Revue des Energies Renouvelables Vol. 21 N°3 (2018) 473 - 485
DOI: https://doi.org/10.54966/jreen.v21i3.705

Indigenous kaolinite ore as cost effective adsorbent for re-refining of used lubricating oil

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(reçu le 29 Septembre 2018 - accepté le 30 Septembre 2018)

Abstract - *Present study evaluates efficiency of indigenous kaolinite ore as environment friendly and low cost adsorbent for re-refining of used lubricating oil. Thermo gravimetric analysis was used to find the mass variation with rise in temperature. Different characterizing techniques such as XRD, SEM and EDS were used to see the physical and chemical changes, composition and available volume of pores before and after activation. Indigenous kaolinite ore was activated at 600 °C for one hour to increase pore size to surface area ratio. Adsorption was carried out by passing exhausted lube oil with commercial grade n-hexane in a fixed 1:2 v/v ratio over the surface of activated kaolinite in a glass column with length to diameter ratio of 16:1. The improvement in the quality of lube oil was determined by comparing quality parameters of used and refined lube oil. The process gave 95 % yield of good quality transparent pale-yellow oil having colour index of 2 with 99 % of metal reduction. Maximum capability of kaolinite ore to adsorbed oil was found to be 160 g of refined oil/200 g of kaolinite. Used adsorbent was regenerated and utilized again for the adsorption of more oil. Regenerated ore provided 150 g refined oil/200 g Kaolinite. The solvent was recovered through rotary evaporator under vacuum. Recovery of solvent and adsorbent regeneration suggest that proposed method for purification of lube oil is environment friendly and cost effective.*

Résumé - *La présente étude évalue l'efficacité du minerai de kaolinite indigène en tant qu'adsorbant écologique peu coûteux pour le raffinage des huiles lubrifiantes usées. L'analyse thermo gravimétrique a été utilisée pour trouver la variation de masse en fonction de l'augmentation de la température. Différentes techniques de caractérisation telles que XRD, SEM et EDS ont été utilisées pour voir les changements physiques et chimiques, la composition et le volume disponible des pores avant et après activation. Le minerai de kaolinite indigène a été activé à 600 °C pendant une heure pour augmenter la taille des pores par rapport à la surface spécifique. L'adsorption a été réalisée en faisant passer de l'huile de graissage épuisée avec du n-hexane de qualité commerciale dans un rapport fixe de 1:2 v/v sur la surface du kaolinite activé dans une colonne de verre avec un rapport longueur/diamètre de 16:1. L'amélioration de la qualité de l'huile de lubrification a été déterminée en comparant les paramètres de qualité de l'huile de lubrification usagée et raffinée. Le procédé a donné un rendement de 95 % d'huile transparente jaune pâle de bonne qualité ayant un indice de coloration de 2 avec 99 % de réduction du métal. La capacité maximale du minerai de kaolinite à adsorber l'huile a été de 160 g d'huile raffinée pour 200 g de kaolinite. L'adsorbant usagé a été régénéré et réutilisé pour l'adsorption d'une plus grande quantité d'huile. Le minerai régénéré a fourni 150 g d'huile raffinée/200 g de kaolinite. Le solvant a été récupéré par évaporateur rotatif sous vide. La récupération de la régénération des solvants et des adsorbants suggère que la méthode proposée pour la purification de l'huile lubrifiante est respectueuse de l'environnement et rentable.*

Keywords: Re-refining - Waste lube oil - Used lube oil - Adsorption - Indigenous kaolinite.

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1. INTRODUCTION

With urbanization on rise, number of industries, industrial machineries and vehicles are increasing day by day which in turn produce huge volume of used lubricating oil. As the vehicle moves, metallic contaminates cadmium, tin, zinc, lead etc. are also released into lube oil [1].

Some other impurities generate in the lube oil due to the burning and degradation of the additives [2]. Although used oil contains carbon chains but their biodegradation takes quite longer time [3]. Usually, in auto mobiles, lube oil needs to be changed after every 1000 to 5000 kilometers of running depending on the vehicles efficiency [4]**.** The huge amount of waste lube oil is dumped in water and land in uncontrolled manner [5].

Almost 40 % of used lube oil is disposed in land and sea [6] whereas only one quarter of it has the ability to contaminate 250000 gallons of water and 40730 square feet of land [7]. The toxic components of used lube oil affect the food chain. In order to protect the environment from this growing environmental problem, used lubricating oil has to be recycled by some standard procedure.

Different recycling procedures for used lubricants have been developed so far. For example distillation and vacuum distillation [8], de-metallizing of used lubricant oil [9], mixing lube oil with a basic solution and a phase transfer catalyst, solvent extraction techniques [10], production of hydrogen gas either through supercritical water reformation or steam reformation [11], passing used oil from an adsorbent bed, clay bed or activated clay bed to further refine and decolourize it for reuse [2, 12–14].

The adsorption technique has gained considerable attention in recent years because unlike chemical treatment, no acid sludge produces and all the impurities are collected in one place. For this purpose suitable low cost, regenerate-able, easily available adsorbents are used [15]. Kaolinite mineral is a soft material with high aluminum and silica content having chemical formula of $Al_2Si_2O_5(OH)_4$.

The crystal structure of kaolinite has a layered silicate mineral, with one tetrahedral sheet of silica (SiO_4) linked through oxygen atoms to one octahedral sheet of alumina (AlO_6) octahedral. It also has variable iron content in its octahedral positions depending upon the iron content in the existing soil [16]. In this study indigenous kaolinite is used as an adsorbent because none of the study has yet performed using kaolin as an adsorbent for re-refining of oil.

It is easily available all over the world and has good adsorption properties as it is used as a cosmetic cleaning agent at homes indigenously. Indigenous kaolinite includes in Fuller's earth with efficient capability to decolorize oil or other liquids without chemical treatment [17].

The objective of this study is to introduce indigenous kaolinite ore as an adsorbent for re-refining of used lube oil. In proposed method, re-refining of lube oil was performed in single step adsorption technique without any other pre or post treatment. The highlight of the process is its being economical and less time consuming.

2. MATERIALS AND METHODS

2.1 Materials

All the glassware were 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 utilized for packing of column. Different used lubricating oil samples were collected by several service stations of Karachi, Pakistan.

Commercial grade n-hexane was used as mobile phase for elution of lube oil. Pakistan has huge amount of deposit of Kaolinite ore. Four Kilogram of kaolinite ore was first crushed by a small hammer to reduce its size to around 16 mesh size and then grinding up to 100-230 and 230-400 mesh sizes

2.2 Analysis of Ore

XRD analysis was carried out using Pan analytical Xpert-Pro Diffractometer system operated at 40 kV and 30 mA with Cu $K\alpha$ and Cu $K\beta$ radiations. The analysis was performed on 100-230 mesh particles of kaolin ore both with and without thermal activation. Scan Step time was 0.60 seconds per interval and temperature was 25 °C.

Microstructures of samples were captured by Jeol scanning electron microscope (SEM), model JSM 6380LA at an accelerating voltage of 30 kV. Images were taken using an in-lens detector under X10000 magnification. Kaolinite particles were coated with a thin gold layer under vacuum to make the surface conductive before placing the samples in SEM device.

Elemental analysis of 100-230 mesh sized gold coated kaolinite particles was performed by EDX unit, model EX-54175JMU attached with SEM operating at 1.0 nA (probe current) and T3 (PAH mode) using liquid nitrogen as coolant. EDX spectra were recorded and analyzed by ZAF method for quantitative analysis.

2.3 Experimental procedures

Initially small glass column of length 26.4 cm and diameter 1.8 cm was packed with fixed 20 g of thermally activated adsorbent of 100-230, 230-400 mesh size. 230-400 mesh size chocked the column and oil couldn’t be collected. Therefore 100-300 mesh size particles were chosen for further study.

Adsorption column was soaked with commercial grade n-hexane. A 1:2 v/v mixture of oil and n-hexane was passed through column. Then column was again washed with solvent to remove all the traces of base oil. After washing, the column was dried by vacuum pump and the ore was removed from column. This process was continued with increased amount of used lube oil and solvent quantity in the same ratio with freshly prepared activated ore, until adsorbent failed to adsorb more impurities of oil resulting the production of oil of high colour index.

It was found that 20 g thermally activated kaolinite ore have ability to refine 16 g of used oil when passed with 32 ml of commercial grade n-hexane. Same experiment was performed by increasing amount of adsorbent, solvent and oil by 10 times in large column. After complete adsorption remaining material of column was collected by vacuum pump and solvent was separated from oil by using rotary evaporator under vacuum.

Vacuum oven was used to remove trace amount of solvent in oil at -40 psi and 60 °C. Purified oil was weighed to get the percentage of oil recovered. The recovered adsorbent was regenerated again at temperature 600 °C for one hour and again used for adsorption of more oil.

2.5 Analysis of lubricant oil

The used and re-refined oil through adsorption experiments were tested to find out their quality and purity for the parameters including: Colour Index by Lovibond Color Comparator using ASTM D-1500 method [18] and Specific Gravity by ASTM D-1480 method [19]. Furthermore, viscosity by Cannon Ubbelohde viscometer of standard 1B by ASTM D-445 method [20], Flash Point and Fire Point by Cleveland Open Cup flash point tester by ASTM D-92 method [21]. Total Acid Number (TAN) was estimated by ASTM D-974 method [22] and Total Base Number (TBN) by ASTM D-2896 method

[23]. To determine carbon Residue, ASTM D-189 method [24] was used and sulphated Ash by ASTM D-874 method [25]. Metal Content Analysis by using Spectroil M-Rotating Disc Electrode, Atomic Emission Spectrometry and FTIR through CaBr cell using Shimadzu IR-460 spectrometer.

3. RESULTS AND DISCUSSIONS

The overall process for the re-refining of used lube oil using indigenous kaolinite ore is presented in the following figure 1. The settling process removed heavy particles and large gummy materials from the used lubricant oil. The selection of particle size was an important factor during the adsorption study. 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 provide larger surface area for the adsorption of oil contaminants and also subject to the 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. Hence the particle size range of 100-230 mesh was found appropriate for the adsorption study.

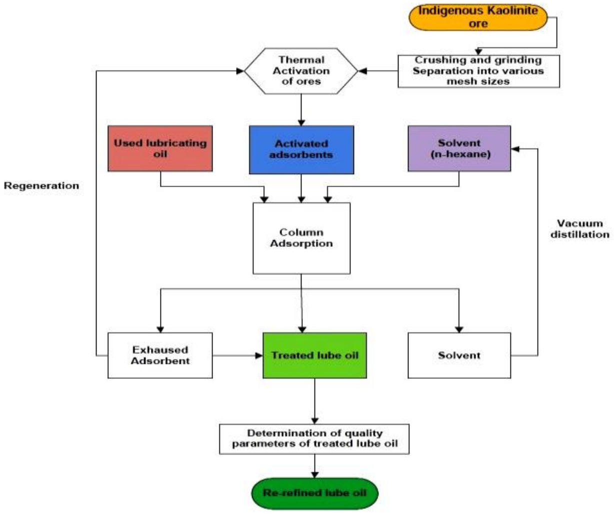

Fig. 1: Re-refining of used lube oil using Indigenous Kaolinite ore

Usually inactivated ores are not considered effective adsorbent for the 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 the increase in activation temperature. TGA of indigenous kaolinite ore also verified the same. Activation temperature for indigenous kaolinite ore was selected on the basis of thermal analysis.

3.1 Characterization of indigenous kaolinite ore

3.1.1 Mass loss determination

Significant mass loss was observed on heating kaolinite ore and it was found that mass loss depends on temperature and duration of heating (figure 2).

However, mass loss stops after certain temperature and no further mass loss was obtained. Maximum mass loss was found to be 15.40 % ,when heating was carried out at 800 °C for three hours which is approximately equal to 14.0 % (mass loss pure kaolinite) [26].

During heating initially moisture is removed, than water of hydration comes out and if the temperatures increases further than dehydroxylation process starts [27]. Mass loss increases the surface area of the ore and it is almost independent of particle size. The increase in surface area is responsible of adsorbing the impurities.

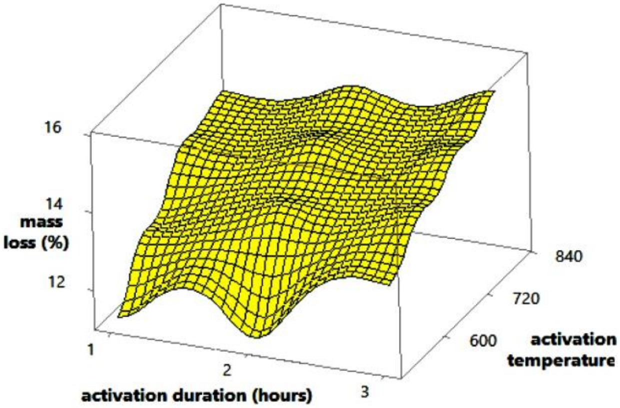

Fig. 2: Graph between mass loss, temperature and activation time

3.1.2 Thermal gravimetric analysis (TGA)

The TGA scan (Figure 3) of selected indigenous kaolinite ore was obtained at a temperature range of 25oC to 1000oC with a heating rate of 10oC/min to know the mass loss depending upon temperature, heating rate and duration at a specified temperature.

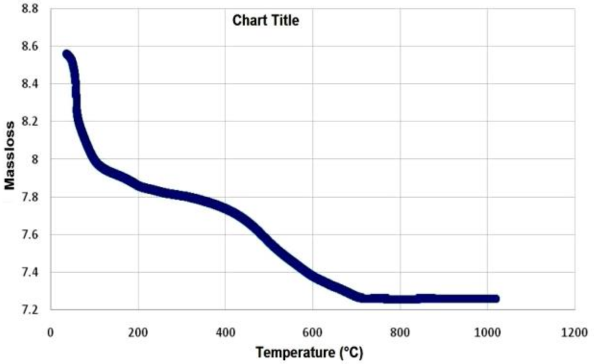

Fig. 3:Thermal gravimetric analysis (TGA) graph of indigenous kaolinite ore

The thermogram given in figure 3 shows that the mass loss was completed in four stages.

Stage I (Room temperature to 100 °C): it shows the removal of absorbed water on the surface.

Stage II (100 °C - 400 °C): it shows weight loss that can be due to pre-dehydration process. The greater the weight loss, the less the crystallinity of the samples.

Stage III (400 °C - 700 °C): it shows the dehydroxylation of a well-crystallized kaolinite ore began at 400oC and completed till 700 °C.

Stage IV (700 °C - 1000 °C): After 700 °C no further mass loss was found. The combined water was lost rapidly at about 700 °C ~ and then the reaction was complete. In well-crystallized samples the decomposition of kaolinite starts at higher temperature. The process of dehydroxylation depends on temperature, heating rate and time, and cooling parameters. Kaolin after dehydroxylation might convert in meta kaolin.

$$Al_2O_3\,2SiO_2\,2H_2O \;\rightarrow\; Al_2O_3\,2SiO_2 + 2H_2O\uparrow$$

3.1.3 X-Ray Diffraction (XRD)

In XRD pattern the characteristic peaks of kaolinite ore appears at 2θ 12.41, 24.85, 45 and 62.26 (figure 4**a**).

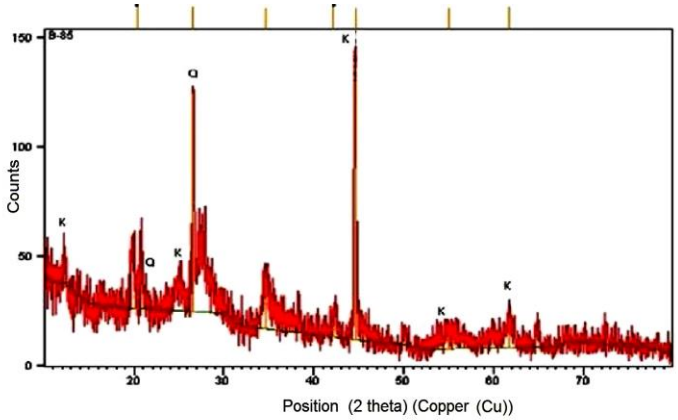

Fig. 4**a**: XRD graph of raw ore showing different compounds in the indigenous kaolinite ore

These peaks reduced after thermal treatment at the 600 °C temperature for one hour duration (figure 4**b**) while peaks assigned to quartz (2 θ 21.22 and 27.45°) remained unchanged.

Reduction of kaolinite peaks shows that some of the crystalline kaolinite ore after the removal of water present in its crystal lattice might convert in amorphous meta-kaolin, thus surface area of the ore increased.

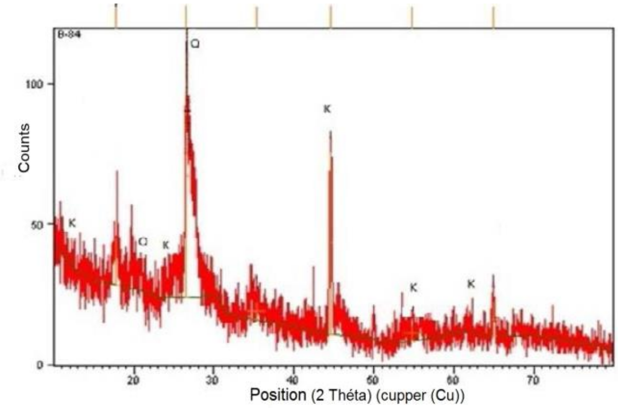

Fig. 4**b**: XRD graph of indigenous kaolinite ore showing different compounds after activation at 600 °C for one hour

3.1.4 Scanning electron microscopy (SEM)

Adsorbent must have pores in it which is responsible to adsorb impurities. Figure 5**a** shows the SEM image of untreated raw kaolinite ore in which some small micro pores are shown clearly indicating kaolinite have adsorption capability.

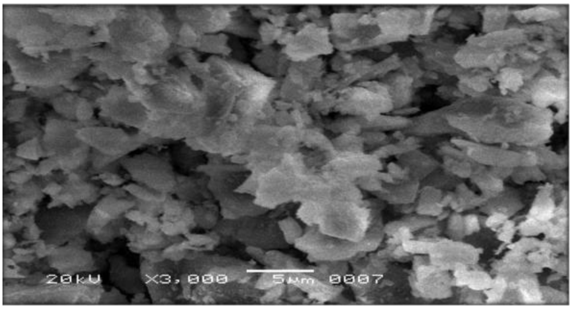

Fig. 5**a**: SEM image of inactivated/raw indigenous kaolinite ore showing particle structure at X3000 magnification

While after thermal treatment at 600 °C for one hour, these pores increased (figure 5**b**). Generation of new micro pores was due to the mass loss in the form of water of hydration and dehydroxylation process.

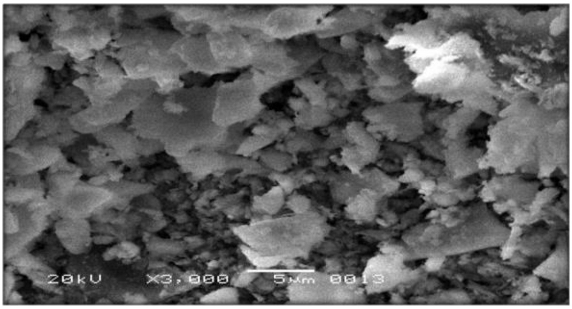

Fig. 5**b**: SEM image of activated indigenous kaolinite ore at 750 °C for one hour showing opened pores at X3000 magnification

3.1.5 Energy dispersive X-Ray spectroscopy (EDS)

Major ingredient of indigenous kaolin clay is kaolinite having formula of $Al_2Si_2O_5(OH)_4$, while magnesium, titanium and iron compounds may present as an impurities. As kaolin is a clay mineral, therefore silica and quarts may also be present in kaolin clay. Elemental analysis of raw indigenous kaolinite ore by EDX spectroscopy (Figure 6**a**) indicated greater oxygen counts as compared to aluminum and magnesium.

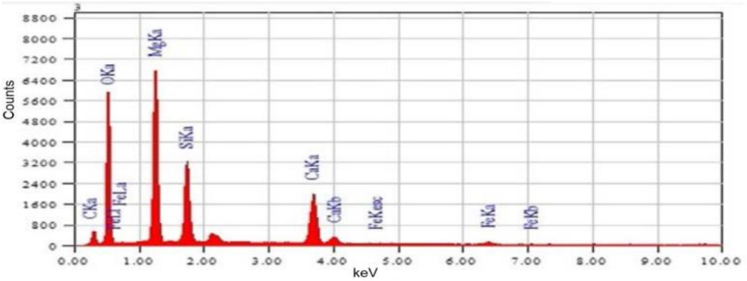

Fig. 6**a**: EDS scan of raw indigenous kaolinite ore showing higher counts of elements present in it

But after activation at 600 °C for one hour, counts of oxygen reduced greatly as shown in figure 6**b**. This was due to the removal of H_2O in different stages already discussed in TGA section.

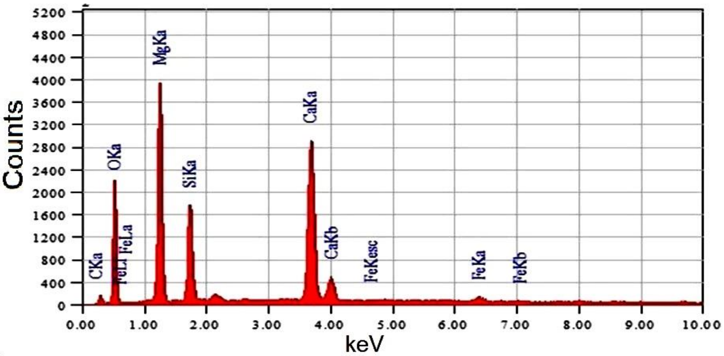

Fig. 6**b**: EDS scan of indigenous kaolinite ore activated at 600 °C for one hour showing lower counts of elements left over in it due to mass loss

Removal of H_2O is the main reason of mass loss in the indigenous kaolinite ore, resulting in the formation of micro pores within ore, thus increase in surface area. As the surface area increased the capability of adsorption also increased after activation process.

3.2 Characterization of Lube Oil

3.2.1 Colour index

The colour index value of the used lubricant oil was greater than 8. High colour index suggests presence of burnt additives and other contaminants in lube oil.

The colour index value of re-refined oil using kaolinite adsorbent was 2 (**Table 1**) that is within acceptable range of colour index 1-3. Improvement in colour index confirmed that the burnt additives had been removed.

3.2.2 Specific gravity

Specific gravity values of used and re-refined lubricant after first and second pass were 0.9128, 0.8722, and 0.8802 respectively (**Table 1**). Higher specific gravity of used lube oil was due to the presence of lower carbon chains and gummy materials. After re-refining decrease in specific gravity values is an indication that the contaminants have been removed.

3.2.3 Viscosity

The viscosity of used lube oil was found to be 87.28 centistokes per second (cSt/s) which is a very high value as compared to the 47.24 cSt/s for extracted base lubricant oil after first pass and 45.74 cSt/s after second pass (**Table 1**). This proved that the proposed adsorption process is very efficient in removing varnish, gummy and sludgy materials.

3.2.4 Flash point and ignition 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 used oil and 210 °C (410 °F) for the re-refined lubricant oil (**Table 1**).

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 (**Table 1**), which might be due to the presence of acidic impurities and oxidation and degradation products at high temperatures inside the engine.

The Nil value of TAN in the refined oil suggested that the impurities have been removed completely and no acidic nature impurity was left in the oil anymore.

3.2.6 Total base number (TBN)

Testing results indicated that the used lube oil had TAN equals to 5.02 mg HCl/g oil (**Table 1**) confirming the 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 the complete removal of those interfering compounds and proved the production of base lube oil of better quality.

3.2.7 Carbon residue

Adsorption process brought remarkable decrease in carbon residue (from 1.87 to 0.13 wt%), indicating kaolinite as efficient adsorbent for used lube oil (**Table 1**). The reduction in carbon residue value of extracted oil confirmed that the applied process is very much efficient for refining of the used oils.

3.2.8 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 the sulphuric acid and wear metals had been removed to a great extent (0.75 to 0.015 wt%) and the remaining amount of sulphated ash in the re-refined oil is almost negligible (**Table 1**).

Table 1: Properties of used and recovered oil by column chromatography using n-hexane as mobile phase, solvent to oil ratio is 2:1

Properties	**Used oil**	**Recovered oil By column Chromatography Using n-hexane**	
		First Pass	**Second Pass**
Specific gravity at 35°C	0.9128	0.8722	0.8802
Viscosity at 40°C (cst)	87.28	47.24	45.74
Sulphated ash (w%)	0.75	0.015	0.016
Total base number (mg HCl/g oil)	5.02	Nil	Nil
Total base number (mg HCl/g oil)	2.27	Nil	Nil
Carbon residue (wt%)	1.47	0.13	0.14
Color (ASTM D 1500)	>8	2	2
Flash point (°C)	185	204	202
Ignition temperature (°C)	204	210	
Yield (%)	-	95	94.8

3.2.9 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 [2].

Comparison of metal content in finished oil and used oil is an indication of usefulness of the process. Kaolinite is an excellent adsorbent for removal of metals. When the used lube oil was passed with n-hexane over the surface of activated kaolinite ore, the metal content in re-refined oil wass almost negligible (**Table 2**).

Table 2: Metals content (mg/kg ± 0.1) in used and recovered oils

Metals	Used oil	Recovered oil
Fe	70.4	0.3
Na	52.1	0.1
Mo	25.6	0.1
Al	14.2	0.4
Cr	4.2	0
Cu	7.3	0.1
B	6.0	0
Ni	04	0
Ag	0.2	0
Ba	0.2	0
Ca	1233	3.9
Zn	755	0.2
P	576	0
Mg	145	0.5
Pb	141	0
Total (mg/kg)	3030.6	5.7
Reduction (%)		99.81

This may be because of non-polar behaviour of n-hexane and high adsorption capability of kaolinite ore which did not allow metals to elute. **Table 2** shows that the metal content has been removed up to 99.8 % which was a remarkable decrease in the metal impurities. It reduced iron content more than using distillation, acid and activated charcoal with clay methods [14].

Chromium, zinc and copper content in refined oil is less than previous studies related to reclamation of used lube oil process [28]. 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.

3.2.10 Fourier transform infrared spectroscopy (FTIR)

The FTIR spectrum of used lubricant oil and extracted base lube oil have been overlaid to observe the difference in peaks of several ingredients (figure 7). This shows the impact of our adsorption technique on re-refining and the quality of the extracted base lube oil obtained after the sorption process.

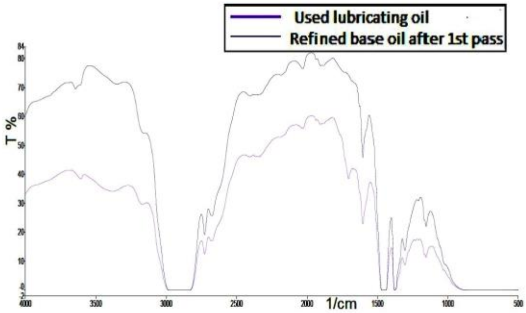

Fig. 7: FTIR bands of used lube oil and extracted base lube oil showing peaks of additives present in it

The FTIR analysis shows different peaks that might be the characteristic peaks of some compounds. The spectrum showed peaks at 1154 cm^{-1} and 1743 cm^{-1} which proved of presence of carboxylic acid or carboxylic compounds in the oil sample [5, 29].

The peak found at 1376, 1445 and 1462 cm^{-1} are the peaks of small chain alkane compounds. The peak present at 1603 cm^{-1} is indicative of nitration products which are responsible for the dark color in oil. It reduced to a great extent in the refined oil due to elimination of nitration contaminants [5].

The peak at 1705 cm^{-1} is due to the presence of succinimide type dispersant present in the lube oil while at 1900 cm^{-1} there are chances of the peak of CH_2 group of base oil [30]. The absorbance at 2725 cm^{-1} proves the presence of aldehydes in the oil sample which is a degradation product [5].

The FTIR spectra also shows that the organic impurities have not been removed completely but the concentration of those impurities have 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 because their presence may reduce the amount of same additives required to reprocess the base oil for next use.

Also the refining process was repeated thrice for the same adsorbent after activation and re-activation. The oil obtained each time was analyzed by FTIR for its quality and eventually no difference was found in the quality of extracted lube oil. Hence the oil quality remained the same for each pass (1st pass, 2nd pass and 3rd pass).

3.2.11 Regeneration of adsorbent

After the adsorption process the exhausted kaolinite ore was collected back in a china dish and regenerated again at 600 °C for one hour and reused for the adsorption process again. The process was repeated twice for the same adsorbent and similar quality of base lube oil was obtained after each regeneration.

The oil collected after each adsorption step was named as 1st pass and 2nd pass. Thus the regeneration of adsorption also reduced the cost of re-refining.

4. CONCLUSION

Indigenous kaolin clay containing high amount of kaolinite was used for re-refining of used lubricating oil. The used lubricating oil was refined through single step adsorption process which reduced the cost and time of re-refining process. n-hexane was used as solvent and experiment was performed at the minimum oil to solvent ratio of 1:2 v/v.

To increase the surface area of indigenous kaolinite ore was activated at 600 °C for one hour. Maximum adsorption capability of ore was determined by several small column runs. Reuse of adsorbent after regeneration and reuse of solvent after their recovery by rotary evaporator further reduced the process cost. Indigenous kaolinite ore was characterized by TGA, EDX, SEM, XRD techniques to find out structural changes occurred due to thermal treatment.

Analysis of re-refined lube oil showed that the color index decreases from >8 to 2 indicating removal of impurities due to their adsorption onto indigenous kaolinite. Up to 99.81 % of metal reduced after adsorption treatment which proved the authenticity of this single step process.

The results of specific gravity, viscosity, colour index, TAN, TBN, carbon residue, sulphated ash, flash point and FTIR also confirmed the production of better quality oil using low cost indigenous adsorbent.

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