Recovery and Characterization of Used Lubricating Oil Using Acid With Two Different Adsorbents

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ABSTRACT This study is a form of experimental analysis that utilizes used lubricating oil (ULO) in order to reclaim base oil by using a combination of acetic acid and two different adsorbents namely aluminum oxide (Al_2O_3) and river sand (RS). The two different adsorbents were used to compare for better quality of oil using the same method. The characterization of the recovered ULO samples was conducted by using Fourier-transform infrared spectroscopy (FTIR) and the viscosity was tested by using the viscometer. Based on the results obtained, the Al_2O_3 seems to be a better adsorbent than RS in several tests such as density, sludge removal and viscosity. For better viscosity and mass of sludge values, the Al_2O_3 adsorbent is more suitable compared to the RS. It was found that by using Al_2O_3, there is a 26% viscosity reduction for ULO samples. By using RS, 6.67% viscosity reduction was found for ULO samples. 24.9% and 25.7% of sludge removal was found in ULO samples by Al_2O_3 and RS, respectively. FTIR analysis showed that before treatment oxidative compounds such as alkens and helides were present in the ULO and UEO samples. However, after treatment by both of the adsorbents, the oxidative compounds were removed. The removal of the alkenes and alkyl halides has evidently indicated the treatment was able to remove the oxidative compounds in the oil.

KEYWORDS: Used lubricating oil, adsorbent treatment, viscosity, metal content analysis, reclamation methods
Received 24 December 2021 **Revised** 11 March 2022 **Accepted** 16 March 2022 **Online** 20 April 2022

Original Article

INTRODUCTION

Lubricating oils (LOs) are viscous liquids that are commonly used for the lubrication of the moving part or surfaces of the engines and machines (Udonne, 2011). In addition to the reducing friction between moving parts, LO also aids in heat transfer. Throughout the usage, LOs endure alterations in terms of termed degradation and contamination. In order to improve the capability of the LO, the base oil is blended with additives (Moura *et al.*, 2010). After lengthened exposure of the high temperature, some of the essential properties of the LO reduces. Therefore, the properties like viscosity, specific gravity and metal wear experience changes from the optimum (Aljabiri, 2018; Osman *et al.*, 2018; Emam and Shoaib, 2012).

In the past, the used lubrication oils (ULOs) were disposed of by land filling, track side foliage control, dumping, extraction for energy production and road oiling was used (Katiyar and Husain, 2010). However, this method is hazardous to the environment as it can cause various serious environmental problems. Another common disposal is burning of the oil to produce energy. There are many reclamation methods for ULO, mainly acid and clay treatment, adsorbent treatment, continuous elusive technique, sharp sand bed filtration, solvent extraction (Abo-Dief *et al.*, 2014; Hamawand *et al.*, 2013; Omolara *et al.*, 2015; Osman, 2019; Rincon *et al.*, 2007; Scapin *et al.*, 2007). In some studies, the combination of some of these techniques has been used for improving the quality of the oil obtained (Abro *et al.*, 2013; Abu-Elella *et al.*, 2015; Anisuzzaman *et al.*, 2020; Oladimeji *et al.*, 2018; Shaikh & Mahanwar, 2018). Regeneration of ULOs seems the most environmentally safe and economical decision.

Therefore, the aim of this study was a form of experimental analysis that utilizes ULO in order to reclaim fresh base oil by using a combination of acetic acid and two different adsorbents namely aluminum oxide (Al_2O_3) and river sand (RS). The two different adsorbents were used to compare for better quality of oil using the same method. The characterization of the recovered ULO samples was conducted by using Fourier-transform infrared spectroscopy (FTIR) and the viscosity was tested by using the viscometer.

METHODOLOGY

Materials

Two different ULO samples (one was lubricant oil and another was engine oil) have been collected from a local car service center. The ULO samples underwent acid treatment followed by treatment with two different adsorbents namely Al_2O_3 and RS. Total 4 samples were prepared (samples 1 and 3 have been treated with Al_2O_3 and samples 2 and 4 have been treated with RS). Then, the oil treated with adsorbents were undergone through centrifuge and filtered. The final LO obtained was compared to the virgin motor oil (VMO) to determine the efficiency of the acid-clay method in reclaiming the oil. Glacial acetic acid for acid treatment and nitric acid, cyclohexane, diethyl ether for RS treatment was purchased from Sigma Aldrich.

Preparation of RS

Initially, the RS was weighed using a measuring scale to 20 g. The sample was then washed in warm water and rinsed in distilled water. Next, 300 mL of cyclohexane and 300 mL of diethyl ether was measured and kept into separate conical flasks in the fume chamber. Following that, the RS sample was washed and partially dried. Next, the RS was washed cyclohexane first and then with diethyl ether. The 300 mL of 0.3 M nitric acid was used to add to the beaker containing the washed RS. The RS sample was washed with distilled water several times until all the nitric acid residue had been washed out and was heated in an oven at 300^oC for 2 h. The dried sand was kept covered with aluminum foil once the drying was done (Ofunne & Maduako 1990).

Treatment of ULO

ULO was filtered to remove unwanted solid particles present in the oil. Then, the oil was subjected to a dehydration process to remove the presence of water in the oil at 250^oC in the oven for 1 h. Next, 8 mL of acetic acid was added to the oil. The oil was then transferred to the glass bottle before centrifuge. The oil was immersed in a hot bath at 100 rpm for 5 h in order to create the separation of the contaminants and base oil. The following treatment was the treatment with adsorbents. First, the sample was to be tested with RS and weighed according to adsorbent to oil ratio of 1:2. The oil was moved to a beaker and mixed with the weighted RS. For the sample that was to be tested with aluminum oxide, the oil was also mixed according to adsorbent to oil ratio of 1:2 of Al_2O_3. The mixture of base oil and sludge was heated up to 250^oC and centrifuged for about 1 h. After centrifugation, the oil was then filtered to obtain the final oil. The centrifuged oils were to be transferred to beakers with conical flasks and filtered out any remaining residue. The filtrate was the reclaimed oil after the treatment process.

Characterization of ULO

The analysis of oxidative compounds of the oil was done using the FTIR. Viscosity was tested with viscometer (Brookfield Programmable Viscometer DV-III + Rheometer) using a S64 spindle. The oxidative properties were compared between used and treated oil. The weight and volume of the oil samples for both before and after the treatments were noted. The mass of sludge was obtained by measuring the net weight of sludge that was collected after the acid treatment.

RESULTS AND DISCUSSION

Specific gravity

Specific gravity was measured by the ASTM D941-55 and was calculated as a ratio of density of oil to density of water (equation 1).

$$\textit{Specific gravity} = \textit{Density of oil, g/ml} \,/\, \textit{Density of water, g/ml} \quad (1)$$

Figure 1 shows the specific gravity comparison for oil samples. As Figure 1 shows, all the oil samples have a reduction in the specific gravity. Sample 4 had the most significant difference in the specific gravity followed by sample 3, sample 2 and finally sample 1. Since both samples 1 and 3 have been treated with Al_2O_3, sample 3 has the bigger difference in the specific gravity. However, it can be said that the entire oil sample has a significant improvement in terms of the specific gravity. This indicates that some pollutant has been removed and the oil has reduced density (Udonne, 2011). In terms of the adsorbents, RS has shown more improvement in the specific gravity compared to Al_2O_3.

Mass of Sludge

After the acid treatment, the oil produces a certain amount of sludge due to the reaction that takes place (Hamawand *et al.*, 2013). From this mass of sludge, the resultant removal of impurities can be gauged. The sludge removal is shown in Figure 2. As Figure 2 shows, the oil sample that has the highest percentage of sludge removal is sample 3 and the lowest sludge removal was for sample 4. This means that the impurities absorbance of acetic acid was higher for sample 3 than that of sample 4. The sludge removal also affects the oil that was recovered. The higher the sludge removal, the lower oil can be recovered after the treatment.

Viscosity

Figure 3 shows the comparison of oil viscosity before and after treatment. It is known that the viscosity testing is able to show the indication of the impurities or contaminants in used oil (Hamawand *et al.*, 2013). Since the oil sample 1 and 2 shows that the highest viscosity, this could indicate that the ULO that has a viscosity of 180 cP has more additives and contaminants compared to used engine oil (UEO) which has a viscosity of 160 cP. The biggest difference in the viscosity is for the sample 3 which indicates the oxidized and polymerized contaminant or products in the used oil has been removed after the treatment. Since the pollutants in the oil are known to increase the viscosity, it can be concurred that pollutants exist in the oil before treatment. The products of the pollutants are oxidized and then produce precipitates which adds to the increase of viscosity. This is an indication of the oil contamination. The lowest difference in the viscosity is shown in the sample 2. This could be due to less contaminant removal or less contaminant present in the used oil itself. When comparing oil viscosity in terms of adsorbents, it can be concluded that the Al_2O_3 has shown significant improvement in the viscosity compared to the RS. Both samples 1 and 3 have a bigger percentage of difference in viscosity reduction compared to sample 2 and 4 which have been tested RS. It was found that by using Al_2O_3, 26% and 55% viscosity reduction for ULO and UEO samples, respectively. On the other hand, by using RS, 6.67% and 25% viscosity reduction for ULO and UEO samples, respectively.

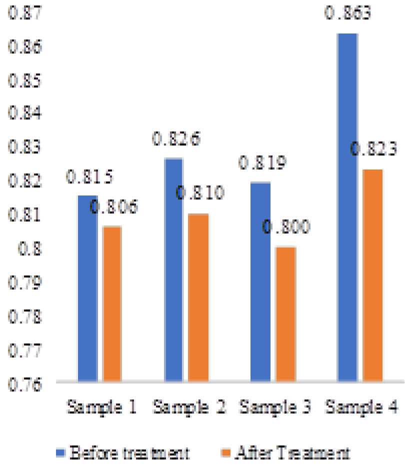

Figure 1. Specific gravity comparison for oil samples

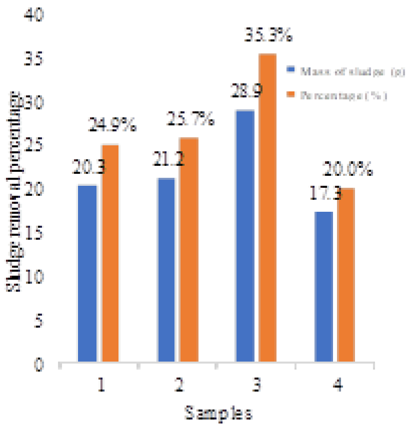

Figure 2. Mass and percentage of sludge removal of oil samples

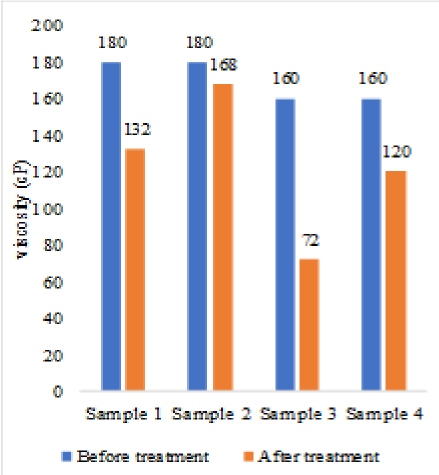

Figure 3. Comparison of oil viscosity before and after treatment

Oxidative Compounds

Figure 4 shows the FTIR analysis of ULO, ULO samples treated with Al_2O_3 (sample 1) and RS (sample 2). Based on Figure 4, it was confirmed that the oxidative compound content in the ULO sample before treatment. The major component identified was alkenes and halides. Alkenes are the common type of the oxidative compounds found in the ULO (Elbashir *et al.*, 2002). As for the alkyl halide, it could be a random compound in the oil. Alkanes found in the oil are mostly stable compounds and will not react with other compounds to create contaminants (Assunção Filho *et al.*, 2010). The silane found in the oil is a compound which has a sharp smell and found mostly in acetic acid (Kamal & Khan, 2009). It was found that the reactive oxidative compounds namely alkenes and alkyl halides were removed from sample 1 and sample 2 (Figure 4). The removal of both compounds has evidently indicated that the treatment was able to remove the oxidative compounds in the oil (Ping *et al.*, 2000). The alkanes however are a stable functional group that did not react and therefore remain in the oil after the treatment (Rincon *et al.*, 2005). The alkanes however are stable functional group that did not react and therefore remain in the oil after the treatment (Rincon *et al.*, 2005). The similarity between both the samples shows that for both treatment methods regardless of Al_2O_3 or RS has been able to produce a significant improvement. Table 1 shows the FTIR data of ULO, ULO samples treated with Al_2O_3 and RS.

Table 1. Comparison in functional group contents of ULO before and after treatment

Functional group	Wavelength (cm^{-1})		
	ULO Frequency (cm^{-1})	Treated ULO using Al_2O_3	Treated ULO using RS
RCH_2CH_3 (Alkanes)	2921.47	2921.47	2921.47
RCH_2CH_3 (Alkanes)	2853.10	2853.09	2853.09
RCH_2CH_3 (Alkanes)	1457.71	1458.32	1458.32
RCH_2CH_3 (Alkanes)	1376.92	1376.91	1376.91
RCH=CHR (Alkenes)	974.62	Not detected	Not detected
RCH_2CH_3 (Alkanes)	721.46	721.68	721.68
R-Br (Alkyl halides)	570.68	Not detected	Not detected

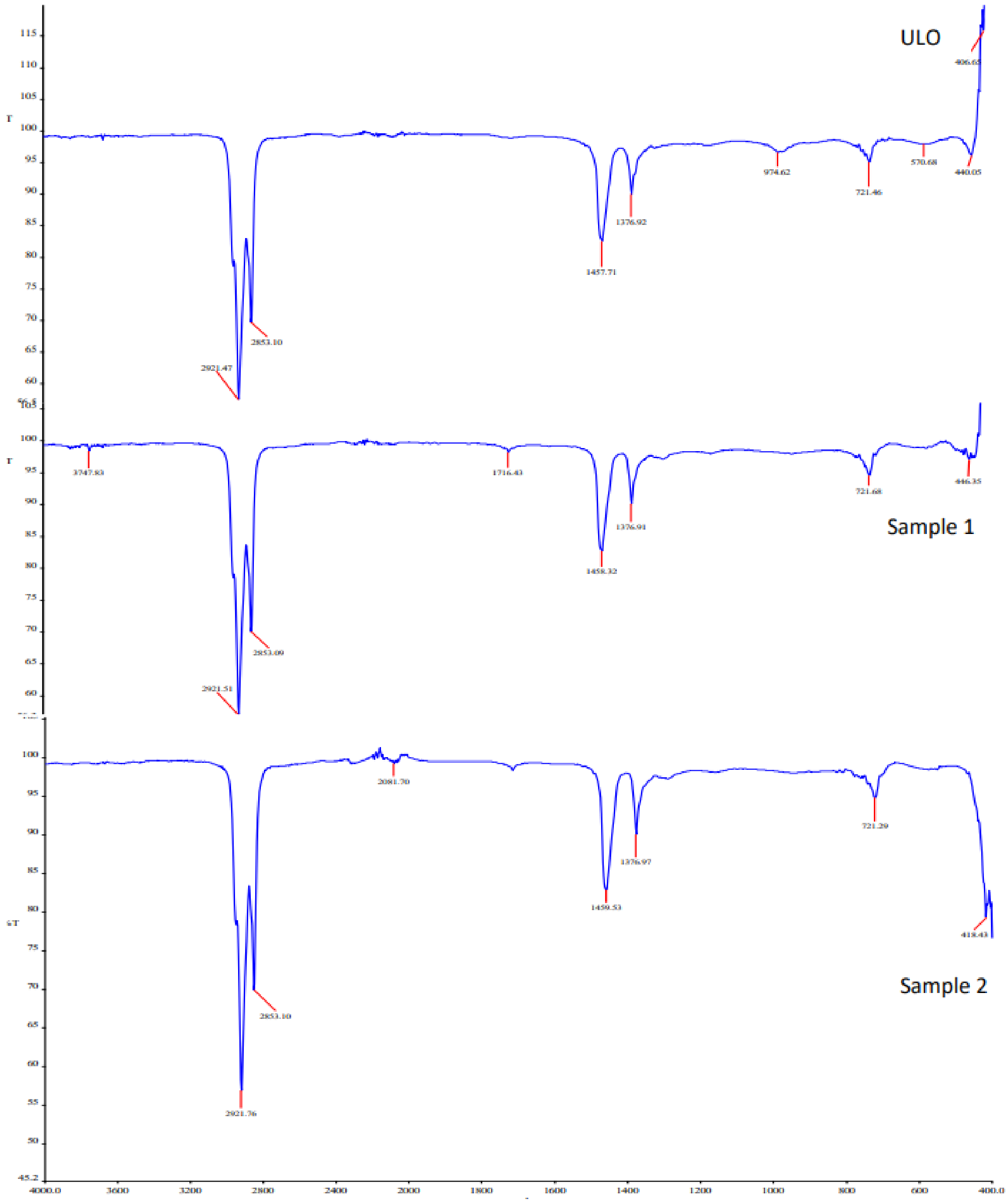

Figure 4: FTIR analysis of ULO, Sample 1 and Sample 2

CONCLUSION

Based on the results obtained, the Al_2O_3 seems to be a better adsorbent than RS in several tests such as the density, sludge removal and viscosity. However, the RS does also give significant changes in the oil analysis and has a good potential to be an economical, organic and cheap adsorbent. Overall, both adsorbents were able to show significant results that improved the quality of oil. For better viscosity and mass of sludge values, the Al_2O_3 is more suitable compared to the RS. On the other hand, for the better specific gravity value, the RS provides the better oil quality. For the oxidative compounds, both adsorbents were performed to remove the oxidative compounds well. However, to meet the strict standards of ASTM, the oil has to be further improved in terms of color improvement and metal content.

ACKNOWLEDGEMENTS

The authors pay their sincere gratitude to Universiti Malaysia Sabah (UMS) for providing necessary research facilities to accomplish this study.

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