

Re-Refining of Used Lubricating Oil Using Indigenous Forsterite Ore as Adsorbent

*Abdul Rub Viqar^{1,2}, Ashraf Kamal², Amtul Qayoom¹, Muhammad Talha^{1,2}, Syed Mumtaz Danish Naqvi², Sadia Amber², Mahwish Mobeen Khan², Naushin Yasin², Kashif Riaz², Sana Awan²

¹Department of Chemistry, N.E.D. University of Engineering and Technology, Karachi 75270, Pakistan.

²Department of Applied Chemistry and Chemical Technology, University of Karachi, Karachi 75270, Pakistan.

ABSTRACT

The recent work focuses on evaluating adsorption capability of indigenous forsterite ore for re-refining of used lubricating oil and to produce oil having characteristics comparable to the original base oil. Thermal gravimetric analysis, x-ray diffraction, scanning electron microscopy and energy dispersive x-ray spectroscopy techniques were used to know the actual behavior of the adsorbent including its composition and morphology. In the light of TGA analysis indigenous forsterite ore was activated at 750°C for one hour. Adsorption experiments were conducted in ambient conditions using glass columns with length to diameter ratio of 16:1. and n-hexane as solvent; collected re-refined lube oil showed remarkable reduction in color index (80% reduction), total acid number (100% reduction), total base number (100% reduction), carbon residue (91% reduction), sulphated ash (98% reduction), metals content (99%); FTIR spectra of lubricating oil before and after adsorption confirmed improved physico-chemical properties. The finding were reviewed and 100-230 mesh sized indigenous forsterite ore from Khayber Pakhtoon Khuah was found to be economical and efficient adsorbent for re-refining of used lubricant oil.

KEYWORDS: Re-refining, Used lube oil, Adsorption, Indigenous forsterite.

1. Introduction

As the number of industries, vehicles, agricultural and industrial machinery is increasing day by day, the volume of used lubricating oil has also increased. This black used oil is source of pollution at industrial and domestic level because it contains moisture, salts, broken down and burnt additives, varnishes, gums and other materials (Kamal and Khan, 2009). Hence the used lubricants are sometimes termed as anthropogenic pollutant (Kanokkantapong et al., 2009). Due to oxidation and thermal degradation of additives, lots of impurities such as unsaturated compounds, aldehydes, phenolic compounds, alcohols, acidic compounds and some hydrocarbon chains are also generated (Bridjanian and Sattarin, 2006). Burning of used oil for incineration purpose poses serious environmental threats as it contains carcinogens like polyaromatic hydrocarbons and polychlorinated biphenyls etc. (Diphare et al., 2013; Merai Yash, 2015; Shri Kannan et al., 2014). Also, used lube oil neither evaporates nor degrades (Yee et al., 2002). Major portion of waste lube oil may be reused after suitable treatment while its improper dumping causes environmental pollution and waste of valuable resources (Inamullah et al., 2015).

Different recycling procedures for used lubricants have been developed so far (Diphare et al., 2013). For example distillation and vacuum distillation (Gantz and Hellwege, 1976), de-metallizing of used lubricant oil (Gantz et al., 1985), mixing lube oil with a basic solution and a phase transfer catalyst and then settling, solvent extraction techniques (Bridjanian and Sattarin, 2006), production of hydrogen gas either through supercritical water reformation or steam reformation (Ramasamy and T-raissi, 2007), passing this oil from an adsorbent bed, clay bed or activated clay bed to further refine and decolorize it for reuse (Abu-Ellella et al., 2015; Diphare et al., 2013; Kamal and Khan, 2009; Moura, 2010; Udonne, 2011). The adsorption technique is more economical as compared to other employed methods because it requires less initial cost, easy apparatus handling, and low running cost and also the environmental concerns are reduced to minimum. The adsorption technique also produces extracted oil of better quality and purity. The present study will be useful for re-refining of used lubricating oil using cost effective indigenous adsorbent. Forsterite is a sedimentary rock which occurs either as peridot (gem stone) or as olivine (forsterite containing high iron content). It is available in Khayber Pakhtoon Khuah and Kohistan areas of Pakistan in very huge amounts.

The present work focusses on the utilization of indigenous forsterite ore as a low cost and abundant adsorbent. Since the ore is less known as compared other ores, it is mostly confused with marble, limestone and talc; and hence wasted. It has excellent porosity and regenerability on heating at higher temperatures suggesting it to be potential candidate as effective low cost adsorbent for re-refining of used lubricating oil.

2. Materials and methods

All the glassware was made of Pyrex glass, washed and oven dried. The large column was specially designed for adsorption purpose with length to diameter ratio of approximately 16:1. Soft rubber tube was used for packing of column. Commercial grade n-hexane was used for column wetting and elution study. Used lube oil was obtained from local service stations of Karachi, Pakistan. The indigenous forsterite ore was collected from Karak area of Khayber Pakhtoon Khuah in the form of lumps and used in the mesh size ranges of 100-230 mesh and 230-400 mesh.

The forsterite ore was crushed and finely grinded in small ball mill and size separation was achieved with sieve shaker (Retsch-AS 200 Tap) using Tyler's standard sieves. The adsorbent was then activated in muffled furnace (Carbolite CFW 1200 electric oven - Maximum temperature 1100°C) at 750°C for one hour. The activated sorbent was filled in the column and oil-solvent mixture (1:2 ratio). The oil-solvent mixture collected after adsorption process was separated through semi-automatic laboratory vacuum distillator and then dried in vacuum oven.

2.1. Analysis of Ore

The selected indigenous forsterite ore (100-230 mesh size) was analysed for the following parameters:

- For thermal gravimetric analysis (TGA) of indigenous forsterite ore Mettler Toledo analyser TGA/SDTA851e/LF) was used at flowing nitrogen quantity of 20mL/min at a temperature range of 25°C to 1000°C.
- Indigenous forsterite ore was analysed using XRD diffractometer (XPERT-PRO) operated at 40kV and 30mA utilizing CuK α radiations. The temperature was kept constant at 25°C and the step time was 0.60s per interval.
- Morphology of indigenous forsterite ore was observed by Jeol Scanning Electron Microscope (SEM), model JSM 6380LA at an accelerating voltage of 20 kV. The scanning electron micrographs were captured using an in-lens detector under the magnification of X1000 to X10000. The ore powder was coated with a thin gold layer under vacuum to make the surface conductive before placing the samples in SEM.
- Gold coated indigenous forsterite particles were also tested for the presence of elements by EDS unit, model EX-54175JMU attached with SEM which was operating at 1.0 nA (probe current) and T3 (PHA mode) using liquid nitrogen as coolant with a counting rate of 6605 cps.

2.2. Analysis of Lubricant Oil

The used and re-refined oil samples were tested to find out their quality for following parameters,

- Colour Index by Lovibond Colour Comparator (ASTM D1500, 2012)
- Specific Gravity (ASTM D 1480, 2015)
- Viscosity by Cannon Ubbelohde viscometer (ASTM D445, 2015)
- Flash Point and Fire Point by Cleveland Open Cup flash point tester (ASTM D92, 2016)
- Total Acid Number by (ASTM D974, 2014)
- Total Base Number (ASTM D2896, 2011)
- Carbon Residue by ASTM D-189 method (ASTM D189, 2014)
- Sulphated Ash (ASTM D874, 2013)
- Metal Content Analysis using Spectroil M - Rotating Disc Electrode Atomic Emission Spectrometry
- FTIR using KBr cell.

3. Result & Discussion

The settling process removed heavy particles and large gummy materials from used lubricant oil. The optimum particle size of adsorbent is an important factor for adsorption studies. Larger particle sizes were not tried because the column would not have been packed evenly and spaces would have been present between the

particles which might affect the adsorption study. Lower particle sizes range (100-230 mesh) was preferred for the study because they provided larger surface area for the adsorption of oil contaminants and also subjected to even packing of the adsorption columns; but very small particle size range (230-400 mesh) found choking the column; not providing a free path even for the solvent.

Usually inactivated ores are not considered effective adsorbent for re-refining of used lube oil. Trial studies showed that heating process improved the adsorption ability of the ore. The adsorption capability of adsorbent increases as the activation temperature increases since pore size also increases with increase in activation temperature. TGA of indigenous forsterite ore also verified the above statement. Activation temperature for indigenous forsterite ore was selected on the basis of thermal analysis.

3.1. Characterization of Indigenous Forsterite Ore

3.1.1. Thermal Gravimetric Analysis (TGA)

The TGA scan (Figure 1) of selected indigenous forsterite ore was obtained at a temperature range of 25°C to 1000°C with a heating rate of 10°C/min. The graph shows that the mass lost was completed in three stages. The ore kept losing moisture and insignificant masses till 485°C (first stage) after which the actual mass loss of ore started. In the second stage the ore kept losing the mass from 485°C to about 600°C which was due to the decomposition of MgCO₃ which was present as an impurity in the ore. After completion of second stage, third stage started from 600°C when silicates started to decompose at the elevated temperature. The ore kept losing its mass and maximum mass lost was observed at around 758°C (third stage completed) where all possible carbonates converted to oxides. After about 750°C the graph became linear showing consistent mass loss and no significant effect of temperature. The findings were studied thoroughly and the temperature of 750°C and the activation time of one hour was preferred for further study.

3.1.2. X-Ray Diffraction (XRD)

XRD pattern of raw indigenous forsterite (Figure 2a) contains several characteristic peaks of indigenous forsterite ore at 2-theta values of 17.40, 22.90, 24.00, 25.50, 26.55, 29.50, 32.35, 32.70, 35.80, 36.55, 38.40, 39.80, 40.15, 52.30, 56.20 and 62.80 (Meng et al., 2015). The XRD of indigenous forsterite ore also contained peaks of magnesium carbonate (2-theta values of 43.05 and 53.90) which were removed during activation process at higher temperature (Figure 2b) due to its decomposition into MgO and CO₂ (Engler et al., 1988).

3.1.3 Scanning Electron Microscopy (SEM)

The SEM image of raw and thermally activated indigenous forsterite is given in Figure 3a and Figure 3b. On activation at 750°C for one hour some micro pores generated in the indigenous forsterite ore due to release of carbon dioxide gas.

This indicated that adsorption capability of indigenous forsterite ore increased on activation of ore at higher temperature.

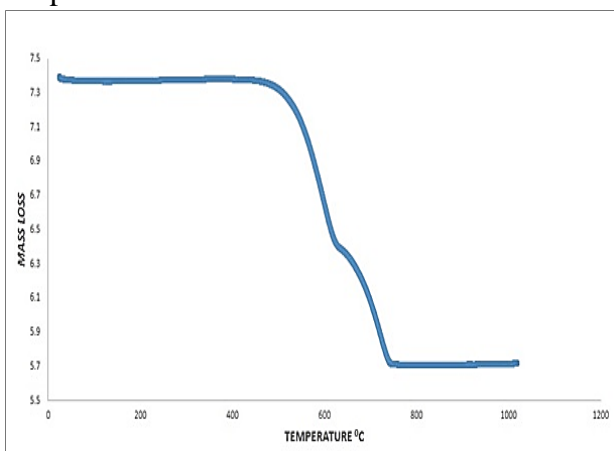


Figure 1: Thermal gravimetric analysis graph of indigenous forsterite ore from Khayber Pakhtoon Khuah

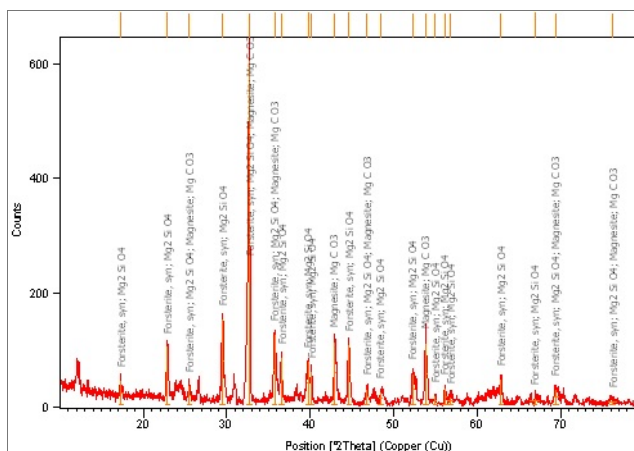


Figure 2a: XRD graph of raw ore showing different compounds in the indigenous forsterite ore

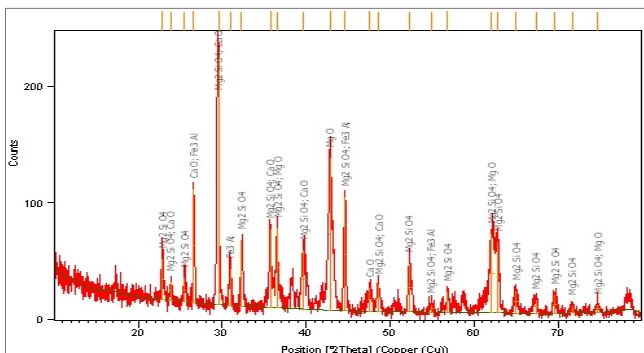


Figure 2b: XRD graph of indigenous forsterite ore showing different compounds after activation at 750°C for one hour

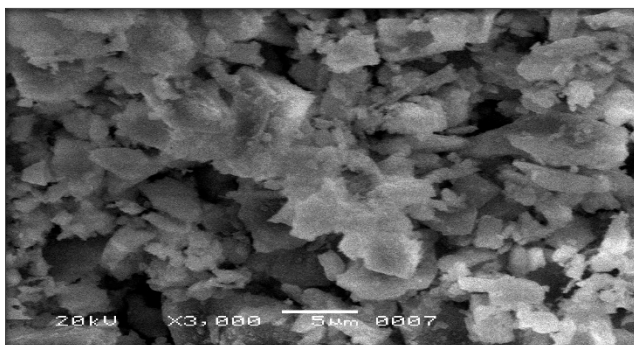


Figure 3a: SEM image of inactivated/raw indigenous forsterite ore showing particle structure at X3000 magnification.

3.1.4 Energy Dispersive X-Ray Spectroscopy (EDS)

Figure 4a shows that major ingredient of indigenous forsterite ore is Mg_2SiO_4 while magnesium carbonate may be present with it as an impurity. It also contains small amount of iron and other siliceous compounds in it. EDX spectrograph indicated greater oxygen counts as compared to magnesium, calcium and the carbon (Figure 4b). The calcination process reduced the counts of oxygen as well as carbon. It may be due to decomposition of carbonates impurities into respective oxides and carbon dioxide heating.

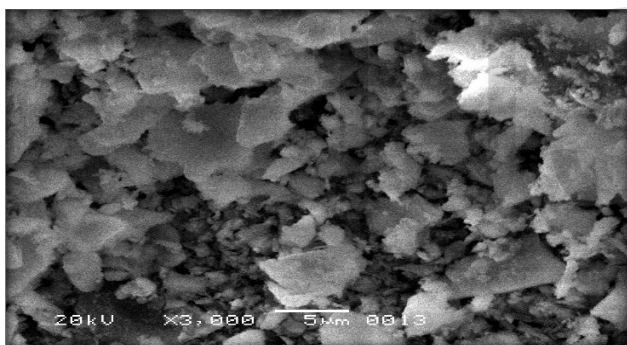
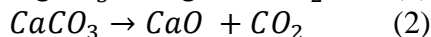
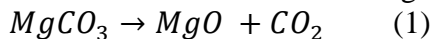


Figure 3b: SEM image of activated indigenous forsterite ore at 750°C for 1 hour showing opened pores at X3000 magnification

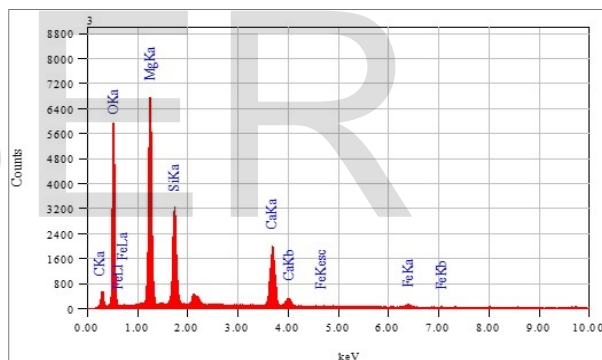


Figure 4a: EDS scan of raw indigenous forsterite ore showing higher counts of elements present in it

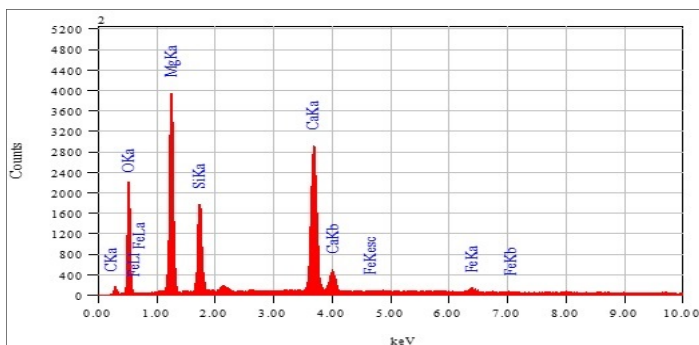


Figure 4b: EDS scan of indigenous forsterite ore activated at 750°C for 1 hour showing lower counts of elements left over in it due to mass loss

3.2 Characterization of lube oil

Comparison between physio-chemical properties of used and re-refined lube oil is given in Table 1.

3.2.1 Colour Index

Colour index value of used lubricant oil was very high due to the presence of burnt additives and other impurities in it. Colour index value of re-refined oil was low (with in the acceptable range of colour index 1-3) which confirmed that the burnt additives had been removed.

3.2.2 Specific Gravity

Specific gravity values for used and re-refined lubricant oil were found to be 0.888 and 0.875 respectively. The decrease in specific gravity value of re-refined oil showed that re-refining of used lubricating oil removed lower chains of carbon, gummy materials and other impurities which were causing higher specific gravity of used lube oil.

3.2.3 Viscosity

At 33°C viscosity of used lube oil was found to be 82.47 centistokes per second (cSt/sec) which was higher as compared to the re-refined lubricant oil (46.45 cSt/sec) indicating effectiveness of adsorption process for removal of varnish, gummy and sludgy materials.

3.2.4 Flash Point and Fire Point

Flash point value of used lube oil is 185°C (365°F) and extracted base oil is 204°C (399.2°F) which is the clear demonstration of burnt additives' removal. The flash point experiment was further continued to find the ignition points which were found to be 204°C (399.2°F) for black used oil and 210°C (410°F) for the base lubricant oil.

3.2.5 Total Acid Number (TAN)

Total Acid Number (TAN) for used lubricating oil was found to be 2.27 mg KOH/g Oil which might be due to the presence of acidic impurities, oxidation and degradation products at high temperatures inside the engine. The Nil value of TAN in refined oil suggested that the impurities have been removed completely and no acidic nature impurity was left in the oil anymore.

3.1.5 Total Base Number (TBN)

Total Base Number of use lube oil was 5.02 mg HCl/g Oil indicating presence of inorganic and organic bases along with some polymers containing nitrogenous groups. The Nil value of TBN in the extracted base lube oil ensured complete removal of those interfering compounds.

3.2.6 Carbon Residue

Amount of carbon residues had been largely reduced (1.87 to 0.18 wt%) in the refined oil after the adsorption process. This proved that the adsorption process for the purification and refining of oil is much efficient as compared to other processes.

3.2.7 Sulphated Ash

The comparison of results for sulphated ash test in used and refined oils indicated that the amount of additives that were reactive to sulphuric acid and wear metals had been removed to a great extent (0.75 to 0.018 wt%) and remaining amount of sulphated ash in re-refined oil is almost negligible.

3.2.8 Metals Analysis

Removal of wear metals from used lube oil was one of the requirements of the refining process. The selected adsorbent was highly porous to adsorb these metal impurities. The selection of non-polar solvent (commercial grade n-hexane) made the process remarkably feasible. The selection of polar solvent could be problematic and the maximum removal of metals might have been impossible (Kamal and Khan, 2009).

Table 2 shows that the metal content has been removed up to 96% which was a remarkable decrease in metal impurities. Especially the chromium was completely removed during the re-refining process (Durrani, 2013). The quantity of remained metals in the extracted base lube oil was found negligible and will not be problematic if the extracted oil is reused.

3.2.9 Fourier Transform Infrared Spectroscopy (FTIR)

Figure 5 presents overlaid FTIR spectra of used and extracted lubricant. The peak observed at 1052cm⁻¹ indicates the presence of Ca(Mg) phosphate while peak at 1076cm⁻¹ indicates stretching band of Ca(Mg) sulphonate (Janina Zi, ba-Palus, 1999; Mohammad A. Al-Ghouti, 2009). The peak at 1150cm⁻¹ suggests the presence of methacrylate which is viscosity index improver (Cerney et. al., 2001).

Methacrylate peaks might also be present around 1733cm⁻¹ and at 1770cm⁻¹ (F. Hui, G. Machtalkre, J. Xie, H. Kolodziejczyk, 1997). Three peaks at 1154cm⁻¹, 1169cm⁻¹ and 1270cm⁻¹ may be due to the presence of polymethacrylate in the oil sample (Janina Zi, ba-Palus, 1999; Mohammad A. Al-Ghouti, 2009). At 1231cm⁻¹ the peak of succinimide is also found (Cerny et al., 2001; Janina Zi, ba-Palus, 1999; Mohammad A. Al-Ghouti, 2009). The peak at 1390cm⁻¹ may be due to the presence of Alkylsalicylates (carbonates) in the sample (Cerny et al., 2001).

The region of 1700-1800cm⁻¹ is the region showing largest number of compounds in the lube oil which are mostly C=O oxidation products e.g succinimide dispersant or characteristic band of polybutenylsuccinimide type dispersant peak at 1705cm⁻¹ (F. Hui, G. Machtalkre, J. Xie, H. Kolodziejczyk, 1997; Machtalsre et al., 1996), characteristic band of carboxylic group (peak value is at 1713cm⁻¹) (Machtalsre et al., 1996), ester of methacrylate or at 1730cm⁻¹ (Cerny et al., 2001; Hui and Rosset, 1995), polymethacrylate stretching bands at 1731cm⁻¹ (Janina Zi, ba-Palus, 1999; Mohammad A. Al-Ghouti, 2009), methyl ethyl ester peak at 1743cm⁻¹ (Machtalsre et al., 1996), C–O aldehydes/ketones peak at 1750cm⁻¹ (Hui and Rosset, 1995; Scapin et al., 2007), peak of secondary band of polybutenyl succinimide type dispersant at 1771cm⁻¹ (Machtalsre et al., 1996), and secondary band of carboxylic group at 1778cm⁻¹ (Machtalsre et al., 1996).

A peak is found at 3500cm⁻¹ which is the peak of metallic sulphonate over-based detergent (F. Hui, G. Machtalkre, J. Xie, H. Kolodziejczyk, 1997; Hui and Rosset, 1995).

The FTIR spectra also shows that the organic impurities have not been removed completely which may be due to the use of organic nature chemicals; but concentration of those impurities reduced to minimum possible level. These are not the impurities actually but the additives which are mixed with the base oil in order to improve or impart certain characteristics in base lube oil. Hence presence of these additives in extracted base lube oil can be taken as positive which may reduce the amount of same additives required to reprocess the base oil for next use.

3.3. Regeneration of Adsorbent

The sorbent used for the adsorption purpose was not wasted after use. After the adsorption process the adsorbent was collected back and regenerated again at 750oC for one hour, and hence reused for the adsorption process. The process was repeated thrice regenerating the same adsorbent again and again, and similar quality of base lube oil was obtained after each regeneration. The oil collected after each adsorption step was named as 1st pass, 2nd pass and 3rd pass. Figure 06 describes % efficiency of adsorbent for each pass. The experiments showed that there was negligible decrease in the refining capability of the adsorbent after each regeneration. The refining efficiencies were 99.93%, 99.91% and 99.79% for 1st, 2nd and 3rd pass respectively. This proves that the adsorbent has the regeneration capability to at least three times. Also, there was no change observed in the appearance of adsorbent after each regeneration and the colour of regenerated adsorbent was as bright as of initially activated ore. This also shows that the adsorbent still has the capability for regeneration and refining more used lube oil. Regeneration of adsorbent also reduced the cost of re-refining technique. Also the adsorbent to oil ratio was kept constant after regeneration of ore each time.

4. Conclusion

In this study, adsorption potential of activated indigenous forsterite ore was evaluated for treatment of used lubricating oil. Presented results suggest that activated indigenous ore is effective in removing contaminants from used lube oil and improving its physio-chemical properties. Moreover, re-refining of used lubricating oil using activated indigenous forsterite doesn't require cumbersome treatment steps therefore consume less time as compared to other available methods. Availability of indigenous forsterite in abundance, it's simple regeneration after use and production of re-refined oil of similar quality as that of virgin oil are key factors in making it economically feasible solution for treatment of used lube oils. The re-refining of used lubricating oil will also result in reduced environment deterioration and reuse of re-refined oil will also be wise because of its finite resources.

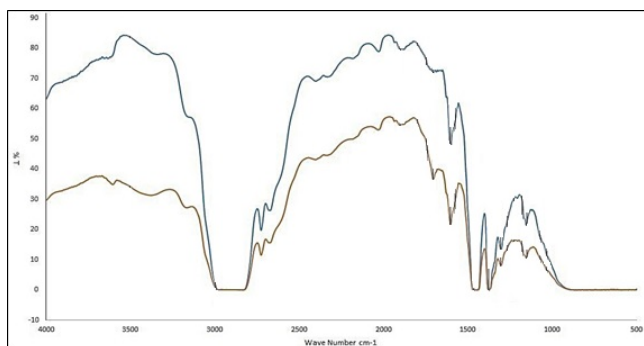


Figure 5: FTIR bands of used lube oil (brown line) and extracted base lube oil (blue line) showing peaks of additives present in it

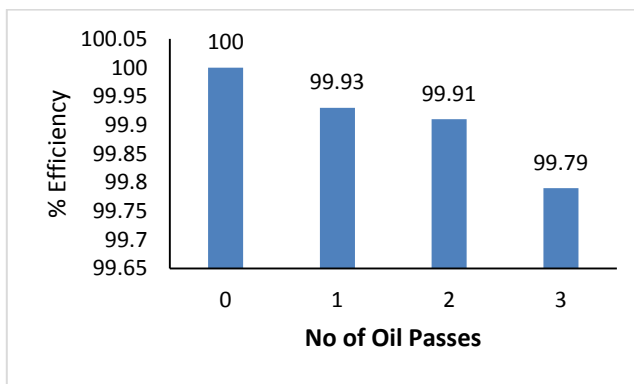


Figure 6: Reusability of activated indigenous forsterite for the re-refining of used lube oil

Table 1: Physio-chemical properties of used and re-refined lube oil.

Parameters	ASTM Method	Fresh Lube Oil	Used Lube Oil	Re-refined Lube Oil		
				1st Pass	2nd Pass	3rd Pass
Specific gravity at 35°C	ASTM D-1480	0.90	0.888	0.875	0.880	0.880
Viscosity at 33°C (cst)	ASTM D-445	82.20	84.47	46.45	45.86	46.24
Sulphated ash (wt%)	ASTM D-874	-	0.75	0.018		
Total base number (mg HCl/g oil)	ASTM D-2896	-	5.02	Nil	Nil	Nil
Total acid number (mg KOH/g oil)	ASTM D-974	-	2.27	Nil	Nil	Nil
Carbon residue (wt%)	ASTM D-189	-	1.87	0.18		
Colour Index	ASTM D-1500	1 – 3	>8	3	3	3
Flash point (°C)	ASTM D-92	188.0	185.0	204.0		
Yield (%)	-	-	100	99.93	99.91	99.79

Table 2: Results of metal content (mg/Kg ± 0.1) in used lube oil and recovered base oil.

Metals	Used Lube Oil (mg/Kg)	Recovered Oil (mg/Kg)	% Reduction
Fe	70.4	0.7	99.01
Na	52.1	0.4	99.23
Mo	25.6	0.3	98.83
Al	14.2	0.6	95.77
Cr	4.2	0	100.00
Cu	7.3	0.1	98.63
B	6	0	100.00
Ni	0.4	0	100.00
Ag	0.2	0	100.00
Ba	0.2	0.1	50.00
Ca	1233	4.5	99.64
Zn	755	2.2	99.71
P	576	3.2	99.44
Mg	145	0.8	99.45
Pb	141	0	100.00
Total % Reduction			95.981

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