. The performance of the extraction agents can also be related to the different interaction between the solvent and oil molecules, due to the size of the main carbon chain and the solvent molecules configuration [13]. Reference [14] reported that the polarity of solvent is significant to the extraction efficiency in removing oil contaminants. Thus, a solvent has good dissolving ability when its polarity is similar to that of oil.

The recovery increased with increase in solvent carbon chain. This is mainly because carbon atoms lose hydrogen ions and act as a carrier medium for the oil molecules. However, this contradicts the findings in this work for n-hexane and heptane. n-hexane extracts more oil because of its favourable interaction and affinity for oil than heptane.

Previous studies [13, 15] have shown that n-hexane has good oil extraction ability on used lubricants. Moreover other factors such as physical properties of the solvent, particularly its low boiling point attribute to the choice of n-hexane as the optimum solvent [16]. Toluene was found to be the second best extracting agent. Reference [17] compared extraction of oil from lubricating grease using toluene and methyl-tert-butyl-ether (MTBE). The tests results indicated that toluene was most suitable for this application based on its higher 26 w/w% of oil recovery than 19.6% for MTBE.

Reference [18] reported that the extracting solvent must have two important properties; they should be miscible with the base oil contained in the waste oil being processed and must reject from the solution the used oil impurities allowing their aggregation to particle sizes big enough to separate from the liquid by sedimentation.

Table I shows the various degrading agents investigated in this study. Fig. 2 shows the influence of various degrading agents on oil recovery. Reference [13] reported that addition of a base promotes a fast flocculation of the impurities, which are segregated from the base oil by the solvent. The flocculation is enhanced in the presence of OH groups that neutralize the electrostatic repulsion. This electrostatic repulsion exists since alcohol groups are linked with the ions from the additive. Furthermore, addition of a solution containing ions that neutralize those charges, break this stability. Participation of alcohol OH groups in ion exchange reactions with the electrolyte ions helps to neutralize the electrostatic repulsion.

Effective separation of thickening agent from primary base oil in used grease is mainly influenced by the properties of the degrading agent used such as solubility in water, concentration and temperature. Alkali-hydroxides are highly soluble in water compared to alkali-carbonates and they tend to have a strong affinity of hydrolysing grease molecules in suspension [17].

At the molecular level, KOH is slightly smaller than NaOH, therefore it can penetrate grease matrix faster than NaOH. This makes KOH to ionize quickly in water and form stronger nucleophile OH’ ions for breaking polar bonds that holds the thickener and base oil in grease.

LiOH is less basic, therefore it is unable to donate OH’ ions rapidly compared to sodium, potassium and other alkali.
metals. The ability of strong inorganic degrading agent depends on the base metal reactivity and concentration. A base is a proton acceptor and the more readily willing it is to accept a proton, the stronger it is. The base molecule, X-O-H, dissociates in water forming X⁻ ions and OH⁻. The hydrogen molecule in the oil molecule C-R-H (the C-R- represents a carbon structure) attaches itself to the OH⁻ in the aqueous solution and forms water and the remaining C-R- oil molecule.

Reference [19] reported that first ionization energy of group 1 alkali metals decreases down the group on the periodic table. When a metal reacts, it loses electrons to form ions with stable electron configurations. When a lot of energy is required for it to lose electrons, it is going to be less likely to react than a metal that easily sheds electrons. Therefore, the higher the ionization energy for alkali metals, the lesser is the reactivity.

Also, as you go down the group, the distance between the nucleus and the outer electrons increases. The electrons therefore become easier to remove since the atoms get bigger and the reactivity increases. There are more shells preventing the attraction between the positive nucleus and the negatively charged outer electron reducing the electrostatic force. Based on this, KOH was found to be the optimum degrading agent.

**TABLE I**

<table>
<thead>
<tr>
<th>Degradation Agent</th>
<th>Solubility 30°C (g/100ml)</th>
<th>H₂O @</th>
<th>Basicity (pKb)</th>
<th>Cost (US$/kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>LiOH</td>
<td>12.8</td>
<td>-0.36</td>
<td>6.38</td>
<td></td>
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<tr>
<td>NaOH</td>
<td>111</td>
<td>0.2</td>
<td>1.42</td>
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<tr>
<td>KOH</td>
<td>121</td>
<td>0.5</td>
<td>2.11</td>
<td></td>
</tr>
</tbody>
</table>

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**REFERENCES**


