Preparation and Characterization of Grease using Used Cooking Oil and Used Engine Oil

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Abstract. Used engine oil (UEO) and used cooking oil (UCO) are treated as hazard to human health and the environment. A proper management of them are necessary to prevent its adverse impacts. This study is to examine the potential of UEO and UCO used as raw material in preparing grease. UEO and UCO has the potential in replacing the base fluid (mineral oil) and thickener precursor, respectively in conventional grease making. The thickener has been prepared by using calcium hydroxide and sodium hydroxide to react with UCO with the ratio of 1:2 (hydroxide: UCO) and denoted as calcium and sodium thickener, respectively. In grease making, different concentration of thickener (20, 30 and 40%) was used to react with UEO for 180 min at 160 – 180 °C. The formation of grease is confirmed by FTIR analysis, where a new peak was form in 1377 cm⁻¹ – 1423 cm⁻¹. Apart from that it was found that, 20% calcium thickener with 80% UEO grease and 40% sodium thickener with 60% UEO grease were comparable with commercial grease in term of dropping point and water resistance properties.

1. Introduction

Used engine oil (UEO) is usually discharge from engine parts of land vehicle such as cars, motorcycles, buses and trucks and marine transportation like ships, boats, as well as aircrafts. UEO is widely used to lubricate engine parts and has been used extensively in the equipment and machines in agricultural, industrial and mining industries [1]. Lubricants are one of the important products derived from crude petroleum. The main purpose of a lubricant is to keep the moving/sliding surfaces apart, so that friction and consequent destruction of material is minimized. Apart from the main function a lubricant may also perform various other supplementary functions such as temperature control by conducting away the heat of friction, removal of contaminants, corrosion and rust prevention, elimination of gum and sludge formation, electrical insulation, power transmission, shock reduction and sealing to prevent loss of pressure or entry of dust.

UEO is a very dangerous polluting product. It contains polynuclear aromatic hydrocarbons (PAH) and high levels of heavy metals. PAH, such as benzopyrene, are well known for their high carcinogenicity. Considerable quantities of heavy metals are contained in used crankcase oil; these metals are highly toxic to organisms [2].

The environment degradation occurs due to disposal of UEO (Burning, land filling, accidental spillages, pipelines leakages and migration into surface or ground water.) This degradation will pose a threat to human life as well as others living organisms. Therefore, there is a great need for the reutilization of UEO and used cooking oil (UCO) into useful recycled products. Recycling is
considered to be beneficial to environment and to economic development since it decrease demand for landfill space and generally involves saving in energy. [3] The objective of this research is to prepare grease from UEO and UCO. The formation of grease was examined by Fourier-transform infrared spectroscopy (FTIR) and the properties were investigated by using dropping point and water resistance test.

2. Methodology

2.1. Material
UEO and UCO are obtained from the restaurant and automotive workshop. UCO was collected from DRB-HICOM University Canteen which used for making food while the UEO was collected from UMW Toyota Service Centre. Calcium Hydroxide and sodium hydroxide (99.9%) were purchased from Sigma Aldich Malaysia and used without further purification.

2.2. Preparation of thickener
Each 26 wt% calcium and sodium hydroxide solution were prepared in distilled water. The distilled water was heated until boiling point. Calcium hydroxide were then dissolved in solution with continuous stirring by using high speed stirrer. Same step is applied to prepare sodium hydroxide. For the purpose to evaporate water from incomplete thickener, mixture temperature was raised to 120°C-130°C for 2 hours without stirring and let it cool down until it reaches room temperature.

2.3. Preparation of grease
Samples of grease with concentration of the thickener at 20% (w/v), 30% (w/v), and, 40% (w/v) of the total volume of waste engine oil were selected to prepare the calcium and sodium grease respectively. Initially 50% of the total volume of filtered UEO was charged in the reactor and heated up to 90 °C. Then 50% of the sample (calcium/sodium) was added and the stirred vigorously for intimate mixing. Then the temperature was gradually raised to 160 °C to let sample (calcium/sodium) soap to be fully melted in the UEO. When the temperature reaches 160°C, another 50% UEO and the 50% thicker (calcium/sodium) was added along with stirring. The temperature is rises to 190°C for calcium thickener. Calcium thickener will dissolve at 190°C, so the reaction temperature must be heated until 190°C. Meanwhile, sodium thickener dissolve only at 160°C. The mixture was vigorously stirred for 180 minutes at 190°C and 160°C respectively to calcium and sodium thickener, and then the temperature was lowered gradually until it reached room temperature whereas the viscous oil turn into semisolid form grease. In these experiments of grease preparation, no additives were incorporated in the formulation of grease, 20% Ca+ 80% UEO, 30% Ca + 70% UEO, 40% Ca + 60% UEO, 20% Na + 80% UEO, 30% Na + 70% UEO and 40% Na + 60% UEO, denoted as 20-Ca based grease, 30-Ca based grease, 40-Ca based grease, 20-Na based grease, 30-Na based grease and 40-Na based grease respectively.

2.4. FTIR analysis
The spectroscopic measurements of the grease were performed using a PerkinElmer FTIR Spectrophotometer. Samples (UCO, UEO, 20-Ca based grease and 40-Na based grease) were scanned and the spectrum was recorded ranging from 4000 cm⁻¹ to 400 cm⁻¹.

2.5. Dropping point
Dropping point is an indicator of the heat resistance of the grease and is the lowest temperature in which the oil can flow and the phase of grease changes from semi-solid to liquid [4]. This temperature was determined by the standard method of ASTM D566. The samples of 20-Ca based grease, 30-Ca based grease, 40-Ca based grease, 20-Na based grease, 30-Na based grease and 40-Na based grease were used in this experiment. The temperature will be increase to investigate the oil leak. The dropping point was identified when the temperature at which the first drop is observable.
2.6. Water resistance

The ability of water to wash greases was obtained by the standard method of ASTM D1264. The purpose of conducting this standard test method is to determine the stability of grease lubricants on the bearings against washing by the water (water washout characteristic). This test was repeated two times to decrease the errors in results.

3. Results and discussion

3.1. Fourier-transform infrared spectroscopy analysis

![FTIR results of UCO, UEO, 20-Ca based grease and 40-Na based grease](image)

**Figure 1.** FTIR results of UCO, UEO, 20-Ca based grease and 40-Na based grease

The FTIR spectra of UEO and UCO are shown in Figure 1. As shown in Figure 1, the spectra demonstrate a number of absorption peaks, representing the multipart nature of the sample examined. The spectra of UCO were fundamentally similar to those of UEO. For example, the multiple absorption band at 2925 cm⁻¹-2855 cm⁻¹ and 1467 cm⁻¹-1376 cm⁻¹ and 722 cm⁻¹, which corresponds to C-H stretching and bending vibrations, indicated the presence of hydrocarbon compounds [2, 5-8]. The broad and strong bond at ~3682 cm⁻¹ discovered in UEO was ascribed to the O-H or N-H stretching vibration [7]. Meanwhile, the bands at 1742 cm⁻¹ and 1740 cm⁻¹ were assigned to C=O stretching which defines that the functional groups of aldehydes, carbonyls and ketones takes place [2, 7, 9]. The bands at 1239 cm⁻¹ and 1302-1234 cm⁻¹ were associated with C-O and C-N, which confirmed the presence of esters and succinimide [5, 7]. Bands associated with C-O were located at 1163 cm⁻¹ and 1159 cm⁻¹, which illustrated the presence of hydrocarbon/carboxylic acid [2, 5-7]. Last but not least, the band at 722 cm⁻¹ and 720 cm⁻¹ attributed to C-H stretching vibrations [8].
It is noticeable that stretching vibration of C-H group in Ca grease showing a red shift from 2922 cm\(^{-1}\) and 2925 cm\(^{-1}\) respectively UEO and UCO to 2921 cm\(^{-1}\). Furthermore, similar pattern of red shift was noticed from 2855 cm\(^{-1}\) to 2852 cm\(^{-1}\) for UCO. Further to this, another red shift was observed from 1464 cm\(^{-1}\) and 1462 cm\(^{-1}\) to 1461 cm\(^{-1}\) to UEO and UCO respectively. In contradiction, blue shift is also involved in this intramolecular bonding for UEO from 2849 cm\(^{-1}\) to 2852 cm\(^{-1}\). In addition, the disappearing peak of C=O referring to aldehydes, carbonyls and ketones were traced in between band 1743 cm\(^{-1}\)-1745 cm\(^{-1}\). Besides that, missing peak in between 1160 cm\(^{-1}\)-1164 cm\(^{-1}\) could possibly be the diminish of C-O which referring to carboxylic acid group. A new peak was observed at 1377 cm\(^{-1}\) likely related to CH\(_3\), which confirming the formation of grease.

Meanwhile, In Na grease, detection of red shift is observed from 2923 cm\(^{-1}\) for UEO and 2921 cm\(^{-1}\) for UCO to 2919 cm\(^{-1}\) occurred in the C-H stretching vibrations. Additionally, another red shift takes place at band 1464 cm\(^{-1}\) and 1466 cm\(^{-1}\) to 1447 cm\(^{-1}\). Further to this, the disappearing peak detected at band 1744 cm\(^{-1}\) could be possibly due to breakdown of carbonyl compounds, C=O. Besides, peak missing detected between 1464 cm\(^{-1}\)-1160 cm\(^{-1}\) is probably owing to the diminish of C-H bonding interactions. Surprisingly, there is two new peaks detected at band 1379 cm\(^{-1}\) and 1423 cm\(^{-1}\) that is likely to be the bonding of COO and CH\(_3\) respectively which verify the grease formation.

### 3.2. Grease appearance

Table 1 shows the physical appearance of the prepared grease samples. All the samples obtained were in dark colour due to absence of bleaching step during grease preparation. Dark brown colour was visualised for Ca based grease, while black colour for Na based grease. Four types of texture were observed which is fluid, semifluid, firm semisolid and solidify. 20-Na based grease is present as fluid texture. Semifluid texture was observed in 30-Na based grease. Both 20-Ca based grease and 40-Na based grease visualise the texture of firm semisolid. Solidify texture were observed for 30-Ca based grease and 40-Ca based grease. The texture of the grease is crucial to ensure the eligibility for testing. The sample that can proceed for the dropping point and water resistance testing are 20-Ca based grease, 20-Na based grease, 30-Na based grease and 40-Na based grease. Both 30-Ca based grease and 40-Ca based grease are not recommended for testing due to solidify properties.

Table 1. Grease appearance

| No | Sample          | Appearance   | Texture         | Testable (Dropping point & Water Resistance) |
|----|-----------------|--------------|-----------------|---------------------------------------------|
| 1  | 20-Ca based grease | Dark brown  | Firm semisolid | Suitable                                    |
| 2  | 30-Ca based grease | Dark brown  | Solidify       | Too hard for testing                        |
| 3  | 40-Ca based grease | Dark brown  | Solidify       | Too hard for testing                        |
| 4  | 20-Na based grease | Black      | Fluid          | Suitable                                    |
| 5  | 30-Na based grease | Black      | Semifluid      | Suitable                                    |
| 6  | 40-Na based grease | Black      | Firm semisolid | Suitable                                    |

### 3.3. Dropping Point Testing

Results of dropping point for both Ca and Na based grease are shown in table 2. For 30-Ca based grease and 40-Ca based grease were not suitable for testing due to its solid state properties. 20-Ca based grease presented 90 ± 5% °C rate of dropping point. Meanwhile, for Na based grease, which are 20-Na based grease, 30-Na based grease and 40-Na based grease obtain 150 ± 5% °C, 160 ± 5% °C and 175 ± 5% °C respectively. Overall, Na based grease is better than Ca based grease in terms of dropping point due to suitable properties from Na based grease. The grease with higher temperature performance presented better stability [10].

Table 2. Dropping point result for calcium and sodium based grease
### Ca based grease (concentration)

| Samples            | Rate of dropping point |
|--------------------|------------------------|
| 20-Ca based grease | 90 ± 5% °C             |
| 30-Ca based grease | Not suitable for testing |
| 40-Ca based grease | Not suitable for testing |

### Na based grease (concentration)

| Samples            | Rate of dropping point |
|--------------------|------------------------|
| 20-Na based grease | 150 ± 5% °C            |
| 30-Na based grease | 160 ± 5% °C            |
| 40-Na based grease | 175 ± 5% °C            |

3.4. Water Resistance Testing
The status of water resistance for all greases at the standard condition is reported at Table 3. 30-Ca based grease and 40-Ca based grease are not eligible for testing owing to its solid properties. Meanwhile, 20-Na based grease, 30-Na based grease and 40-Na based grease resulted in poor water resistance as it is soluble in water. As for 20-Ca based grease showing an excellent water resistance and resist water washout. Ca based grease is better than Na based grease in terms of water resistance due to the water washout characteristics. Results indicate that lower concentration of Ca based grease caused better performance of water resistance and application for moisture condition [10].

**Table 3. Water resistance testing result for calcium and Na based grease**

| Ca based grease (concentration)                  |
|--------------------------------------------------|
| **Samples**                                      |
| 20-Ca based grease                               |
| 30-Ca based grease                               |
| 40-Ca based grease                               |
| **Status of water resistance**                   |
| Excellent water resistance, resist wash out      |
| Unable to do testing due to solid state          |
| Unable to do testing due to solid state          |

| Na based grease (concentration)                  |
|--------------------------------------------------|
| **Samples**                                      |
| 20-Na based grease                               |
| 30-Na based grease                               |
| 40-Na based grease                               |
| **Status of water resistance**                   |
| Poor water resistance, soluble in water          |
| Poor water resistance, soluble in water          |
| Poor water resistance, soluble in water          |

3.5. Discussion
Converting used engine oil to grease which derived from waste product, useful to support the growth of green environment and technology. From FTIR spectra, new peak detected in the range 1377 cm⁻¹ - 1423 cm⁻¹ proves that the formation of grease [3, 4]. Ca based grease (20-Ca based grease) is predominant among Ca based grease with a good water resistant which suitable for application in moisture condition [11]. On the other hand, Na based grease (40-Na based grease) in sodium grease
gives the best result among them with high dropping point which is suitable for high temperature and extreme pressure usage condition [12].

4. Conclusion
Ca and Na based grease are successfully prepared through combination of sodium/calcium thickener with UEO at concentration of 20%, 30% and 40% without any additives involved. Both Ca and Na based grease were tested through FTIR, dropping point testing and water resistance testing to verify the formation of grease, suitability of sample towards higher temperature and moisture condition. Results from FTIR showing grease formation confirmed when new peak appeared in the range of 1377 cm\(^{-1}\) - 1423 cm\(^{-1}\) for Ca based grease and Na based grease respectively. Dropping point testing proves that that 40-Na based grease is the best grease to withstand higher temperature condition. Meanwhile, water resistance testing resulted that 20-Ca based grease is effective to be applied under moisture condition.

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