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Recycling Used Lubricating Oil Using Untreated, Activated and Calcined Clay Methods

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Abstract: This study addresses recycling of used lubricating oils treated using different methods involving untreated clay as the control sample, clay activation and calcined clay methods. A recycling process of the used lube oil was carried out which eventually led to comparison of results of the recycled oil with the fresh lube oil using ASTM standards. Tests carried out on the used, fresh and recycled lube oils include: flash point, kinematic viscosity at 40 and 100°C, respectively, specific gravity/density, appearance and total base oil. The results show that recycled lubricating oil had the following properties: flash point was 227°C for the calcined clay, 229°C or activated clay and 224°C for untreated clay, compared with 204°C for untreated waste oil. Also Ttal Bse Number TBN) gave the following: 6.25mg $KOHg^{-1}$ for calcined clay method, 6.24 mg KOH g^{-1} for activated clay and 5.56 mg KOH g^{-1} for untreated clay method, compared with 4.41 mg KOH g^{-1} for untreated waste lubricating oil. This gives the recycled lube oil the potential to be reused in car engines after adding the required additives.

Key words: Used lubricating oil, refined lube oil, activated clay, calcined clay, environmental pollution

INTRODUCTION

Lubricating oils are viscous liquids of petroleum origin, used for the protection of moving parts of engines and machines (Boyde, 2002). Typical lubricating oil contains 90% base oil, most often petroleum fractions calledmineral oils and <10% additives. The chemical breakdown of these additives during use resulted to build up of halogenated hydrocarbons in the oil. Polycyclic Aromatic Hydrocarbons (PAHs) as well as other polycyclic compounds are generated and accumulate inthe oil (Wong and Wang, 2001) together with metals from wear and tear of the engine being lubricated (Chung *et al*., 2007). These compounds gradually reduced its quality leading tochange in its physical and chemical properties and thereby deteriorated (Kamal and Khan, 2009). These components are highly toxic upon released to the environment posing harmful effects toaquatic lives and human beings (Kanokkantapong *et al*., 2009). The improper disposal of used lubricating oil pollutes environment to a great extent as each volume of it can pollute not lessthan two hundred and fifty thousand volumes of water (Udonne, 2011). Proper management of this hazardous material is therefore important in order to make it a valuable product bygreatly reducing the quantity being disposed improperly (Dang, 1997). Therefore, re-refining of used lubricating oil will eliminate the environmental threats posed and preserves crude oilreserves (Durrani *et al*., 2011). Several techniques are available for the regeneration of used lubricating oil, among which are chemical treatment (Ogbeide, 2010), acid activated process that is similar to acid-clay process with little modification, physical treatment by distillation and thin film evaporation (Brinkman *et al*., 1981) and solvent extraction (Katiyar and Husain, 2010).

The purpose of this research is to obtain a high quality production of lubricating oil from used lubricating oil by the re-refining of it, thereby reducing environmental pollution caused by used lube oil. This process makes, it essential for the used lube oil to be re-used to perform different functions such as for lubricating moving part of engines and machine, reduce friction, transfer heat, carry away contaminants and debris and transmit power.

The conventional steps in lubricating oil manufacture are pretreatment of the crude oil charge, followed by distillation of the crude in two steps (an atmospheric tower and vacuum tower), de-asphalting (as required by the nature of the crude oil charge), de-waxing, solvent extraction, filtering and blending including mixing various additives with the final lubricating oil. Recycling of used lubricating oil is the process of regenerating its used substance so that it can be used again. It is the process involving the removal of the impurities in the used oil and bringing it back to the initial state The conventional

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methods of recycling of waste engine oil either requires a high cost technology such as vacuum distillation or the use of toxic materials such as sulfuric acid. These methods also produce contaminating by products which have high sulfurlevels.The adsorption treatment of waste lubeoil is based on the ability of adsorbent to selectively extract resinous and sulfur containing compounds, unsaturated and polycyclic material and also organic residues of sulfuric acid and solvents from oils. Natural clay, activated and calcinedare used as adsorbent in the treatment of oils. There are basically two treatment methods and they include contact treatment and percolation or filtration through a bed of granulated adsorbent.

MATERIALS AND METHODS

Clay soil, filter paper, fresh premium motor engine oil, used premium motor engine oil and distilled water which was used throughout the experiment for sample preparation and dilution.

Collection of test samples: Clay soil was collected from Iraye-Epe area of Lagos state, Nigeria. The test sample of fresh lube oils (premium motor oil SAE-40) was collected from MRS oil service station. The used lubrication oil was collected from a car mechanic's used oil dump in Iba-Ojo area of Lagos, Nigeria.

Apparatus/equipment: Apparatus and equipments used include conical flask, beaker (Pyrex), hydrometer, plasticbowls, aluminum pan, Bunsen burner, weighing balance, oven, mechanical shaker, Thermostatbath (FALC WB-MF Model).

Experimental procedure: The methods used in the re-refining of the used lubricating oils include methods such as, filtration process to remove impurities, clay preparation before clay activation and calcined clay soil.

Filtration process: The used lubricating oil was filtered to remove impurities such as metal chips, sand, dust, particles, micro impurities that are contained in the lube base oil. This was done using a funnel with a filter paper placed in it.

Clay preparation: The clay sample was first ground and then made into slurry with distilled water. Impurities such as sand and stones were first allowed to settle at the bottom in plastic bowls and were then removed by decantation. The slurry was kept in an oven at temperature of 110°C and allowed to dry up. The dried clay was again ground into very fine particles and sieved to a mesh of 0.5 mm using test sieve on a mechanical sieve shaker. The clay soil was then sub-divided into three portions to be used for untreated clay, activated clay and calcined clay preparations.

Untreated clay: This is the portion of clay without chemical activation after the clay sample was ground, washed and dried in the oven at 110°C.

Clay activation: The 200 g of clay (after dirt, sand and stone have been removed) was made into slurry with 80 cm^3 of distilled water and followed by the addition of 60 mL of 0.35 M solution of sulfuric acid. The slurry was then poured into aluminum pan and left for 1 h at temperature 100°C. The slurry was then washed several times with distilled water in order to remove any excess acid, until it was neutral. The washed clay mixture was dried in an oven for 1 h and grounded into powdery form (Oboh and Aworh, 1991).

Calcined clay: The third portion of the clay sample from section 2.3.2 above was then calcined by heating the clay soil in a furnace at about 600°C for 5 h.

Purification process of the used oil: The used engine oil was purified using the various clay samples. Beginning with the activated clay, it was ground and mixed with the used engine oil that had been pre-heated for 5 min. The mixture was then thoroughly blended together with the aid of a stirrer. The mixture was kept 24 h to allow for prolonged contact and chemical reaction between the spent oil and the activated clay. The impurities in the used engine oil and the clay particles were allowed to settle and with the aid of sieve cloth, the oil was sieved to obtain the purified lube oil sample. The purification of the used lube oil was repeated using other clay types.

RESULTS AND DISCUSSION

The results of the research research which investigates the recycling of used lubricating oil and compared with the fresh lubricating oil sample using three different clay methods have been carefully detailed in Table 1 and Fig. 1-7. The Table 1 also includes the results of the various quality tests (appearance, viscosities at 40 and 100°C, flash point, specific gravity, density and total base number) performed using ASTM methods on the lubricating oil products.

Table 1: Summary of analysis of result

Tests	Unit	Test method	Specification	Fresh oil	Used oil	Untreated clay method	Clay activation method	Calcined clay method
Appearance	Visual	Bright and Clear	Dark	Dark	Dark	Dark	-	-
Flash point	(°C)	ASTM-D0092	Min. 225	230.0	204.0	208.0	229.0	227.0
Viscosity kin, 40°C	(CST mm sec^{-1})	ASTM-D0445	130-150	146.50	135.52	139.83	145.77	144.41
Viscosity kin, 100°C	(CST mm sec^{-1})	ASTM-D0445	12.5-16.3	14.70	12.87	13.44	14.32	14.11
Viscosity index	(CST mm sec^{-1})	Calculated		99.17	85.38	89.59	95.62	94.11
Total base number	(Mg KOH g^{-1})	ISO 3771	7.00-10.00	8.50	4.41	5.56	6.26	6.20
Density	(Kg m^{-3})	ASTM-D1298		896.5	892.0	894.6	896.0	895.9
Specific gravity	(g cm^{-3})	ASTM-D1298		0.8965	0.8920	0.8946	0.896	0.8959

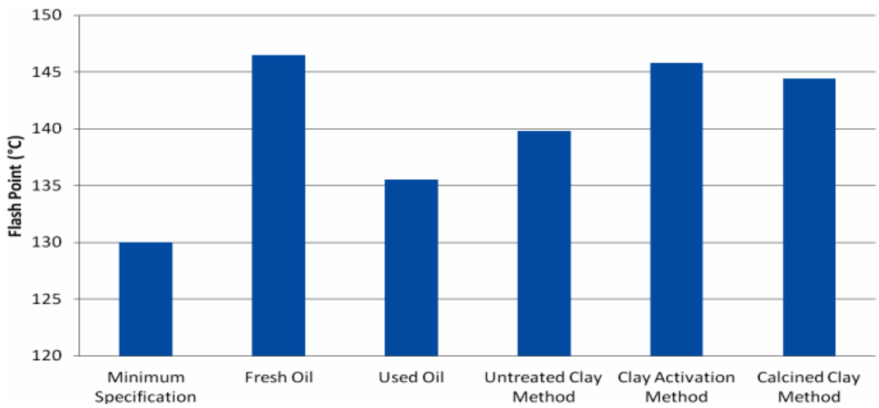

Fig. 1: Effects of the recycling of used lube oil on its flash point

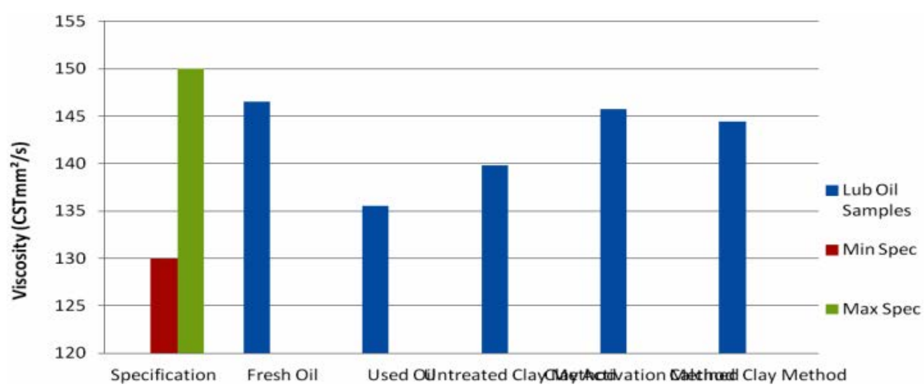

Fig. 2: Effects of the recycling of used lube oil on its viscosity at 40°C

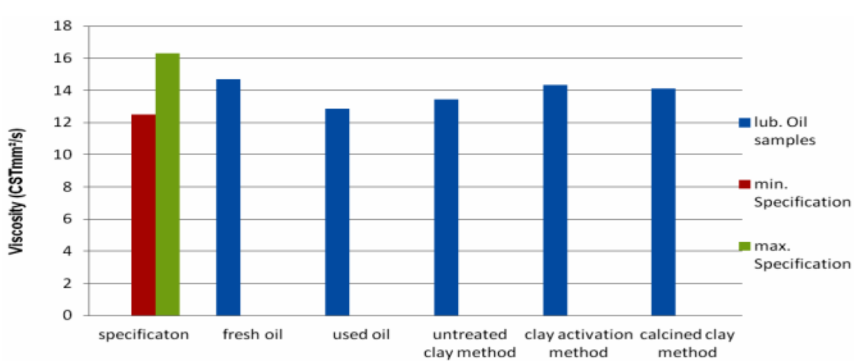

Fig. 3: Effects of the recycling of used lube oil on its viscosity at 100°C

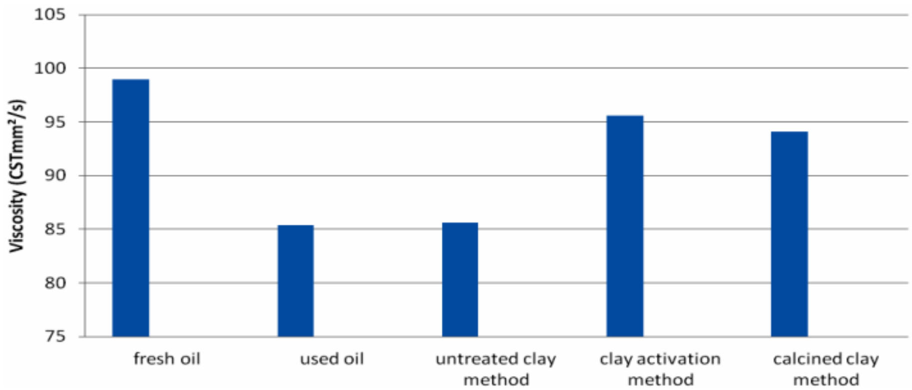

Fig. 4: Effect of recycling of waste used lube oil on its viscosity index

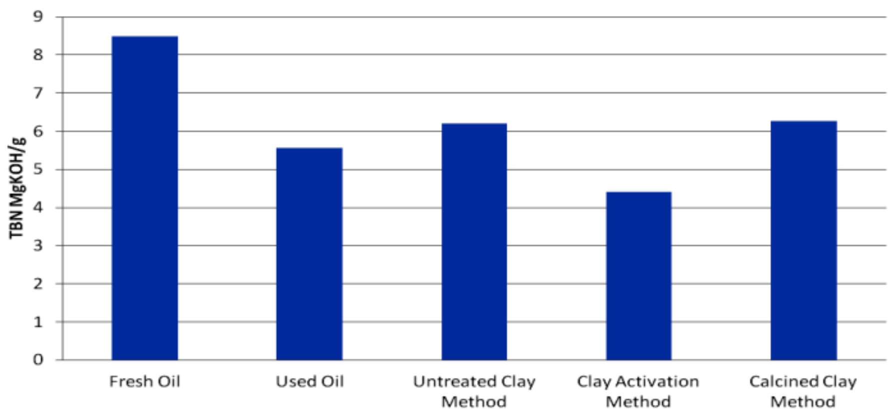

Fig. 5: Effect of recycling of used lube oil on its Total base oil

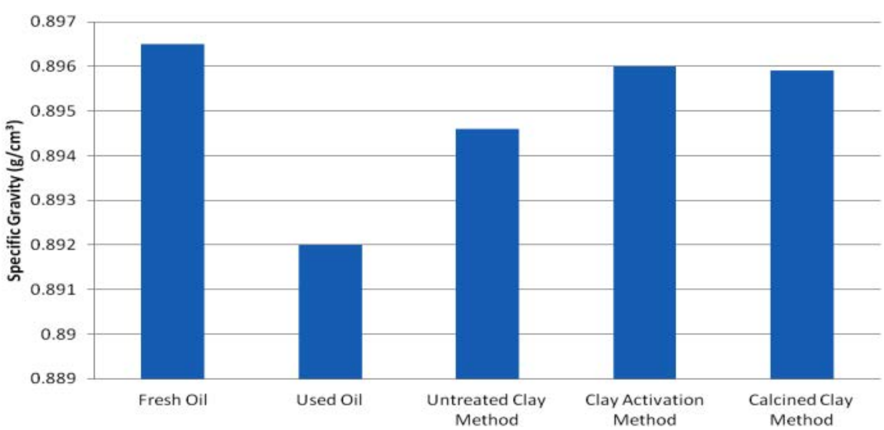

Fig. 6: Effect of recycling of waste used lube oil on its specific gravity

Appearance: This indicates the brightness and clarity of petroleum products. The results of the appearance (Table 1) of the fresh lube oil, used lube oil and the treated lube oil show that the fresh oil have a clear and bright color while the used and the treated lube oil have a dark appearance. The dark appearance of the used lube oil and those from the treated methods were due to their combustion and oxidation at high temperature by the combustion engine for a very long time.

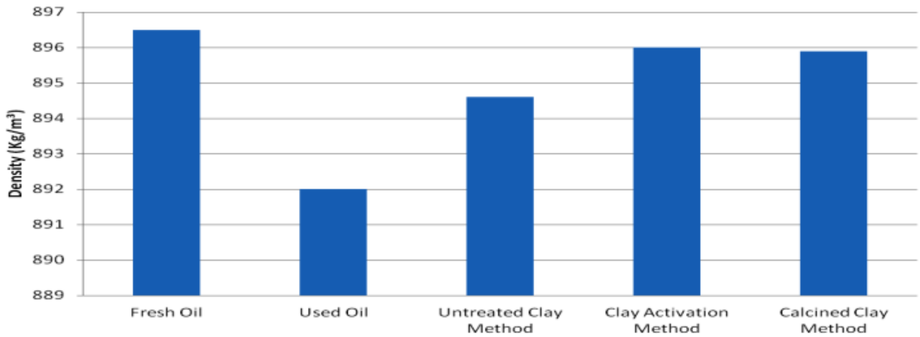

Fig. 7: Effect of recycling of used lube oil on its density

Flash Point: The flash point indicates how easy lubricating oil may ignite and burn. The flash point of lubricating oil is the lowest temperature where it will evaporate enough fluid or vapor to form a combustible concentration of gas with air that can be ignited spontaneously by a specified flame.

The result of the flash point of the fresh lube oil, used lube oil and the treated lube oil above shows that, there was a decrease in the used lubricating oil which is below the minimum specification of SAE-40 lube oils. The decrease in the used lube oil was due to contamination of very volatile component. On treating, the volatile components were extracted from the used lube oil. There by increasing the flash point above the minimum specification of SAE-40 except the untreated clay method.

Viscosity at 40°C: The results (Table 1) of the Kinematic Viscosities (KV) at 40°Cusing the ASTM-D 0092 Method shows that the waste used oil sample with Kinematic Viscosity (KV) of 135.52 CST mm sec^{-2} and those of the recycled lube oil are as follows from the untreated clay method, KV is 139.83 CSTmm sec^{-2}, from Clay Activation Method, KV is 137.41 CSTmm sec^{-2} and from calcined method, KV is 139.77 CST mm sec^{-2}. The plot of kinematic viscosities of each of the lube oils (Fig. 2) reveals thatthey all fell within the required specification range (Brinkman *et al*., 1981). This shows that at 40°C, the waste used oil and the recycled methods still retained the viscosity specification range (130-150 CST mm sec^{-2}).

Viscosity at 100°C: The result of the kinematicviscosity at 100°C shows that there is a decrease in the viscosity of lube oil as the temperature is increasing. The result also indicates that, the used lube oil have the least viscosity compared to the recycled and fresh lube oils. This may be due to contamination of a very volatile component (light oil) present in the oil. After treatment, the volatile components were extracted from the used oil and consequently increased the viscosity of the lube oils as shown in Table 1. The plot of kinematic viscosities of each of the lube oils (Fig. 3) reveals that they all fell within the required minimum and maximum specification limits of lube oils.

Viscosity index: The viscosity index is obtained by electronic software which depends on the kinematic viscosity of the samples at 40 and 100°C, respectively and shows a linear relationship. The result also indicates that, the used lube oil have the least viscosity index which indicate the dilution of light oil or very volatile component but after treatment, the volatile components were extracted to an extent which then increased the viscosity index of the lube oil. Figure 4 shows the various viscosity indexes of four lube oil samples compared to the fresh oil sample.

Total base number: Total Base Number (TBN) is a measure of reserved alkaline additives put into lubricants to neutralize acids, retard oxidation and corrosion, enhance lubricity, improve viscosity characteristic and reduce the tendency of sludge buildup. It is a test to measure the ability to neutralize corrosive acids that may be formed during normal operation.

Internal combustion engine oils are formulated with a highly alkaline base additives package to neutralize the acidic products composition. The TBN is a measure of this package and it may be used as an indication for the engine oil's replacement time. This is because TBN depletes with time in service. Higher oil TBN values are more effective at neutralizing acids for longer periods of time (Dong *et al*., 2001). The results of the analysis of the total base number show that, there is a decrease of the TBN of used oil and those from the recycled methods compared to that of the fresh oil. This is because some amount of base has been used up to neutralize the acid formed during usage of the oil. After treatment there was an increase of the total base number from 5.56 (used lube oil) to 6.20 for oil from the untreated clay method, 6.26 for oil from the calcined clay method except for the clay activation method where a decrease in TBN (4.41) was noticed. But in comparison with the TBN (8.20) of the fresh lube oil, there is a decrease TBN of lube oil in all the methods used. This may be due to the presence of the metallic and acidic elements present in the clay sample. The decrease is however, more pronounced in the clay activation method because of the 60 cm^3 of concentrated sulfuric acid (H_2SO_4) added during the clay activationstage. Hence, the increase in the quantity of acid present during activation,leads to a decrease in the total base number of the lube oil. The graph is shown in Fig. 5.

Specific gravity/density: The specific gravity of contaminated or used lube oil could be lower or higher than that of its fresh lube oil depending on the type of contamination. If the used lube oil is contaminated by dilution of light oil/volatile component or contaminated due to fuel dilution and/or water originating from fuel combustion in the engine and accidental contamination by rain, its specific gravity will be lower than that of its fresh lube oil. But, on the other hand when the specific gravity of the used oil is greater than that of the fresh lube oil, then the lube oil may have been contaminated heavy oil or component with a higher carbon atom. The results for the fresh and used lubricating oils are 0.8965 and 0.8920, respectively. This shows that the used lube oil may have been contaminated by dilution of light oil. It is also very likely that some of the heavier components of the lube oil had broken down to lighter fractions at the high operating engine temperature. After undergoing different treatments by the various methods, the volatile components were extracted whichconsequently increased the specific density of the re-refined oil obtained to: 0.8946, 0.8960 and 0.8959 from untreated clay, activated clay and calcined clay methods, respectively. The plots are shown in Fig. 6 and 7.

CONCLUSION

This research has shown that used lubricating oil can be recycled using activated, calcined and untreated clay samples. Theresults show that there were contaminants of light oil/a very volatile components present in the used oil and it was shown that the three methods used removed the volatile contaminants from the used lubricating oil and returned the oil to a quality essentially equivalent to fresh lube oils. Judging from the results obtained, the most effective of the recycling methods were the activated clay and calcined methods.

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