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TREATMENT, CHARACTERIZATION, AND RECYCLING OF USED SYNTHETIC OIL

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ABSTRACT
The study evaluates the treatment for reuse of used synthetic oil. Three solvents: n-butanol, n-Hexane, and the blended solvent of n-Hexane and n- butanol were considered. Each of the solvents was used to reclaim the base oil from the sludge at different extraction factors considered. The results of the properties of the samples of treated used synthetic oil: n-Hexane Light Oil (HLO), n-Hexane with Heavy Duty Oil (HHO), n-Hexane with Blended Oil (HBO), Butanol Light Oil (BLO), Butanol Heavy Duty Oil (BHO), Butanol with Blended Oil (BBO), n-Hexane/Butanol - Light Oil (HBLO), n-Hexane/Butanol-Heavy Duty Oil (HBHO), n-Hexane/Butanol - Blended Oil (HBBO), Light Oil Control (L-Ctrl), Heavy Oil Control (H-Ctrl), Blended Oil Control(HB-Ctrl) obtained are in the range: Flash point: 157 – 178 ^{o}C. Pour point: -16 - -27^{o}C. Total acid values (TAV): 0.20 - 0.26 mgKOH/g Oil. Densities: 0.8315 - 0.9623 g/cm^3. Cloud Point:-10 -14 ^{o}C. The flash point is found below the Society of Automobile Engineers (SAE) specifications of new engine oil which is typically 204 ^{o}C. Pour points results for the treated oils are higher than the results specified for the control, lube oil SAE 20, 30, and 40 which are −10, −10 and -12 ^{o}C respectively. The treated oil density results fall within the range of the specified SAE values (i.e.0.7000g/cm^3 - 0.9500g/cm^3). To ensure proper fluidity at low temperatures, the cloud point ideally should be more than 20^{o}F (10 ^{o}C). This study revealed that the mixtures with n-butanol show the best extraction performance.

Keywords: *Synthetic, Oil, Treatment, Recycling*

INTRODUCTION
Lubricating (lube) oils have a predefined useful time after which their quality degrades and therefore oil fails to provide sufficientlubricity and is therefore replaced as used oil. Currently, in Nigeria waste lube oil is used in oil furnaces, wood treatment plants, loosening rusted bolts, and in preventing rusting in a different metal (Onuigbo 2016). During the period of oil usage, it is mixed with impurities such as water, dirt, and metal scrapings. Also the degradation of the lubricating oil additives tends to introduce some contaminants and hence, the lubricating oil must be replaced. Once replaced; the oil is termed as used oil and requires proper management to avoid environmental pollution associated with the disposal of used oil (Pinheiro, *et al.* 2020). The burning of these used oils has significant effects on the environment as these pollutants are released into the atmosphere after combustion.

Literature review revealed that indiscriminate disposal of waste engine oils in the environment cause pollution of grievous consequence (Altaee, *et al*., 2022, Botas, et al., 2017, Fong Silva, et al., 2017, Keloth, et al., 2021, Sánchez-Alvarracín, et al., 2021). Recycling such contaminated oil will be beneficial in reducing the cost of importing base lube oil as well as the consequence of environmental pollution (Hameed, (2021). This has led to the development of techniques both to reduce the environmental hazard associated with either indiscriminate disposal or burning of the oil, and to conserve valuable used oil resources that can be recycled into base lube oil. Although there

are many technical ways in which these used lubricant oil can be recycled but currently in Nigeria, the waste oil is not collected for recycling purposes. In Nigeria, 50million liters per annum of lubricating oils are disposed annually which cost an enormous amount of money to produce (Boadu, *et al.*, 2019)**.** Thus, when discharged into the environment or even burnt as fuel, causes severe pollution problems (Osman *et al.*, 2018). The way forward, therefore, is the recycling of the waste oil so that the oil can be used again as a base stock.

Lubricating oil is commonly used for the reduction of friction, wear and heat between mechanical parts that are in contact with each other, as well as motor oil and transmission fluid in vehicles. Engine oil is the main active lubricants in automobiles that function to keep the engine cool and ensures proper sealing of the piston rings in the automobile engine (Adewole, *et al.*, 2019). Therefore, the study is to treat used engine and lubricating oil for recycling.

MATERIALS AND METHODS

Used engine oils were obtained from the Mechanic Village, Auchi, Edo State, Nigeria. N-butanol, N-hexane, Potassium Hydroxide, were products of Sigma-Aldrich, Germany and activated charcoal, Loba Chemie PVT. LTD, India.

Sample Collection

Five (5) Litres of used heavy-duty and light-duty motor oils were collected from a mechanic site service station in Auchi, Edo State, Nigeria. Each of the oils had been used for one month by the motor and Siena car before collection.

Modification and Preparation of Sample

100 ml each of the sample oil was poured into two different beakers, labeled as E_1, and E_2. Also, another 50 ml of each sample oil of both heavy-duty oil and light-duty oil was poured into a third beaker labeled E_3 (Blended oil). The labelled Samples, E_1, E_2 and E_3 were characterized using standard methods for specific gravity, pour point, flash point, and kinematic viscosity at 40 ^oC and 100 ^oC.

Table 1: Some Standard lubricating oil testing methods.

S/N	Test	Designation	Apparatus
1	Kinematic Viscosity	ASTM D-445	Viscometer
2	Pour Point	ASTM D-97	Pour Point tester
3	Specific gravity	ASTM D-1298	Density bottle
4	Flash Point	ASTM D-97	Flash Pont tester

Characterization

The flash points of the samples were analyzed by ASTM D97 using the flash apparatus. The pour point indicates the temperature below which the oil loses its ability to flow or circulate in the lubricating system. The specific gravity was determined using the dry pycnometer (ASTM D941-55). This procedure was also repeated for other samples, in triplicates, and an average value of the samples was taken as the result for the samples.



Figure 1: Pycnometer on a weighing balance

The density of the oil was determined using the equation:

Specific gravity = $\frac{W_1 - W}{W_2 - W_1}$

Density = Specific gravity × 1.0g/cm^3

Where W_1 is the weight of the empty pycnometer, W_2 is the weight of empty pycnometer and water, and W_3 is the weight of the empty pycnometer and oil sample.

Fourier Transform Infra-red (FTIR): The infrared (IR) spectrum of the samples were carried out using a Perkin-Elmer model RX1 Fourier transform infrared (FT-IR) spectrometer (thane, Mumbai, India). Sample tablets were prepared by mixing each sample with potassium bromide (the ratio of sample to KBr was 1:100) before analysis. The spectra were recorded within the frequency range of 4000 cm^{-1} to 500 cm^{-1}.



Figure 2: FTIR Machine

Viscosity (ASTM D445)

Viscosity was determined, according to the ASTM D445 procedure. The programmable rheometer shown in Figure 3 was used in carrying out this analysis. The rheometer comprises a spindle, stainless cup and a rotor. The sample was preheated to 60^{o}C and was poured into the stainless cup of the DV-III –ultra – programmable rheometer (Brookfield: model DV III Ultra). (Zgheib, et al, 2022).



Figure 3: The Programmable Rheometer

The spindle was immersed into the cup and the rheometer was set to a speed of 100 rpm. Dynamic viscosity was immediately recorded when the temperature dropped to 40^{o}c

.Cloud Point was determined using the method described by Tariq, et al (2022), and Parekh, et al., (2022) using a control digital thermometer (Model D1227) the procedure was repeated for other samples). The acid number was determined by consistent titrating of the solution of the weighed amount of the sample in the solvent containing the

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indicator. The total acid value determined using the formula:

$$\text{Total Acid Value (TAV)} = \frac{(V_1 - V_2) \times C \times 56.1}{m}$$

Where, V_1 is the volume of alcoholic potassium hydroxide solution with $C_{(KOH)}$ = 0.1mol/L, V_2 is the volume of alcoholic potassium hydroxide solution with $C_{(KOH)}$ = 0.1mol/L, used for titration of the blank sample; C is the molar concentration of KOH and m is the mass of the sample taken (g).

Treatment procedure

The oil samples were allowed to settle at room temperature for 24 h and filtered to remove solid particles, then water was added to the oil. Solvent extraction treatment was done with the use of butan-1-ol and n-Hexane, potassium hydroxide (KOH), and activated charcoal. 100 ml each of the oil samples were mixed with n-butanol, n-hexane and a composite solvent (60% of butanol and 40% of n-hexane) respectively with 5 g of potassium hydroxide (KOH).

The mixture was stirred for 30 min, while heating on a heating mantle at 60°C and at atmospheric pressure to remove the light hydrocarbons. The mixture was then allowed to cool and settle in a separation flask for 24 h. The mixture samples E_1, E_2, and E_3 were separated from the sludge and then heated at 120°C to remove the solvent in the mixture. The oil obtained from the oil solvent mixture was mixed with 4g of activated charcoal and heated at 60°C for 1 h. The heated mixture was then left for 2 h to settle. It was then filtered with filter paper to recover base oil from the mixture. Figures 4a and 4b: show the used and treated oils respectively.



Figure 4a: Used engine oil



Figure 4b: Refined engine oil

RESULTS AND DISCUSSION

Table 2: The physiochemical properties of the reclaimed engine oil

Samples	Properties				
	Density (g/cm^3)	Pour point(^{0}C)	Cloud point(^{0}C)	Flash point(^{0}C)	Total Acid number(mg KOH/g)
L-Ctrl	0.8370	-27	-14	163	0.26
HLO	0.8341	-24	-16	157	0.23
HHO	0.9442	-18	-10	170	0.22
HBO	0.8315	-20	-12	176	0.26

H-Ctrl	0.9623	-16	-12	174	0.21
BLO	0.8616	-25	-14	168	0.24
BHO	0.9441	-16	-12	175	0.23
BBO	0.8909	-18	-10	178	0.20
HB-Ctrl	0.8852	-22	-14	165	0.21
HBLO	0.8800	-21	-13	167	0.23
HBHO	0.9429	-17	-11	164	0.23
HBBO	0.8747	-20	-12	168	0.21

Cloud Point

The cloud point results obtained for the reclaimed oil when compared to the used oil values (Table 2) showed that the cloud point results are relatively close to the paper machine oil (PMO) value of -150C (Huey, et al., 2022). The temperature at which certain hydrocarbons (Paraffin) began to convert into crystalline form is called cloud point. To ensure proper fluidity at low temperatures, the pour point ideally should be more than 20^0F (10^0C).This corroborates the earlier assertion by Hameed, (2021) and Sánchez-Alvarracín, et al., (2021).

Total Acid Value (TAV) Analysis

Total acid number (TAV) were obtained for the three treated oil sample E_1, E_2 and E_3 respectively (Table 2). These results reveal that the acid concentration in the treated oil is low and thus, removes the cause of friction and corrosion in the automotive engines. The standardization of the acid value of engine oil according to EN 14104: 2003, is 0.5 mg KOH/g, which also relates to ASTM D6751. The values of treated oil E_1 and E_3 were found within the range specified for general internal combustion engines (Jose, *et al*., 2020).

Specific Gravity and Density

Densities were obtained for the reclaimed oil respectively compared to the value of 0.8370, 0.9623 and 0.8800 for the used oil E_1 (L-Ctrl), E_2 (H-Ctrl) and E_3 (HB-Ctrl) (Table 2). The results corroborate the outcome of the earlier work of Hameed, (2021) in which the density of fresh engine oil was 0.90, while the density of used engine oil was 0.93. The reclaimed oil values however are within the range of the specified SAE values which fall within the range of 0.7000g/cm^3 to 0.9500g/cm^3. However, it could be lower or higher than fresh engine oil depending on the nature and type of contaminations (Jose, *et al.*, 2020).

Flash Point Analysis

The flashpoint values obtained for the reclaimed oil samples are higher than those of used oil as shown in Table 2. Flashpoint values obtained for treated oil samples respectively were compared to the values of the used oil. Flashpoint indicates the presence of volatile compounds in the oil. It also gives a relative measure of the safety properties of lubricating oils. The flash point values: 176, 175, and 178^{o}C were lower when compared to the flash point of virgin engine oil which is typically 204 ^{o}C. The low flash point observed in the used oil is an indication that the oil is contaminated with unburned fuel (Hameed, 2021 and Sánchez-Alvarracín, *et a*l., 2021).

Kinematic viscosity

Table 3: Kinematic Viscosity of samples

Samples	Viscosity (cP)		
	1	2	3
L-Ctrl	2.75	2.77	2.74
HLO	2.69	2.70	2.70
HHO	2.60	2.58	2.62
HBO	2.80	2.84	2.83

H-Ctrl	2.85	2.83	2.86
BLO	2.73	2.72	2.72
BHO	2.68	2.65	2.60
BBO	2.82	2.83	2.82
HB-Ctrl	2.70	2.67	2.68
HBLO	2.66	2.64	2.66
HBHO	2.62	2.60	2.59
HBBO	2.64	2.70	2.69

The kinematic viscosity at 40^{o}C for L-Ctrl 1, 2, and 3 are shown in Table 3. For L-Ctrl 1, 2, and 3, HBO values appear to be higher than the control oil (L-Ctrl). For H-Ctrl: 1, 2, and 3, the sample's viscosity values are lower than the control oil (H-Ctrl). For HB-Ctrl: 1, 2, and 3, HBBO Sample viscosity values appear to be higher than the control (HB-Ctrl) which indicates higher viscosity. This result corroborates the work of Hameed, (2021) who studied the characterization and suitability of treated automotive lubricating oils reprocessed by solvent extraction technology. A higher viscosity value of the treated oil would enhance its lubricating performance as virgin oil for use in automotive engines (Hameed, (2021). Kinetic viscosity of used engine oil can be increased due to oxidation or contamination, while it may also be decreased due to dilution with light fuels (diesel or petrol) (Huey, et al., 2022). Similar study shows that the viscosity of fresh lube oil is 90 cP, used engine oil 120 cP, which shows presence of contaminations in used engine oil, where the viscosity values of refined engine oils by composite solvent method, single solvent method, and acid treatment method are 94 cp, 98 cP, and 92 cP, respectively (Jose, *et al.*, 2020, Hameed, 2021).

Pour point

Higher pour point values were also obtained for the reclaimed oil samples in comparison with values obtained for the used oils. Pour points were obtained for the reclaimed oil samples respectively compared to the values for the used oil. The Pour point indicates the temperature below which the oil loses its fluidity and will not flow or circulate in the lubricating system. Pour points value for the reclaimed oils are far higher than the values specified for lube oil for SAE20, 30, and 40 which are −10, −10, and−12◦C respectively. Pour point of lube oil may be decreased and increased, depending on the nature of lube oil method of refining (Huey, *et al.*, 2022). The reclaimed oil, however, is very far higher compared to the specified SAE values; this can be attributed to the continuous usage at varying temperatures resulting in a loss in fluidity as asserted by similar works (Jose, *et al*., 2020, Hameed, 2021).

pH Measurement

pH is a measure of the hydrogen ion (H^{+}) concentration in an aqueous solution. In the case of fresh engine oil, the pH is 10.0, and pH 9.0 to 11.0 is appropriate (Bhattacharya, *et al.*, 2015). When it falls below pH 9.0, deterioration starts. The lower the pH value is, the more degradation progresses. The various pH results obtained for the various samples of the reclaimed oil and used oil are shown in Table 4.

Base oils for the production of lubricating oils contain neither acids nor alkaline components. The pH value of these base oils, to which no active ingredients have has been added, is pH = 7. The base oils therefore are completely neutral. In lubricants production, additives are added to the base oils, which are slightly "acidic". Especially additives to improve wear or corrosion protection are based on slightly acidic compounds. This could have accounted for the results as shown in Table 4. Even engine oils whose additives have alkaline effect already have some acid-reacting components as fresh oil, (Hameed, 2021, Sánchez-Alvarracín, *et al.*, 2021). These production-related acid components are harmless. However, negative effects occur when the proportion of acids in the oil increases.

There are several reasons for this: The acid load of engine oils increases when in use, due to combustion

products. "Blow-by" gases enter the oil circuit. Sulfur, which stems from the fuel, adds further acidity to the oil. They all have to be neutralized and rendered harmless by the engine oils. This means that the base number of an oil in internal combustion engines, also known as the alkaline reserve, decreases during operation. However, no acidic combustion products contaminate the oil in hydraulic oils and industrial lubricants, such lubricants also become acidic through oxidation, (Hameed, 2021, Sánchez-Alvarracín, *et al.* 2021).

Table 4: Results obtained for the pH test

S/N	Solvents	pH
1	Recycled oils with n-hexane	
	Light oil	8.43
	Heavy oil	8.40
	Blended oils	8.46
2	**Recycled oils with n-butanol**	
	Light oil	10.03
	Heavy oil	9.73
	Blended oils	10.40
3	**Recycled oils with blended solvents**	
	Light oil	9.91
	Heavy oil	8.72
	Blended oils	8.65
4	**Used engine oils**	
	Light oil	1
	Heavy oil	1
	Blended oils	5

Results of the FTIR Analysis

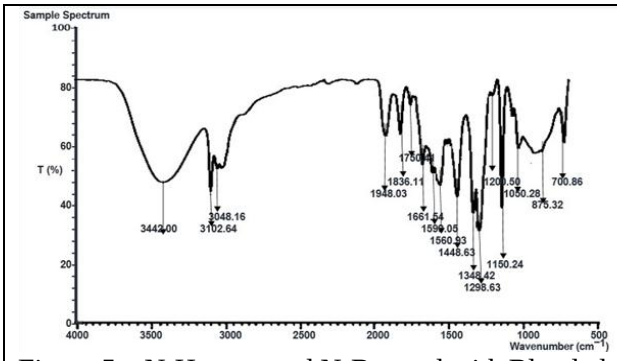


Figure 5a: N-Hexane and N-Butanol with Blended oil (HBBO)

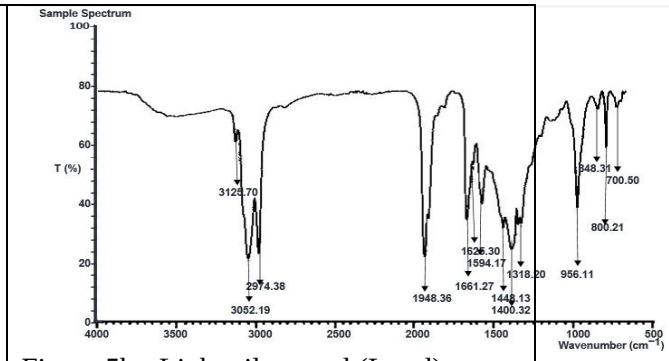


Figure 5b: Light oil control (L-ctrl)

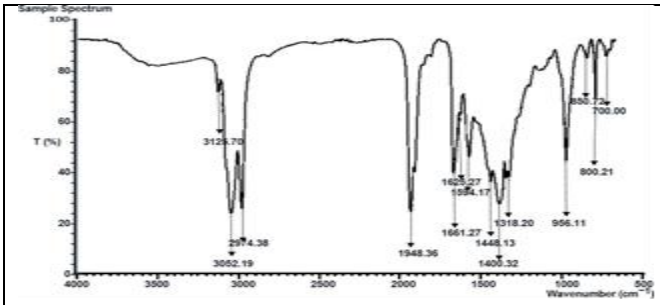


Figure 5c: Light oil refined with N-Hexane (HLO)

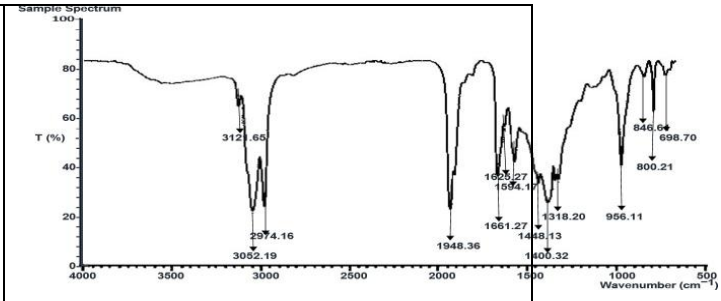


Figure 5d: Blended oil with N-Hexane (HBO)

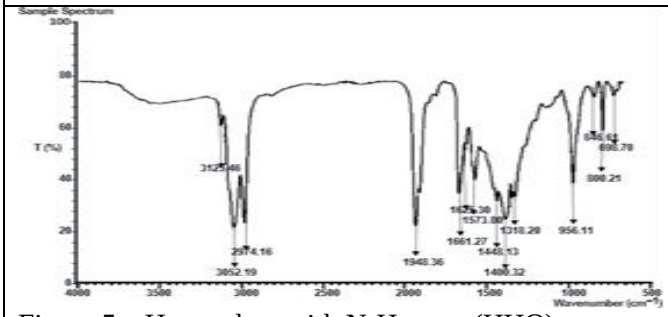


Figure 5e: Heavy duty with N-Hexane (HHO)

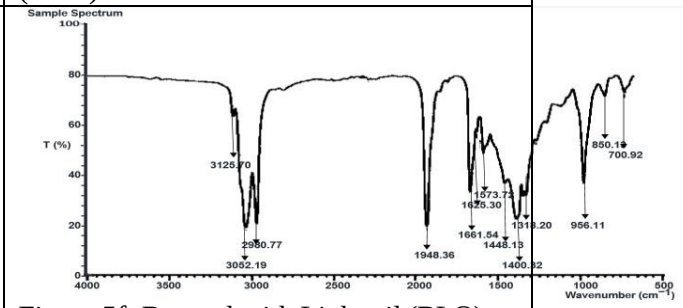


Figure 5f: Butanol with Light oil (BLO)

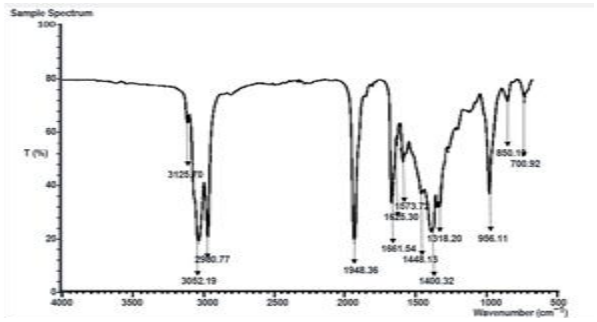


Figure 5g: Butanol with heavy -duty oil (BBO)

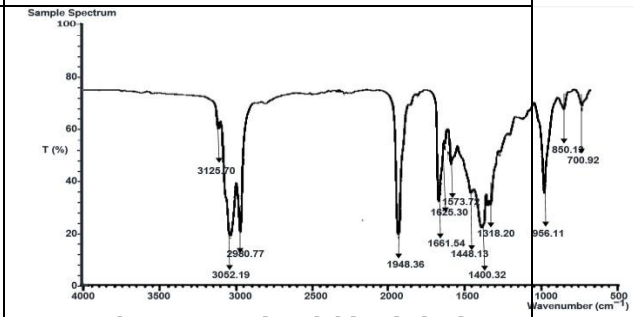


Figure 5h: N-Butanol with blended oil (H-ctrl)

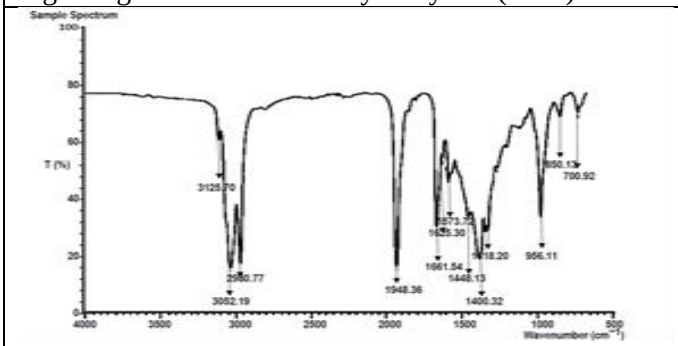


Figure 5i: N-butanol with heavy- duty oil (BHO)

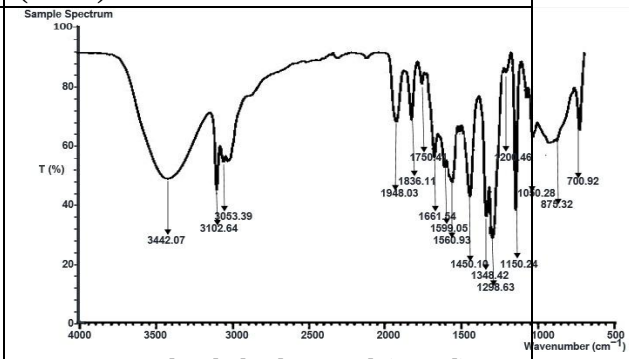


Figure 5j: Blended oil control (B-ctrl)

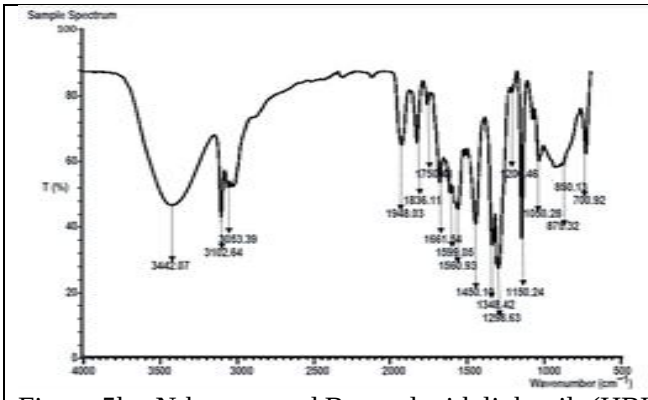


Figure 5k: N-hexane and Butanol with light oil (HBLO)

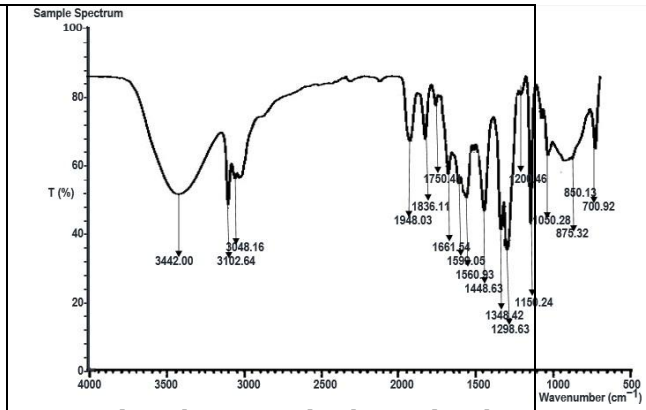


Figure 5l: N-hexane and N-butanol with heavy duty (HBHO)

Fourier transform infrared (FTIR) spectroscopy is a versatile tool that is used to detect common contaminants, lube degradation by-products and additives within lubricating oils. It has become a widely used technique for quickly assessing multiple lubricant characteristics.

The FTIR results **a**s can be seen from Fig. 5a to Fig. 5i with strong intensity of the stretching alkene peak at 3125.46 cm^{-1} and 3052.19cm^{-1}(between the range of 3020 to 3140cm^{-1}) indicate =C-H band while those for Fig. 10 to 13 occur at 3442.00 and 3102.64cm^{-1} within the range of 3000-3500cm^{-1}. Band within 3000-3500cm^{-1} in the reclaimed oil under HB- Ctrl and is due to bending and stretching vibration of O-H bonds which are a result of the presence of water molecules according to a similar research work that was carried out by Jose, *et. al.*, (2020) and Doaa, *et al.*, (2018).

To minimize the effects of the base oil and additive molecular resonances, FTIR analysis of used oil samples is in three-stages. 1. The FTIR spectrum record of a new oil sample was secured to obtain a baseline FTIR trace. 2. The same record of the same FTIR spectrum of the used oil sample was obtained. 3. Subtraction of the new oil baseline, often referred to as the new oil reference, from the used oil spectrum to obtain the difference spectrum. The difference in spectrum allows the changes in both the chemical composition of the oil, such as oil oxidation as represented by an increase in a peak centered around 1740 cm^{-1}, (Figure 5) and any contaminants to be measured without interference from the new oil molecular resonances (Jose, *et. al*., 2020, and Doaa, 2018).

CONCLUSION

The study presents the use of solvent extraction method for the treatment of used engine oils and evaluates its suitability for reuse in automotive engines. Assessment and comparison of the properties of the treated oil samples with SAE specifications for lube oils show that the flash point of the treated oil is lower than the value specified for SAE20, SAE30, and SAE40. The Pour Point of the treated oil is below the standard values while the specific gravity was in the range of the SAE standards. The viscosity of the treated oils for H-CtrL:1, 2, and 3, the sample's Viscosity values are lower than the control oil (H-CtrL) which indicates less viscous. For HB-CtrL:1,2, and HBBO sample Viscosity values appear to be higher than the control (HB-CtrL) which indicates higher viscosity. Total acid indicates the acid concentration in the treated oil is very low and thus, eliminates the cause of friction and corrosion in the automotive engines.

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