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Research Article

Assessment of the Conventional Acid-Clay Method in Reclaiming Waste Crankcase Lubricating Oil

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In this study, the conventional acid-clay method was used to evaluate its potential for recycling waste crankcase lubricating oil (WCLO). The results showed that the acid-clay method was effective in re-refining the WCLO and returning the oil to a quality comparable to oils produced from fresh lube oil stocks. This method has been reported to account for around 90% of the global waste crankcase lubricating oil treatment and is considered to be an inexpensive process. The results revealed that the acid-clay method improved the viscosity of the oil at 40°C from 104 cSt to 105.6 cSt. The flash point of the oil was also increased from 192°C in the WCLO to 204°C in the re-refined crankcase lubricating oil (RCLO). The water content reduced from 0.01% in the WCLO to 0% in the RCLO, indicating the effectiveness of the acid-clay method in removing water traces from the WCLO. Additionally, the contaminants present in the WCLO were reduced drastically, with iron and aluminum content reduced from 23.0% and 21.0% to 0.0% and 0.0%, respectively. The fuel ingress in the WCLO reduced from 4.0% to 1.0%. However, it was observed that the acid-clay method did not significantly impact the viscosity index, TBN, and density of the oil. The re-refined base oil produced by the acid-clay method can be fortified with appropriate additives and reused in vehicles, reducing environmental pollution, depleting fossil resources, and saving the country's foreign exchange used in importing fresh lubricating oil.

1. Introduction

The global demand for lubricant (oil and grease) is estimated at 40 million metric tons with an annual increase of 200,000 metric tons. Approximately, 50% of all lubricants produced globally are used in the transport sector. Assuming 0.5% of crankcase lubricating oil is lost to engine blow-by during engine operation, over 19.9 million metric tons of waste crankcase lubricating oil (WCLO) is generated every year. Out of the total WCLO generated globally, less than 45% is available for collection. The remaining 55% is either reused or inappropriately released into the environment, which causes pollution if not properly disposed of.

According to reference [1], the National Petroleum Authority of Ghana and the Tema Lube Oil Company Limited estimated that the amount of lubricating oil consumed in Ghana (in 2013) was 45 million litres, totalling approximately US$750 million. This amount of lubricating oil accounted for a sizable portion of the country's gross domestic product (G.D.P.); therefore, its re-refining will not only curb the environmental issues that arise as a result of the indiscriminate disposal of the waste lubricating oil but would also impact the economics of a developing country such as Ghana.

Crankcase lubricating oil is composed of 75–85% base oil and the rest is additives. As a result, the indiscriminate dumping of the waste crankcase lubricating oil represents a loss of valuable natural resources, and many waste lubricating oil management processes have been developed to address this issue of indiscriminate disposal [1–4]. Also, according to a report issued by reference [5], it takes 67 litres of crude oil to produce one litre of crankcase lubricating oil, whereas it only takes 1.6 litres of waste crankcase lubricating oil to produce the same amount of crankcase lubricating oil.

As a result, the waste crankcase lubricating oil management process prioritizes recycling over recovery and disposal. A common recycling process in waste crankcase lubricating oil recycling is re-refining. Re-refining is considered a preferred option in resource conservation, after waste reduction, and avoidance of environmental pollution [6].

Some researchers have assessed the various methods for re-refining waste crankcase lubricating oil where the pros and cons of conventional approaches to re-refining waste crankcase lubricating oil such as acid-clay, solvent extraction, vacuum distillation, and modern techniques such as membrane technology, catalytic process, Vaxon process, chemical engineering partners process (CEP), EcoHuile process, and cyclone process among others are discussed [7, 8]. Although a review [7] recommended a combination of advanced methods such as membrane technology and pyrolysis as the most environmentally friendly techniques, those technologies are not available in many underdeveloped and developing countries and those that are available are beyond the financial reach of many Ghanaian businessmen and women. However, the acid-clay method is less expensive, more effective, and easier to operate on a small to medium scale. Therefore, from an economic standpoint, the conventional acid-clay method is most appropriate among the waste crankcase lubricating oil re-refining methods. It is also worth noting that the major waste (the sludge) generated from this process is in high demand because it can be mixed with bitumen in road construction to improve mechanical properties, moisture resistance, and avoidance of stiffness [9, 10].

Figure 1 indicates a setup for a small to medium-scale acid-clay method used to reclaim waste crankcase lubricating oil reported by [12]. The setup appears to be robust and straightforward and can be adopted in most light industrial setups in developing countries such as Ghana.

The base oil properties are determined by parameters such as density or specific gravity, viscosity, viscosity index [13, 14], flash point [15, 16], and pour point. Water, sulphur, metal, and ash content [17] are also important parameters to consider as well as neutralization number [3, 14, 17], oxidation stability [17], and colour [17].

Considering the amount of foreign currency spent in importing base oil into the country, and the amount of waste crankcase lubricating oil generated and unaccounted for in Ghana, any drive toward an effective WCLO management programme will be highly commendable.

Hence, this study explores the potential of the acid-clay method in re-refining waste crankcase lubricating oil in a developing country such as Ghana.

2. Materials and Methods

An experimental approach was employed in this study. Three different crankcase lubricating oils (fresh crankcase lubricating oil (FCLO), waste crankcase lubricating oil (WCLO) which was generated after running the FCLO for 5000 km in a RAV 4 Toyota vehicle, and re-refined waste crankcase lubricating oil (RCLO)) were subjected to standard laboratory tests and the results were compared.

2.1. Materials. The primary materials used for this project were 5 litres of WCLO Total quartz 15W40, 98 wt.% sulphuric acid, activated clay, and lime. The distillation and filtration setups were used. Sulphuric acid was used as a solvent to dissolve impurities in the WCLO due to its ability to dissolve a wide range of impurities. The activated clay was used to improve the colour of the re-refined oil and the lime was used to neutralize the acid after the addition of the sulphuric acid.

2.2. Methods. The processes were grouped into re-refining processes and analytical processes. The re-refining processes included filtration, dehydration, mixing and agitating the oil-acid mixture, decanting and separation, and distilling. The analytical processes were used to characterize the oil samples taken from FCLO, WCLO, and RCLO.

In the re-refinery process, WCLO was first filtered to remove solid particles and then dehydrated by heating at 110°C. Sulphuric acid with 98% concentration was added to the dehydrated WCLO to remove metallic salts, acids, aromatics, and other impurities. The sludge formed was removed after 24 hours. The acidic oil was mixed with clay to remove contaminants such as mercaptans and to improve the oil colour. The oil was then neutralized with lime.

Figures 2 and 3 show the experimental setup used in the dehydration and sludge separation from the WCLO re-refining process, respectively.

The resulting lubricating oil was analyzed and the results were compared with that of the virgin crankcase lubricating oil and the waste crankcase lubricating oil. The efficacy of the purification process was determined from the laboratory analysis. The properties analyzed included density or specific gravity, viscosity and viscosity index, flash point, neutralization number, water content, and wear metal particles. The various test methods used in the analytical process are shown in Table 1.

2.3. Density. The densities of the three oil samples were calculated at 30°C by employing the density formula, i.e., density = mass/volume. An oil sample was poured into a density bottle with a known mass and volume. The mass of the oil and the bottle was noted. The mass of the oil was calculated at the known volume of the bottle. With the known mass and volume, the density was calculated as follows:

$$\begin{aligned}\text{Mass of empty density bottle} &= X_1 g,\\ \text{Mass of oil and density bottle} &= X_2 g,\\ \text{Mass of oil only} &= (X_2 - X_1) g,\\ \text{Volume of oil} &= V\text{cm}^3,\\ \text{Density} &= \frac{\text{Mass of oil only}}{\text{Volume of oil}} = \frac{(X_2 - X_1)\, g}{V\text{cm}^3}.\end{aligned} \tag{1}$$

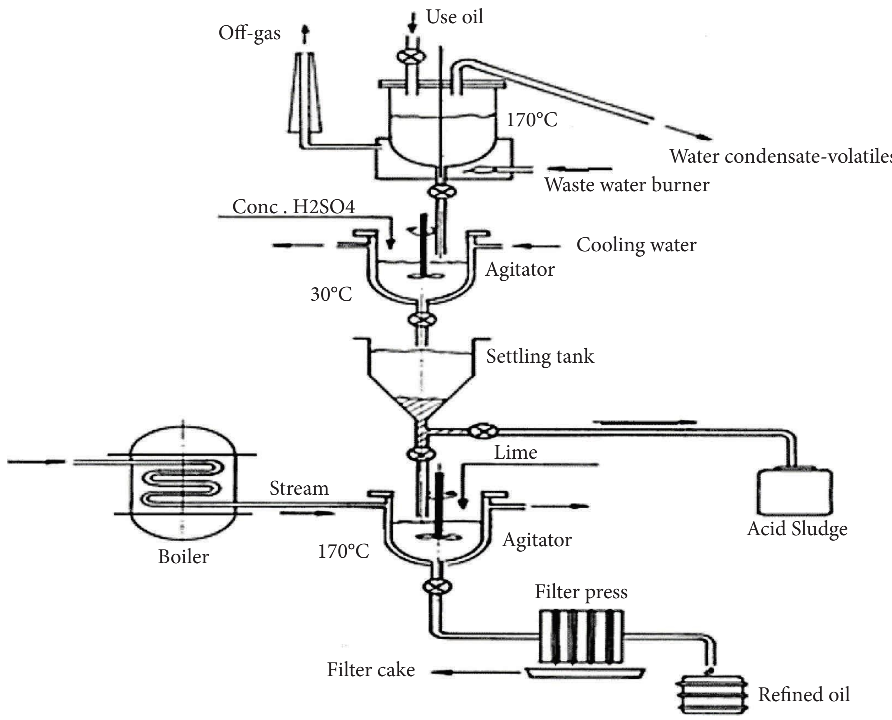


Figure 1: Small-scale acid-clay process. Source: [11, 12].

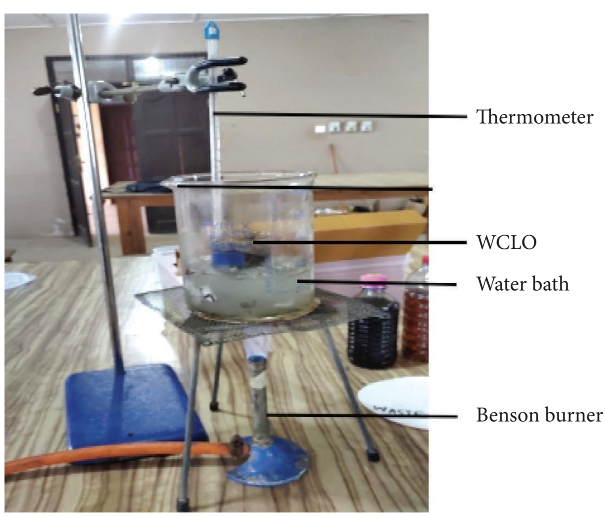


Figure 2: Dehydration of WCLO.

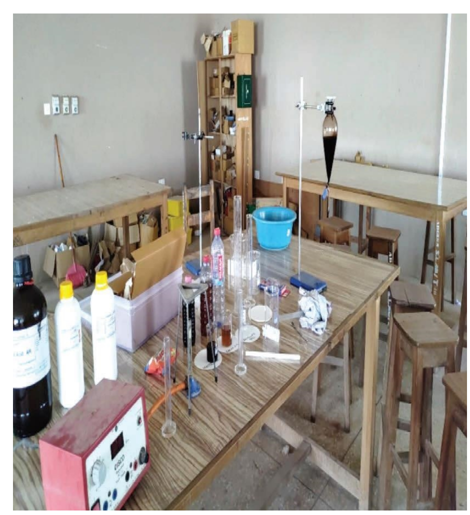


Figure 3: Laboratory apparatus for WCLO re-refining.

2.4. Kinematic Viscosity and Viscosity Index. Kinematic viscosity is the commonest and most important property of lubricating oil. It is determined by measuring the time taken for a fixed volume of lubricating oil to flow through the capillary tube of the viscometer under gravity. First, the oil sample was drawn into the viscometer tube by suction. The tube was then immersed in a constant temperature bath. The temperature was raised to 40°C and then 100°C. The time for the oil to flow from the start to stop mark on the viscometer tube was measured in seconds at each temperature. The kinematic viscosity at each temperature was calculated by multiplying the time in each case by the tube calibration number (constant).

The viscosity index (VI) is a number which expresses the rate of change of viscosity of oil with temperature. It is determined from the viscosities measured at 40°C and 100°C for the unknown oil; Y = kinematic viscosity at 100°C and U = kinematic viscosity at 40°C. Two reference oils are required, one with a VI of 100 and the other with a VI of 0. Both oils must have the same viscosity as that of the oil whose VI is being determined at a temperature of 100°C. VI is then calculated by using the following relation:

Table 1: Test methods used in the analytical process.

Parameter	Method	Instrument/test kits
Kinematic viscosity	ASTM D7279	Houillon viscometer ISL VH1
Flash point	ASTM D4206	Setaflash point tester
TAN	ASTM D664	Metrohm titrator
TBN (by FTIR)	ASTM D7066	Perkin Elmer FTIR
TBN (by Metrohm)	ASTM D2896	Metrohm titrator
Water content	N/A	CaH_2 test kit
Elemental and contaminant	ASTM D6595	SpectrOil Q100

Source: Researchers' lab analysis.

$$\begin{aligned} \text{H} &= \text{Kinematic viscosity at 40°C of the oil with VI of 100,} \\ \text{L} &= \text{Kinematic viscosity at 40°C of the oil with VI of 0.} \end{aligned} \tag{2}$$

The VI of the unknown oil is then calculated as follows:

$$\text{VI} = 100\frac{(\text{L} - \text{U})}{(\text{L} - \text{H})}. \tag{3}$$

2.5. Flashpoint. Flashpoint determines the temperature at which a lubricating oil ignites into a flame when it is heated and brought in contact with a test flame. In determining the flash point of lubricating oil, a sample of oil is poured into a clean Cleveland open-cup tester up to the meniscus line. Any excess oil was removed before the test proceeded. After that, the cup was positioned on the Bunsen burner with a thermometer inserted vertically into the Cleveland cup with the help of a thermometer holder. The test flame was then lighted while the oil sample was being heated. The test flame was passed through the centre of the Cleveland cup slowly.

This process was repeated as the test oil was heated. At a particular temperature of the test sample, the ignition source burst into flames and a flash then appeared at the surface of the oil. The temperature of the oil sample at which the flash appeared was noted as the flash point.

2.6. TAN/TBN. The total acid or base number was determined through titration. The titrants used were a mixture of perchloric acid and glacial acetic acid while the analyte or titrant was crankcase lubricating oil mixed with a mixture of chlorobenzene and glacial acetic acid. The indicator was para-naphthalene. The titrant was formed by adding a sample of the crankcase lubricating to the perchloric acid and glacial acetic acid in a ratio of 2 g : 100 ml. So, 25 ml of the titrant with two drops of the indicator was titrated against 0.1 M of the titrant. A blank titration was carried out without the waste crankcase lubricating oil. The TAN/TBN was then calculated using the following relation:

$$\frac{\text{TBN}}{(\text{TAN})} = \left[\frac{(V_{s2} - V_s)x56.1xN}{W_s}\right], \tag{4}$$

where V_{s2} = the volume of titrant used for titrating a sample of engine oil, V_s = the volume of titrant used for titrating blank, N = the normality of the titrant = 0.0641, and W_s = is the weight of the sample taken for titration.

2.7. Water Content. A quantitative test was performed to determine the amount of water present in the WCLO in parts per million. The volumetric technique was used although the coulometric method could also be used. The WCLO sample was dissolved in KF methanol and the iodine was added to the KF reagent. The endpoint was detected potentiometrically and the readings on the potentiometer were calibrated to give the amount of water in the test sample.

2.8. Wear Metals and Contaminants. Metal content and contaminants in the three oil samples were measured using the Rotating Disc Electrode Atomic Emission Spectrometry. The Spectroil Q100 detected traces of suspended or dissolved particles and worn metals. This portable analyzer provided accurate and reliable results and it was fast and easy to operate. It quickly displayed the results. A sample (2 ml) of crankcase lubricating oil samples was dropped into a dish and introduced onto the Spectroil Q100 and turned on. After 30 seconds, the result was displayed in a graphical form.

3. Results and Discussion

3.1. Density of Oil Samples. Figure 4 gives the density of the three samples of crankcase lubricating oil (CLO). The density of fresh CLO was 0.87 g·cm^{-3}, that of waste CLO was 0.85 g·cm^{-3}, and that of re-refined CLO was 0.91 g·cm^{-3}. The difference in densities can be attributed to fuel ingress. Fresh CLO had no fuel ingress, and its density was 0.87 g·cm^{-3}. Waste CLO had a fuel ingress of less than 4%, and its density was 0.85 g·cm^{-3}, which was slightly lower than that of the fresh CLO. Re-refined CLO had a fuel ingress of less than 1% after the re-refining, and its density was 0.91 g·cm^{-3}, which is relatively higher than the fresh CLO with no fuel ingress [18]. The relatively high density of the re-refined CLO may be due to traces of dissolved $CaSO_4$ salt that remained in the oil after neutralization and distillation as well as finer particles of activated clay that were not filtered out due to their finer sizes. This result may be common with the acid-clay method as finer particles of clay remains in the oil after filtration and this contributes to the higher density of the re-refined oil [19]. However, the densities of all the lubricating oils fell within the density of lubricating oil (0.7–0.95 g·cm^{-3}) [20]. A similar study [21] also showed a similar pattern of the density of the RCLO being slightly higher than the WCLO.

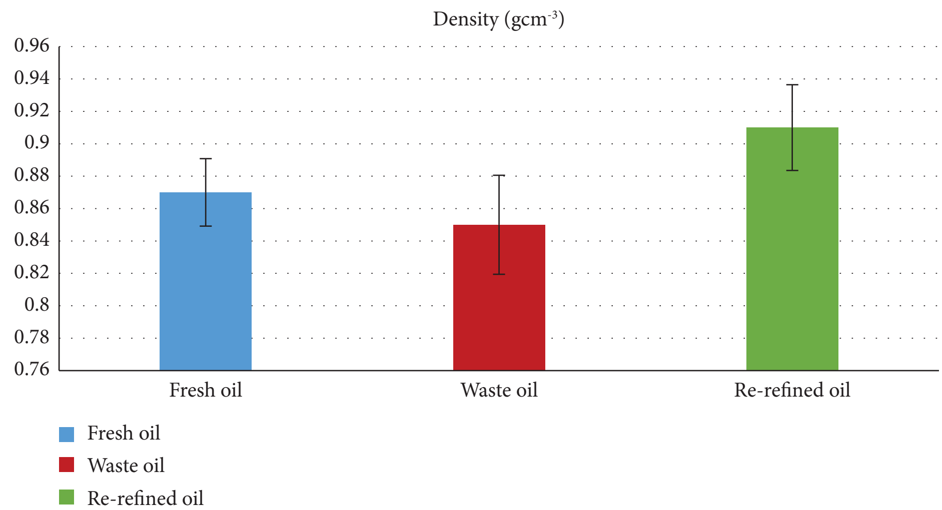


Figure 4: Density of oil samples.

3.2. Kinematic Viscosity and Kinematic Viscosity Index (cSt). Figure 5 shows the viscosity of fresh crankcase lubricating oil (FCLO), waste crankcase lubricating oil (WCLO), and re-refined crankcase lubricating oil (RCLO) at 40°C and 100°C. The viscosity at 40°C of FCLO was 104.5 cSt, that of WCLO was 104.0 cSt and that of RCLO was 105.0 cSt. The data show that the re-refining process improved the viscosity of the CLO at 40°C compared to that of the FCLO. However, at 100°C, the viscosity of the RCLO was 13.5 cSt, which was slightly lower than that of the FCLO (13.9 cSt) and WCLO (13.5 cSt). This may be attributed to the traces of fuel (less than 1%) and other contaminants that were still present in the re-refined CLO, and the gradual deterioration of the viscosity improvers (additives) due to the thermal breakdown in the crankcase and also because of the further heating in the laboratory [22].

The viscosity index is a measure of the change in viscosity with temperature and is used to predict the performance of lubricants under different conditions. The viscosity index of the FCLO was 134, that of the WCLO was 129, and that of the RCLO was 126. The data shows that the RCLO had the fastest rate of change in viscosity with temperature change as compared to the FCLO and the WCLO. This reduction in the viscosity index could be explained by the gradual deterioration of the viscosity improvers (additives) that are present in the CLO. Viscosity improvers are added to CLO to increase the oil's ability to maintain its viscosity over a wide temperature range. However, as heat is applied to the FCLO in the crankcase and with further heating in the lab, these additives deteriorate and their effectiveness is reduced. This is similar to what was observed in a study by the authors of references [21, 23].

3.3. Flashpoint (°C). The flash point is an important property of lubricating oils as it is a measure of the safety and stability of the oil under operating conditions. Figure 6 shows that the re-refined crankcase lubricating oil had a higher flash point (204°C) than the waste crankcase lubricating oil (192°C) and is only slightly lower than that of the fresh crankcase lubricating oil. This low flash point for the WCLO is most likely due to the presence of high volumes of fuel (less than 4%) as compared to that of the RCLO (less than 1%).

3.4. Total Base Number (TBN). The total base number (TBN) is a crucial parameter in determining the performance of oil in an engine. The TBN measures the ability of the oil to neutralize acidic substances produced during engine operation, such as the oxidation of oil, combustion deposits, and condensation of acidic gases. Figure 7 shows that the TBN of the fresh crankcase lubricating oil was 10.4, which is considered good. However, the TBN of the waste crankcase lubricating oil was found to be 8.0, indicating that the oil was already starting to lose its ability to neutralize acidic substances. The TBN of the re-refined crankcase lubricating oil was 8.6, which showed a slight improvement over the waste crankcase lubricating oil but not as high as the fresh crankcase lubricating oil. The slight improvement in the TBN of the re-refined crankcase lubricating oil can be attributed to the neutralization process that took place during the acid-clay re-refining process.

3.5. Water Content. The presence of water was initially determined by a crackle test which revealed a positive result. A follow-up calcium hydride test, however, indicated minute traces of water. According to Figure 8 , the FCLO contained 0%, the WCLO contained 0.01%, and the RCLO contained 0% of water. This communicates that, despite the small amount of water in WCLO, the acid-clay method was able to remove the traces of water found in the WCLO. Comparing this result to the result obtained by the authors of reference [23], it is clear that the acid-clay method has the ability to remove any traces of water in WCLO.

3.6. Wear Metals. Figure 9 shows the presence of iron (Fe) and aluminum (Al) in the three crankcase lubricating oils.

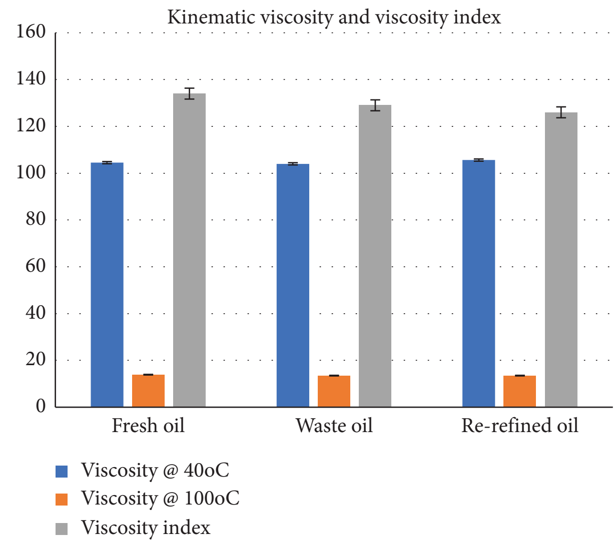


FIGURE 5: Kinematic viscosity and kinematic viscosity index of oil samples.

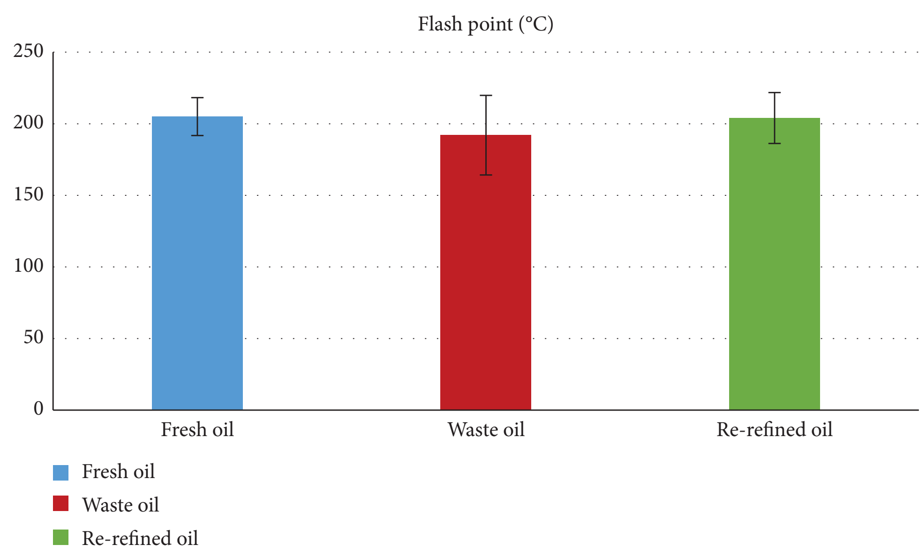


FIGURE 6: Flashpoint of the oil samples.

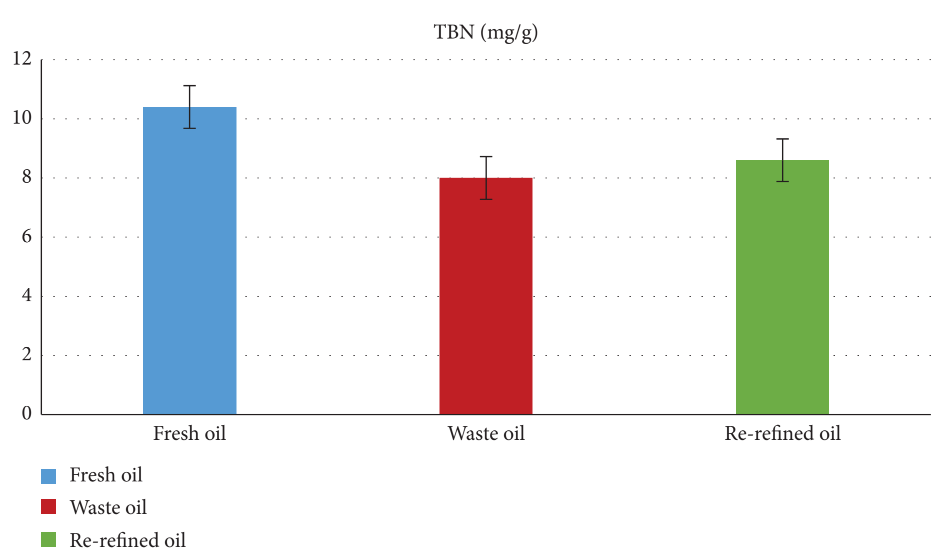


FIGURE 7: Total base numbers of oil samples.

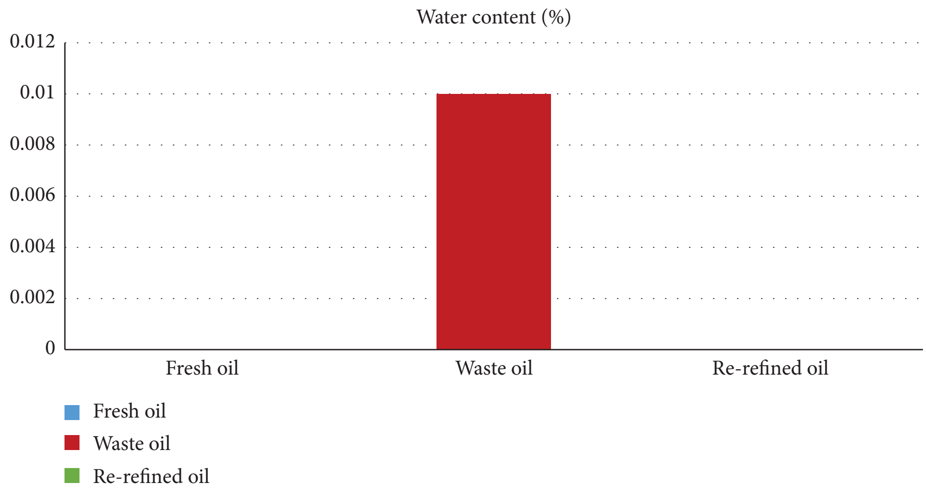
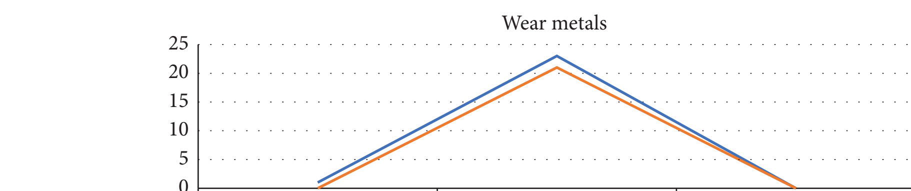


Figure 8: Water content in the oil samples.



	Fresh oil	Waste oil	Re-refined oil
Iron	1	23	0
Aluminum	0	21	0

Figure 9: Wear metal content in ppm.

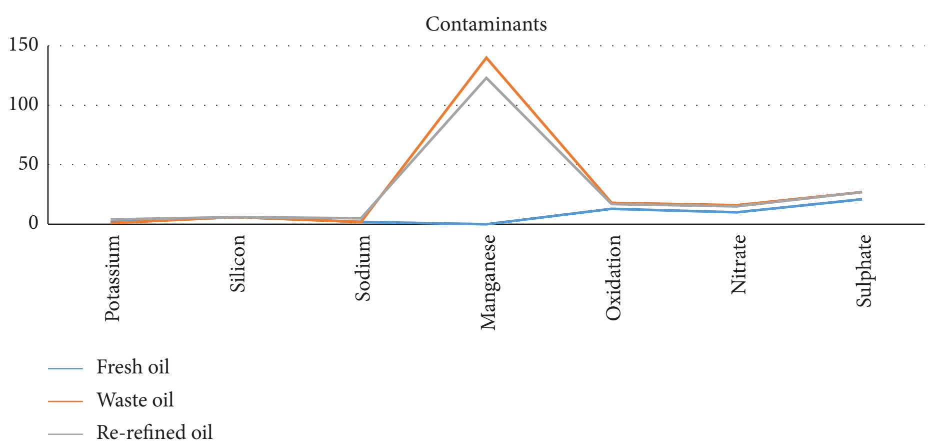


Figure 10: Contaminants found in the oil samples.

The increased concentration of Fe and Al in the waste crankcase lubricating oil (WCLO) indicated that the engine was experiencing high wear and tear. This is evident as the WCLO had a higher wear metal content of 23 ppm Fe, than that of 1 ppm Fe in the fresh crankcase lubricating oil (FCLO). The acid-clay method was effective in reducing the wear metal content of the WCLO to 0 ppm Fe and 0 ppm Al, as seen in the re-refined crankcase lubricating oil (RCLO). For the 1 ppm Fe in the FCLO, reference [24] shows that most crankcase lubricating oils contains small concentrations of heavy metals.

3.7. Other Contaminants. Figure 10 shows the contaminants present in the fresh crankcase lubricating oil (FCLO), waste crankcase lubricating oil (WCLO), and in the re-refined crankcase lubricating oil (RCLO). The results from Figure 10 demonstrate that the fresh crankcase lubricating oil contained some level of contaminants, however, the contaminants in the waste crankcase lubricating oil (WCLO) increased significantly after 5000 km of usage. This increase can be attributed to the internal engine conditions and the length of time the oil was in use. Manganese had a significant increase from 0 ppm in fresh crankcase lubricating oil to 140 ppm in waste crankcase lubricating oil. Fuel contamination also increased from 0 ppm in fresh crankcase lubricating oil to <4% in waste crankcase lubricating oil.

The acid-clay method used in this study was able to significantly reduce the level of contaminants present in the waste crankcase lubricating oil. The RCLO had manganese and fuel concentrations dropping to 123 ppm and <1%, respectively. This result is confirmed by a similar study [25] where the clay-acid method was able to reduce moisture from 0.00943 to 0.0015 and soot from 0.004715 to 0.000177 in absorbance values signifying a drastic reduction in particle quantities.

4. Conclusion and Recommendation

The conventional acid-clay method showed promising results in re-refining waste crankcase lubricating oil and in improving its properties. Analyzing the results obtained, we found that the acid clay method was able to

(i) improve the viscosity of the WCLO from 104 cSt to 105.6 cSt

(ii) improve the flashpoint of the WCLO from 192°C to 204°C

(iii) reduce the water content in the WCLO from 0.01% to 0.00%

(iv) reduce the contaminants present in the WCLO, such as iron and aluminum, from 23.0% and 21.0% to 0.0% and 0.0%, respectively

(v) reduce fuel ingress in the WCLO from <4% to <1%

However, the acid-clay method did not significantly improve the viscosity index, TBN, and density, which could be a potential area for improvement in future studies.

The sludge produced by the acid-clay process is rich in carbon elements and can be used in the production of carbon rods and for industrial heating of boilers. However, the sludge produced can pose a challenge in large-scale re-refining, and therefore, further studies can also be conducted on the efficient ways of removing the sludge.

Data Availability

All data are included in the manuscript with an additional supplementary datasheet attached.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

Supplementary Materials

The supplementary dat sheet attached is made up of the additional results obtained from the analyses. A reference was made to the fuel ingress in the various samples and that is presented in the supplementary data file. (*Supplementary Materials*)

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