



Recycling of Used Lubricating Oil Using Three Samples of Acids and Clay as a Method of Treatment

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ABSTRACT

This research investigated the effect of three different acids (sulphuric acid, hydrochloric acid and nitric acid), on three different clay deposit samples, to recycle used lubricating oil. To achieve this objective, filtration of the used oil was carried out first to remove impurities like: sand, metal chip and other micro impurities using: Buncher funnel, filter papers and vacuum pump. The filtered oil was allowed to settle for twelve hours (12hrs), before being centrifuged for another 20mins at 10000rpm so any particles in the oil can settle and be decanted. Next, the oil was preheated to degrade some additives, which helped to reduce the work load when acid was added. 100ml of the preheated oil was measured and treated with 10ml of various acids (Sulphuric acid, Nitric acid and hydrochloric acid) in varied strengths, and then allowed to stand for thirty minutes in a separating funnel before being allowed to pass through the bed of clay using a vacuum pump. Caustic Soda was then added to the extracted oil to help neutralize the acid in the oil. The resulting oil was slightly dark in colour. Tests carried out were: flash point, cloud point, pour point, viscosity index, sulphur content and water content. The result show that compared with the standard value of fresh lube oil, the resultant oil from the analysis compared favourably. An appreciable increase in the flash point, cloud point, viscosity index, and sulphur content of the oil was noted when compared to the fresh lube oil.

KEYWORDS: Lubricants, clay samples, environment, recycling, viscosity index

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INTRODUCTION

Lubricating oil are used to reduce friction on our engine, protection against wear and tear, dust, carry away heat and prevent contamination. Over a period of time, properties of lubricating oils are adversely affected by contamination that we tend to prevent [1] such as: soot, water, acids, dirt and fuel. Some contaminants are important to monitor and analyze because they are root causes of premature oil degradation and engine failure. Other contaminants are symptomatic of an active failure condition that requires a response other than just an oil change [2]. The recycling of used lubricants has been practiced to various degrees since the 1930s and particularly during the Second World War when the scarcity of adequate supplies of crude oil during the conflict encouraged the reuse of all types of materials including lubricants [3]. Environmental considerations regarding the conservation of resources have maintained interest in the concept of recycling up to the present day. The reclamation of spent crankcase oils is now a subject of pressing national interest in some countries. On the other hand, pollution by used lubrication oils is recognized now to account for greater pollution than all oil spills at sea and off-shore put together. Some countries have petroleum storage, and spent oils represent a precious commodity, which must not be wasted. It was because of this, the conservation of petroleum resources have been declared national policy for several countries and the benefit of wise resource management are obvious. Recent impetus on waste recovery leads to renewed interest in re-distilling to convert this used lubricating oil into useful original lubricating oils.

Re-distilling or re-refining is the use of distilling or refining processes on used lubrication oil to produce high quality base stock for lubricants or other petroleum products. The use of this method has increased tremendously in the developed countries; in some countries reaching up to 50% of the countries' need for lubricating oil [4-6]. In some developing countries however, recycling of used lubricants is yet to attract expected attention. Used lubricants are either dumped in the open, or used for such things as wood preservatives; further polluting the environment. Lubricating oils from petroleum, consists essentially of complex mixtures of hydrocarbon molecules mostly isoalkanes having slightly longer branches, and the monocycloalkanes and monoaromatics which have several short branches on the ring. Such hydrocarbon molecules generally range from low viscosity oils having molecular weights as low as 250, up to very viscous lubricants with molecular weight as high as 1000 and carbon atoms range from 20 to 34. Lubricating oils are viscous liquid used for lubricating moving parts

of engines and machines. Grease, which is a semi-solid, also belongs to this group. There are three major classes of lubricating oils, namely: lubricating greases, automotive oils and industrial lubricating oil. When lubricating oils are used in service, they help to ease the movement of connected parts. In the process, they serve as a medium to remove high build up of temperature on the moving surfaces. Further build up of temperature degrades the lubricating oils, thus leading to reduction in properties such as: viscosity, specific gravity, etc. Dirt and metal parts worn out from the surfaces are deposited into the lubricating oils. With increased time of usage, the lubricating oil loses its lubricating properties as a result of over-reduction of desired properties, and thus must be evacuated and a fresh one replaced. With the large amount of engine oils used, the disposal of lubricating oils has now become a major problem.

The basic principle remains the same and utilizes many of the following basic steps:

- 1) Removal of water and solid particles by settling.
- 2) Sulphuric acid treatment to remove gums, greases, etc
- 3) Alkaline treatment to neutralize acid
- 4) Water washing to remove "soap".
- 5) Clay contacting to bleach the oil and adsorb impurities
- 6) Stripping to drive off moisture and volatile oils
- 7) Filtering to remove clay and other solids
- 8) Blending to specification.

METHODOLOGY

COLLECTION OF CLAY SAMPLES

The experimental set up was intended to test the possibility of obtaining different levels of separation using clays from different deposits. This was done in order to verify if clay from different deposits can produce different levels of bleaching and adsorption of impurities from used lubricating oil. Clay samples were obtained from three deposits, Sample A (Ekiti) Clay, Sample B (Ondo) Clay, Sample C (Epe-Lagos) Clay, each about 200 kilometers apart.

Reagents used were: Sulphuric acid, Caustic Soda, Hydrochloric acid and nitric acid.

Equipment used: Buchner Funnel, Filtering Flask, Vacuum Pump, Beaker, Measuring cylinder, Water Bath, Filtering Paper, Conical Flask/Round bottom Flask, Separating Funnels, Sulfur Content analyser, Thermometer, Atomic Absorption spectroscopy, Ubbelohde Viscometer, Flash point Tester

COLLECTION OF OIL SAMPLE

The oil samples of used (Total Quartz 2000- SAE 40) were collected from a service station Lagos State, Nigeria. The used lubricant was obtained from a Saloon Car, whose oil has been used for a month and half respectively. Three litres of used oil was collected for the samples.

EXPERIMENT PROCEDURE

Purification of the used lubricating Oil

Filtration of the used oil was carried out to remove impurities such as sand, metal chips, micro impurities that contaminated the lube oil. This was done using a vacuum pump, Buchner funnel, and a filter paper. The Buchner funnel and the vacuum pump were connected to the filtering flask to which the Buchner funnel was fixed with a rubber stopper. Three litres of the used oil was filtered for the sample.

The lube oil was allowed to settle for twelve hours. The sample was further filtered by centrifugation for 20 minutes at 1,000 rpm and the suspended particles in the used oil allowed to settle at the bottom of the flask, and the liquid portion was decanted. The decanted liquid was preheated to degrade some of the additives and reduce the work load of the acid. 100 ml of the pre-treated oil was measured into three different beakers for the first sample A at 10 ml each. The first beaker was treated with 98% sulphuric acid (H_2SO_4), the second beaker with hydrochloric acid and third beaker with nitric acid. Each conical flask was shaken thoroughly to ensure homogeneous mixing of the acid, and the samples were allowed to stand for thirty minutes. The oil samples were measured into a separating funnel; acid sludge was discharged at the bottom of the separating funnel. This was removed gradually and repeated for the two other samples (B & C). 30 grammes of clay was packed in a Buchner funnel, with a filter paper inserted in it. This was plugged to a vacuum pump, allowing the oil to pass through the bed of the clay. 100 ml solution of 10% NaOH (Caustic soda) was added to neutralize the acid of the three oil samples. The process yielded refined oil which was dark in colour. The method was then repeated for the other three samples (B & C) using Sulphuric acid, Nitric acid, Hydrochloric acid respectively, and the refined lube oil obtained for analysis.

QUALITY TEST PROCEDURE

Flash point

Procedure

10ml of the three samples were measured in a 100ml separated beaker, each sample having different acid mixing. For sample A (a, b, c) each beakers were placed in a buchner burner and the lube oil was stirred. A flame source was brought at intervals to determine the temperature at which a flash would appear on the surface while the lube oil in the beakers is being heated at a specific rate. Same test was repeated for samples B (x, y, z) and sample C (m, n, o) the results are recorded in the table above.

Cloud point

20ml of each samples of the re-refined lube oil were poured into a conical flask and were introduced to an ice cold container. When the lube oil samples are chilled at a specified rate certain paraffin hydrocarbons in a wax form would begin to solidify and the temperature was recorded same procedure was repeated for other samples and the results are recorded in the table above.

Pour point

Procedure

This is same procedure with cloud point but the difference applies when further chilling continues until the lube oil first ceases to flow or pour and this point recorded as the pour point. The results are recorded in the tables above.

Water content

100ml of each sample were collected into a round bottom flask and was distilled. The volume of the water collected was recorded the result as a percentage by volume using the below method

Percentage of water (H₂O)

$$= \frac{\text{Volume of water trapped} \times 100}{\text{Volume of sample}}$$

The results calculated are recorded in the table above.

Sulphur content

The instrument used in this process is a spectrophotometer and the test carried out is known as sulfur analysis.

1ml of the sample for the three groups is introduced into a cuvette and attached to the spectrophotometer clip when the accurate position is achieved; radiation bottom is then allowed to emit its rays. The radiation obtained from x-ray tubes, the resulting excitation effect observed as the beam passes through the solution is measured as its merged from counts obtained from the standards. The results are recorded on the tables. This excitation effect is proportional to the concentration of sulphur in the sample as concentrations are generally obtained in 10% of the solution separately funnel, the process yields some quality of base oil produced by the acid/ clay process is usually dark in colour. The acid/ clay treatment method was carried out for both sample and the re-refined lube oil obtained were analyzed and the results were shown in table above.

Viscosity index

This analysis is carried out using a viscometer. The 9 re-refined samples obtained and the fresh lube oil were heated one after the other attaining a temperature of 100°C. The bulb of the viscometer was filled with the hot oil for the first sample to mark while immersed in a thermostat. The oil was drawn up by liquid level along the level mark. The liquid was then allowed to drain, and the time necessary for the liquid to fall from the point (A to B) was taken with a stop watch. The whole operation was repeated for the other 8 samples and the results were measured after the whole analysis. The viscometer was cleaned and the standard oil was added and the analysis was then repeated the time of drain of the oil were obtained.

Metal content

Metal content in re-refined lube oil was determined using atomic absorption spectrometry (AAS). Sample containing the metals to be analyzed was dissolved in water, when insoluble, digested in acid dissolved it. The cathode lamp for the element 1 was put in position and the element characteristic wavelength selected using a wavelength selector. Standard solutions of the element to be determined were first prepared and their absorbency measured at selected wavelength against the absorbency of a blank solution. The instrument gave directly the absorbency reading of the sample in solution.

RESULTS AND DISCUSSION

SUMMARY RESULTS OF RE-REFINED LUBE OIL										
	Fresh Lube Oil	Sample A (Ekiti Clay)			Sample B (Ondo Clay)			Sample C (Epe –Lagos Clay)		
	Standard Value	H ₂ SO ₄	HCL	HNO ₃	H ₂ SO ₄	HCL	HNO ₃	H ₂ SO ₄	HCL	HNO ₃
Flash Point	188°C	173°C	159°C	168°C	178°C	168°C	165°C	171°C	158°C	167°C
Viscosity Index	92.8	86.3	79.5	85.1	88.6	78.5	82.6	85.7	80.2	83.5
Sulphur Content %	-	0.042	0.040	0.044	0.041	0.046	0.043	0.041	0.047	0.045
Water Content v/v wt % <0.2	< 0.2	0.49	0.60	0.52	0.48	0.57	0.50	0.48	0.59	0.55
Pour Point	-9°C	-13°C	-18°C	-15°C	-11°C	-14°C	-14°C	-13°C	-12°C	-15°C
Cloud Point °C		-9°C	-14°C	-12°C	-9°C	-11°C	-12°C	-10°C	-9°C	-10°C
Specific Gravity	0.8966	0.8658	0.8013	0.8247	0.8735	0.8466	0.8762	0.8762	0.8346	0.8588

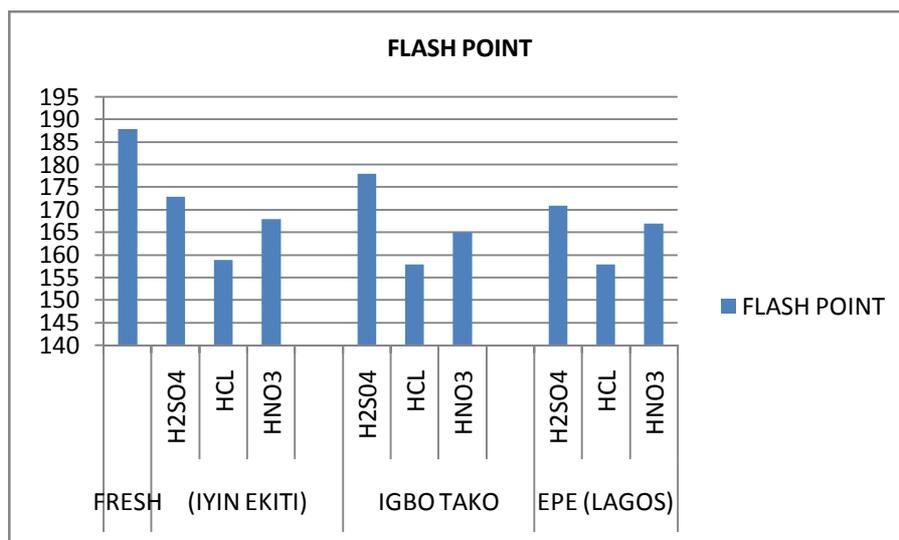


Figure 1. Effect of various acids on flash point of fresh and recycled lube oil.

The normal prescribed flash point for fresh lube oil is 188°C, for the 3 samples of clay used for this experiment, sample A 173°C, sample B 176°C and sample C 179°C. The decrease in value of flash point can be accredited to presence of light ends of oil (Udonne J.D., 2010). The chemistry behind this is that after undergoing combustion and oxidation at high temperature, the oil breaks down into component part, which include some light ends, from the result obtained in table 1, the flash point of the recovered lube oil is still acceptable in reference to standard.

Increase in viscosity of used lube oil can occur due to oxidation or contamination with insoluble matter while its decrease can be caused by dilution with light fuel, this might still be within an acceptable range. The viscosity index of lube oil will also change as a result of: temperature changes, thermal degradation leading to chain scission and dilution with fuel amongst others. From table 1. above, there is variation in the viscosity index of samples, with that of sample B which showed more pronounced viscosity index. This is also evident from fig.2, the clay sample (b) produced higher viscosity with sulphuric acid than with nitric acid or with hydrochloric acid.

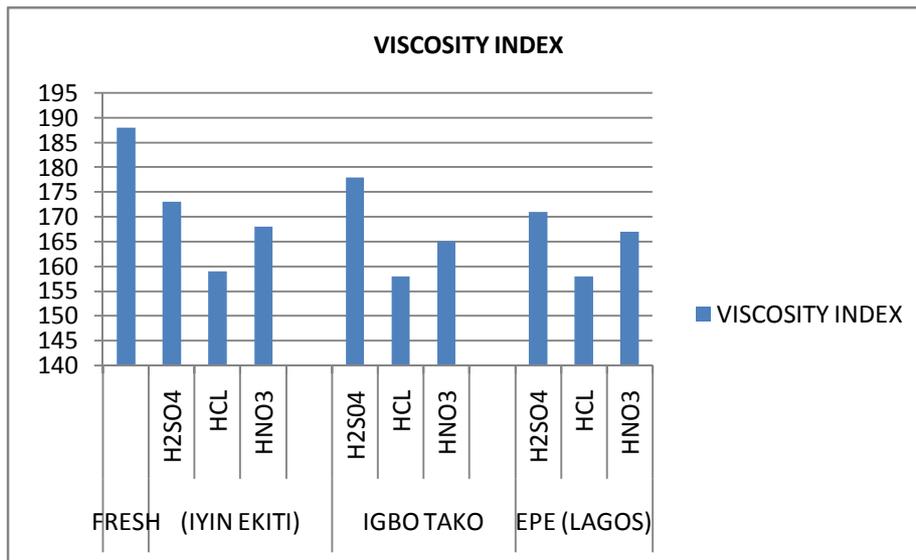


Figure 2. Effect of the various acids on the viscosity of fresh and refined oil.

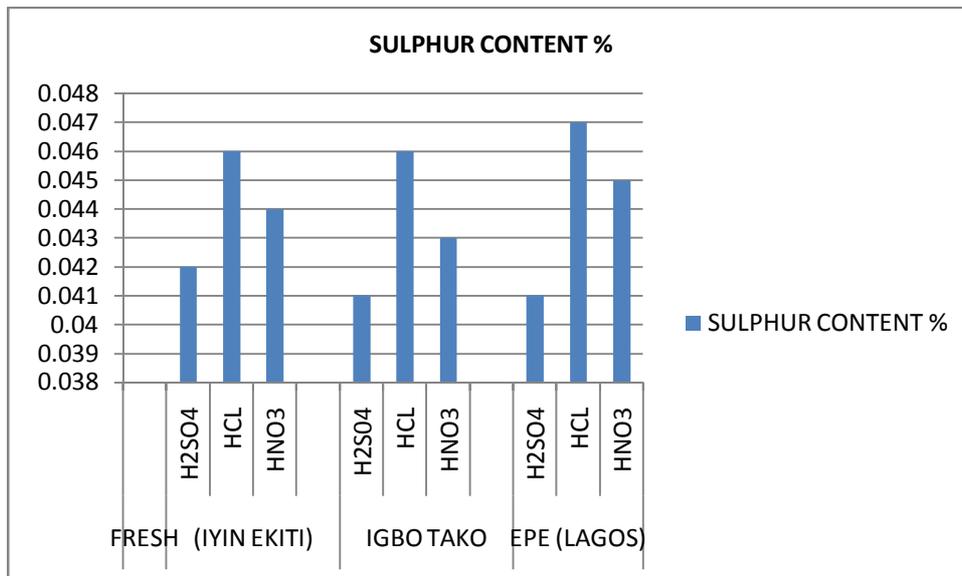


Figure 3. Effect of various acids on sulphur contents of fresh and recycled lube oil.

From fig.3, refined oil produced using clay sample (b) and sulphuric acid gave the least value of sulphur content, followed by sample (c) and (a) respectively. It is certain that usage of lube oil will definitely cause the sulphur content to increase, which is a function of the wear caused between the moving parts. Sulphur reacts with the metal to form compound of lower melting point that are more readily sheared without catastrophic wear. Engine corrode by mineral acid formed by oxidation of sulphur compounds in fuel internal combustion engine with refined oil; those hydrocarbon that were inherently unstable will have been oxidised during use. There is need to ascertain the minimum sulphur content required for maximum performance of lube oil.

The result show that sulphuric used with clay sample (b) produced the lowest water content level followed by (c) and (a) in that order. The level of water content in the samples fall within acceptable limits, that for fresh oil is less than 0.2.

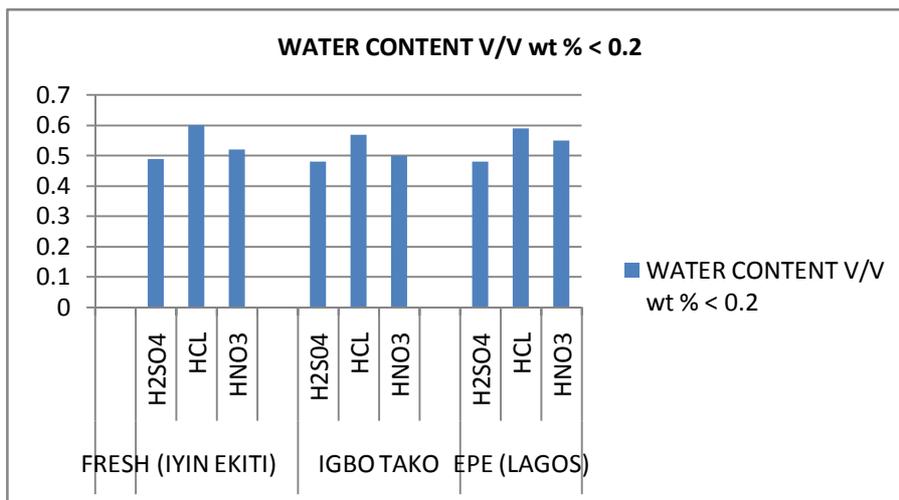


Figure 4. Effect of various acids on water content of fresh and recycled lube oil.

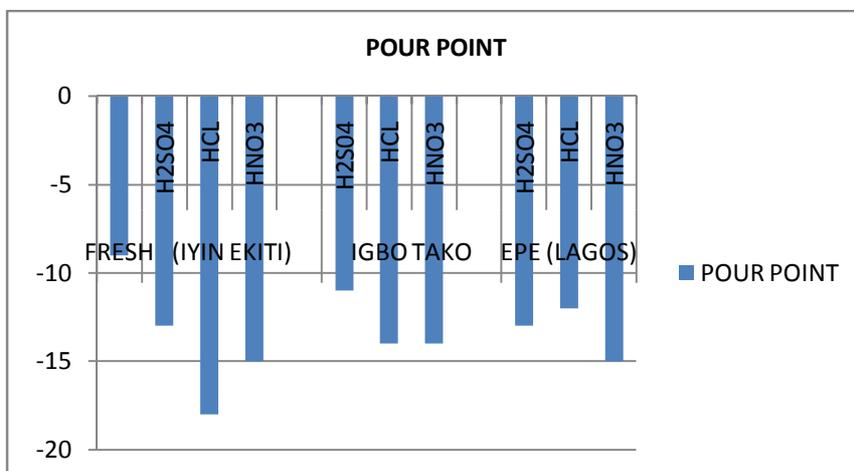


Figure 5. Effect of various acids on pour point of fresh and recycled lube.

The degradation of additives in lube oil causes the pour point of used lube oil to be high. The result obtain as shown in table 1 shows that the re-refined oil still possesses good pour point that can still be re-used, because pour point is important when considering oil under a cold weather or condition. Sample C shows more preferable clay to be used for Acid/clay treatment using concentrated H₂SO₄ as acid.

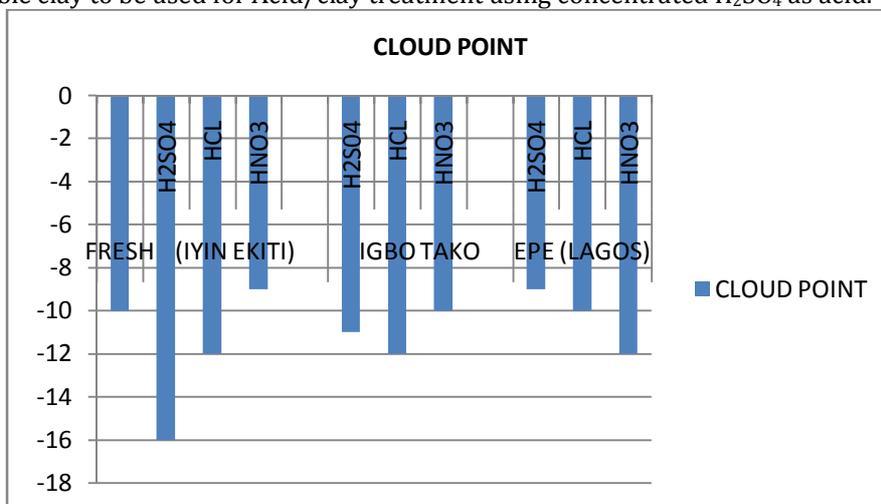


Figure 6. Effect of various acids on cloud point of fresh and recycled lube oil.

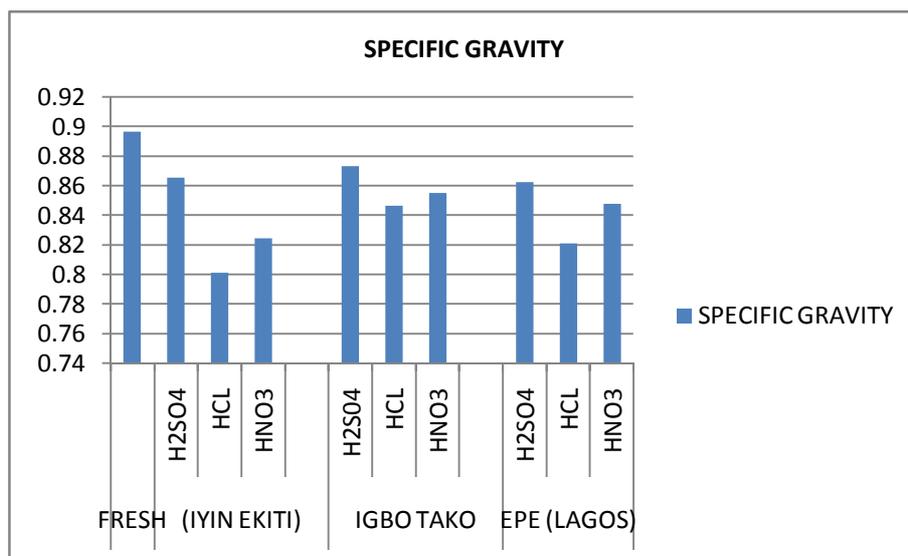


Figure 7. Effect of various acids on specific gravity of fresh and recycled lube oil.

The specific gravity of contaminated oil could be higher or lower depending on the type of contamination (Chevron lubricating oil FM ISO 100). The result obtained from sample A, B, C are 0.8658, 0.8568, 0.8762 respectively while that of fresh lube oil is 0.8966. This shows that sample C i.e. clay from Ondo state is more suitable for Acid/clay treatment method in order to obtain a better specific gravity to recover used oil.

CONCLUSION

From the result presented, it prove that the three acids use effectively activated and remove the slug from the used Lubricating oil and return the oil to it quality form. It is noted that the three clay samples possess high adsorption properties which enabled the removal of impurities and the black colour from the lubricating oil. Judging from the results obtained, Sulphuric acid yields 90% while Nitric acid yield 70% to 80%. It's noted that Nitric acid can be used in place of sulphuric acid and has proved to yield about 75% since it would cost less to obtain used oil. This would reduce the degree and nature of contamination, environmental/health risks associated with disposal. Economics (economic viability/sustainable market and commercial feasibility product value), Socio-economic benefits (i.e. employment opportunities) here in Nigeria. The major drawback to the acid/clay method is the difficulty of removal of the clay sludge.

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