

ISSN: 0794 - 4756



NIGERIAN JOURNAL OF ENGINEERING

Vol. 21 No. 2, March, 2015



Afowale
Eyitayo,
Amss



NIGERIAN JOURNAL OF ENGINEERING

Vol. 21 No. 2, March, 2015

Published by

Faculty of Engineering
Ahmadu Bello University
Samaru, Zaria,
Nigeria

Email: abufeng@abu.edu.ng



Editorial Board

Editor - In - Chief

Prof. S. A. Yaro

Dept of Metallurgical and Materials Engineering
Ahmadu Bello University, Zaria, Nigeria

Financial Secretary/Treasurer

Production/Business Manager

Dr M. T. Isa

Dept of Chemical Engineering
Ahmadu Bello University, Zaria, Nigeria

Secretary

Dr D. B. Adie

Dept of Water Resources & Environmental Engineering
Ahmadu Bello University, Zaria, Nigeria

Associate Editors

Prof. M. L. Suleiman

Dept of Agricultural Engineering
Ahmadu Bello University, Zaria, Nigeria

Dr A. Ocholi

Dept of Civil Engineering
Ahmadu Bello University, Zaria, Nigeria

Dr B. Jimoh

Dept of Electrical Engineering
Ahmadu Bello University, Zaria, Nigeria

Dr D. Yawas

Dept of Mechanical Engineering
Ahmadu Bello University, Zaria, Nigeria

Dr M. Abdulwahab

Dept of Metallurgical and Materials Engineering
Ahmadu Bello University, Zaria, Nigeria

Dr I. A. Mohammed-Dabo

Dept of Chemical Engineering
Ahmadu Bello University, Zaria, Nigeria



1. ANALYSIS OF CLIMATE VARIABILITY AND ITS IMPACT ON CROP YIELD IN ZARIA 1 - 11
A. Adamu, D. B. Adie and C. A. Okuofu
2. VALIDATION OF PLANTING PERFORMANCE OF A MECHANICAL PLANTER ROBOT 12 - 23
K. Ahmad, M. B. Muazu and D. D. Dajab
3. STRESS CORROSION CRACKING OF AUSTENITIC STAINLESS STEELS IN CHLORIDE ENVIRONMENT 24 - 28
I. I. Ahmed, A. G. F. Alabi, J. K. Odusote, N. I. Aremu, J. A. Adebisi, T. Yahaya, S. I. Talabi, R. A. Yahya and S. B. Lyon
4. CALCINATION KINETICS OF OKPELLA LIMESTONE FROM THERMOGRAVIMETRIC DATA FOR LIME PRODUCTION 29 - 38
M. N. Bello, J. O. Okafor and A. Mukhtar
5. STUDY OF E-WASTE GENERATION AND MANAGEMENT IN HOUSEHOLDS WITHIN KADUNA METROPOLIS 39 - 47
F. B. Ibrahim, D. B. Adie, A. Giwa and C. A. Okuofu
6. PROMOTING TRANSESTERIFICATION OF COTTON SEED OIL (CSO) BY USE OF CO-SOLVENT 48 - 53
F. Iwalaiye, B. O. Aderemi, M. S. Galadima and C. S. Ajinomoh
7. MODELLING OF FLOW OVER OBLIQUE COMPOUND CRESTED WEIRS 54 - 59
M. M. Muhammad, A. Ismail, J. A. Otun and D. B. Adie
8. APPROPRIATE WATER PUMPING SCHEDULE AND THE DETERMINATION OF THE REQUIRED STORAGE CAPACITY 60 - 65
I. M. Sanni, D. B. Adie, A. Ismail and M. O. Nwude
9. EFFECTS OF THERMAL TREATMENT ON THE HARDNESS OF ANTIMONY-MODIFIED AL-SI-MG ALLOY 66 - 70
G. O. Adeboye, M. Abdulwahab, F. Asuke and S. A. Yaro
10. EFFECTS OF ACID TYPES ON THE RECYCLING OF USED LUBRICATING OIL 71 - 80
B. Suleiman, A. S. Abdulkareem, E. A. Afolabi and E. O. Oluwaseyi
11. THERMO-ECONOMIC ANALYSIS OF THIDC-PSA SYSTEM FOR BIOETHANOL REFINING 81 - 91
B. Suleiman, A. S. Olawale and S. M. Waziri
12. EFFECT OF SILICA SOURCE ON THE FORMATION OF NANOSIZED ZEOLITE Y VIA SEEDING METHOD 92 - 97
S. Yunusa, S. A. Abdulkarim, G. B. Solomon, F. I. John and D. Mohammed
13. CLASSIFICATION AND CHARACTERIZATION OF BULARAFA DIATOMITE USING AIR CYCLONE 98 - 102
M. Abubakar, A. S. Ahmed, I. A. Mohammed-Dabo, S. G. Bawa and F. Mohammed



EFFECTS OF ACID TYPES ON THE RECYCLING OF USED LUBRICATING OIL

*B. Suleiman¹, A. S. Abdulkareem^{1,2}, E. A. Afolabi¹ and E. O. Oluwaseyi¹

¹Department of Chemical Engineering, School of Engineering and engineering technology, Federal University of Technology Minna PMB 65, Niger State, Nigeria.

²Centre for Genetic Engineering and Biotechnology, Federal University of Technology Minna PMB 65, Niger State, Nigeria.

*Corresponding Author: E-mail: bilyaminusuleiman@yahoo.com Cell: +2347036970741

ABSTRACT

This study focuses on the effects of acid types on the recycling of used lubricating oil. Three different acids, H₂SO₄, HCl, and HNO₃ were used for the treatment of used lubricating oil and subsequent neutralization of the oil with NaOH. The properties of fresh and used oil tested for were kinematic viscosity at 40 °C and 100 °C, viscosity index, flash point, pour point, specific gravity at 40 °C, water content, sulphur content, total base number (TBN), and metals present (Pb, Zn and Cu). The result obtained revealed that the quality of lubricating oil was affected after usage as a result of its property degradation and presence of heavy metals such as Pb, Zn and Cu. The result obtained shows that treatment of used oil with acids greatly improved the quality of the used oil. The extent of the recycled oil quality improvement and its yield varied with acid type. The result showed that flash point increased from 220 °C for used oil to 232 °C, 225 °C and 227 °C for oil sample recycled with H₂SO₄, HCl and HNO₃ respectively. This is compared with 245 °C for fresh oil. Also, viscosity index increased from 91.2 for spent oil to 125, 116 and 119 for oil sample recycled with H₂SO₄, HCl and HNO₃ respectively. It was also compared with 127 of fresh oil. Sulfur content was reduced to 0.046, 0.2 and 0.35 wt % using H₂SO₄, HCl and HNO₃ for used oil treatment. In general, based on the various analyses conducted in this study, acid treatment efficiency of used lubricant with H₂SO₄ is the best followed by HNO₃ and HCl respectively. The use of H₂SO₄ also gives the highest yield of 70 % recycled oil.

1.0 INTRODUCTION

Lubricant is an oil or grease that is applied as a surface coating to moving parts to protect and reduce friction of two surfaces in relative motion, eliminate temperature build up and keep the engine clean (Toolingu, 2013; Udonne 2011; Ogbeide, 2010). It also serves the purpose of transportation of foreign particles, heat transfer, corrosion prevention, cutting of metal and protection against wear (Udonne 2011; Ogbeide, 2010). The lubricating oils are either bio or petroleum based and the petroleum base feedstock are mainly complex mixtures of hydrocarbon molecules (Udonne, 2011; Hamad *et al.*, 2005) ranging from low viscosity oils to high viscosity lubricating oil. Analysis shows that the presence of impurities depends on the petroleum process and production method but concluded that lube oil contains aromatics in the range 4 to 12 % because nearly all the sulphur and nitrogen in an oil are present in its aromatic structures (Oluwaseyi, 2014), free sulphur and other impurities. Machineries/engines that make use of oil lubricant include all vehicles, motor bikes, generating plants and it is also used in various

industries/factories equipments. Spent oil is any petroleum-based or synthetic oil that has been used and as a result, is contaminated and has lost nearly all its physical or chemical properties but do not wear out and the contaminant present in it can be removed to the point that it can be reused as engine or machine oil (Garthe, 2005). Contaminants or impure lubricants are drained off from cars and disposed off as used or waste oil containing undesirable oxidation product, sediments, metallic wear particles, water, degraded additives and lead hazardous substances (James, 1967; Kamal and Khan, 2009; Durrani *et al.*, 2011; Emam and Shoaib, 2012; Abdulkareem *et al.*, 2014).

Disposal of used oil into the environment, used as weed killers, pouring into ditches or dumping illegally is dangerous to life. It contaminates water as research (Hamawand *et al.*, 2013) shows that a gallon of used oil can contaminate one million gallons of water. Used oil can also affect plant life if poured on the ground and it sinks down into the water table (EPA 530-F-94-008). It is therefore obvious that indiscriminate disposal of used

lubricating oil into the environment constitutes hazards to both fauna and flora. Hence, the need to recycle used lubricating oil. There have been several approaches in an attempt to regenerate used lubricants (Isah *et al.*, 2013; Abdulkareem *et al.*, 2014; Durrani *et al.*, 2011; Jhanani and Joseph, 2011; Ogbeide, 2010; Josiah and Ikiensikimama, 2010; Abdul-Jebbar *et al.*, 2010; Kamal and Khan, 2009; Rahman *et al.*, 2008; Gorman, 2005).

Industries and private users of lubricating oils demand necessitates the need to find ways of regenerating the oil after use due to the rapid depletion of fossil fuel reserve which is the source of oil feedstock, shortage of fresh oil, price increase and high demand (Zambiri, 1988). The increased use of lubricating oil globally for which Nigeria produces 364 166 000 litres of used oil annually disposed as a common practice into gutters, water drains, open plots and farms which led to streams, ground water, lakes and oceans pollution (Isah *et al.*, 2013; EPA 530-F-94-008). The frequent disposal of waste lubricating oils globally is alarming and requires serious attention. Presently, many countries in the world are presently giving serious attention to the problems of environmental degradation and hazards caused by the disposal of waste lubricating oils (Hamad *et al.*, 2005; Jilner, 1997). Oluwaseyi (2014) referred regeneration of used lubricating oil as the means of removal of contaminants or impurities by sulphonating agents such as sulphuric acid, oleum or sulphur trioxide and concluded that the process is made up of two cycles, the removal of about 80 % aromatics and other impurities in the first cycle and other 20 % impurities (such as sulphur and nitrogen) in the second operation cycle. However, Isah *et al.* (2013) carried out regeneration of used engine oil treated with sulphuric acid and bleached using industrial bleaching earth and activated carbon for different formulated grades. The results obtained by the group showed that the best grade in comparison with fresh oil in terms of viscosity, specific gravity and total acid number was obtained using industrial bleaching earth. Abdulkareem *et al.* (2014) compared the performance efficiency of used lubricating oil treatment methods and concluded from their findings that acid and acid/clay treatment methods were the most attractive with consequences of environmental pollution resulting from acid sludge. Emam and Shoaib (2012) also made comparison between acid/clay percolation and solvent/clay methods of recycling lubricating oils and concluded that the

used of acid treatment method was the most attractive in terms of quality while solvent method gave the highest yield. Owolabi *et al.* (2013) carried out acid treatment of waste lubricating oil using concentrated sulphuric acid and solvent treatment using methanol to study their performance in the extraction process to recover the lubricating oil. Udonne and Bakare (2013) investigated the effects of using HCl, HNO₃ and H₂SO₄ to regenerate waste Total lubricating oil used by a particular car for a period of one and half month. The treated oil characterization was only limited to pour point, cloud point, flash point, water content, sulphur content and viscosity index.

The used of sulphonating agents specifically sulphuric acid to treat used lubricant has been fully established by many researchers (Abdulkareem *et al.*, 2014; Isah *et al.*, 2013; Emam and Shoaib, 2012) and its performance has been very competitive but environmental consequences are part of its major limitations. In addition to environmental consequences, there is a dire need to quantitatively and qualitatively analyze the effect of other acids in recycling a mixture of other types of waste lubricating oil (such as Lubcon) used at different periods. The performance of various acid types to treat a mixture of single brand or various brands of waste lubricating oil used at different time span has not been properly investigated. This work suggests investigation of suitability and effects of nitric, sulphuric and hydrochloric acids in the treatment stage of used lubricating oil mixture from different cars used at different service period.

2.0 Methodology

2.1 Collection and Dehydration of Samples

The virgin oil sample was obtained from Lubcon oil, in Ilorin Kwara State Nigeria. The spent oil was obtained (Lubcon Super XV) from Logdisc Limited, a subsidiary (maintenance department) of Lubcon. It was filtered to remove impurities like sand, metal chips, dust and particles present in it. The filtered oil was transferred into a clean beaker and stirred thoroughly with magnetic stirrer to obtain homogeneity after which the oil was allowed to settle for about six hours. Water present was removed by gravity settling using separating funnel. After which, 250 ml of this pretreated spent oil was transferred into three different 500 ml beakers and treated with concentrated H₂SO₄, HCl and HNO₃ acids respectively.

2.2 Acid Treatment

A known volume (250ml) of spent oil was poured into a 500ml beaker and treated with 25ml of 98 % concentrated H_2SO_4 . The mixture of spent oil and acid was stirred vigorously with magnetic stirrer at constant temperature of 50 °C for 60 minutes. After which the mixture was transferred into a separating funnel fixed to a retort stand and left undisturbed for 48 hours to allow the acid sludge to settle. After 48 hours, two layers were formed with the upper layer greenish in colour but not clear while the lower layer "sludge" which was thick and black. This process was repeated for HCl and HNO_3 acids.

2.3 Removal of Sludge

At the end of the acid treatment step, the acidic oil was allowed to settle for at least 4 hours to form sediment at the bottom of the beaker known as sludge. Beyond this period, the acidic-oil was properly sedimented and was decanted into another 500 ml beaker while the residue i.e acidic sludge at the bottom of the beaker was removed and discarded.

2.4 Basic Treatment

After the removal of the sludge, 50 ml of 10 % NaOH was added to the oil to neutralize the acid, and allowed to stand for 30 minutes after which an alkaline phase formed at the bottom of the separating funnel was removed and the oil was poured into a sample bottle. The same process was repeated with HCl and HNO_3 . After 48 hours of separation for HCl, very large amount of sludge was formed at the bottom compared to H_2SO_4 and 25 ml of 10 % NaOH was used to neutralize the acid in the recycled oil sample after the sludge has been removed. While for HNO_3 , after agitating the oil and acid on the stirrer and poured into the separating funnel, large amount of sludge was formed but not as much as the sample treated with HCl. 25 ml of 10 % NaOH was also used to neutralize the acid in the recycled oil sample after the sludge has been removed. The treated samples were poured into sample bottles as well as the untreated oil sample and taken for characterization to determine ; kinematic viscosity at 40 and 100 °C, viscosity index, flash point, pour point, specific gravity at 15 °C, water content, sulphur content, total base no (TBN) and metals present (Pb, Zn and Cu).

2.5.0 Characterization

2.5.1 Determination of Kinematic Viscosities at 40 °C and 100 °C

This process was carried out using Viscometer and a Water Bath. In this process, each sample of spent and recycled oil was poured into a viscometer and placed into the Kinematic Water bath operating at 40 °C. The sample was drawn up the tube until it reached the upper meniscus mark using a pump, then the suction was released and the time taken for the oil to flow back to the lower meniscus mark was noted. The same process was used for water bath operating at 100 °C (ASTM D445).

2.5.2 Determination of Flash Point

20 ml of each sample of spent and recycled oil was introduced into a beaker and was placed on a Heating Mantle, then a thermometer was inserted into the oil sample. As the sample was heated, flame source was brought at interval to determine the temperature at which a flash is noticed on the surface of the sample (Udonne, 2010).

2.5.3 Determination of Pour Point

20 ml of each sample of spent and recycled oil was introduced into a beaker and chilled at specific rate. Some paraffin hydrocarbons which were in form of wax began to solidify and separated out in form of crystalline. After further chilling, the lube oil sample stopped to flow and the temperature was determined (Udonne, 2010).

2.5.4 Determination of Specific Gravity at 40 °C

The Specific Gravity is the ratio of the density of oil to the density of equal volume of water. It was determined using hydrometer. The density was observed at 40 °C and the value was documented (Udonne, 2010).

2.5.5 Determination of Water Content

60 ml of each sample spent or recycled was introduced into a round bottom flask and was distilled. The volume of water collected was recorded and water percentage in the sample was calculated using Equation 1 below:

$$\text{Water Content (\%)} = \frac{\text{Volume of Water Traped}}{\text{Volume of Sample}} \times 100 \quad (1)$$

2.5.6 Determination of Sulphur Content

Spectrophotometer was used to determine the Sulphur analysis. A burette containing 10 ml of each sample was clipped to the spectrophotometer. After achieving the position, the radiation bottom was allowed to emit its rays from X ray tube. The output excitation effect sobered as the beam was passed through the solution was measured as its merged from counts obtained from standard (Udonne and Bakare, 2013).

2.5.7 Determination of Total Base Number

Total base number determination involves titrating a sample of engine oil dissolved in a mixture of titration solvent (chlorobenzene and glacial acetic acid) with a titrant (perchloric acid in glacial acetic). The engine oil sample tested was weighed into a beaker and titration solvent was added in the ratio 2g to 100ml and shake to allow mixing of the sample engine oil with the solvent. The titrant was titrated with 0.1N per chloric acid in glacial acetic acid against the solvent and for visual determination, two drops of para Naphthal benzene indicator was added to the titrant before titration begins. The orange colour changes to green brown green at end point of titration. A blank titrant was then prepared by adding 10ml of titration solvent into a beaker without any sample and titrated. The total base number (TBN) was calculated using Equation 2.

$$TNB = \frac{(V_{S2} - V_S) \times 56.1 \times N}{W_S} \text{ (mgKOH/g)} \quad (2)$$

Where V_{S2} = volume of titrant used for titrating sample of engine oil V_S = volume of titrant used for titrating blank N = normality of the titrant = 0.0641 W_S = weight of sample taken for titration

2.5.8 Determination of Metals Present

Metals content was determined using Atomic Absorption Spectrometry. Each sample was dissolved in water, digesting in the acid dissolved it. A cathode lamp was used and the element characteristic wavelength was selected with a wavelength selector. Standard solutions of Zn, Pb and Cu were prepared and their absorbency was measured at selected wavelength against black solution absorbency. The absorbency of the sample in solution was given directly by the instrument (Udonne and Bakare, 2013).

3.0 Results and Discussion

The result presented in Table 1 and 2 shows the properties such as kinematic viscosity at 40 °C and 100 °C, viscosity index, flash point, pour point, specific gravity at 15 °C, water content, sulphur content, total base no (TBN) and metals present (Pb, Zn and Cu) of fresh, spent and treated oil in this work and previous works. Meanwhile Figure 1 shows the percentage yield of regenerated lubricating oil. The properties of fresh oil in Table 1 shows that there was no sulphur, water and metals in the fresh oil, an indication that the oil has not undergone oxidation, thermal degradation, contamination, corrosion or shearing. The effect of different acids types in this treatment method on the quality and yield performance of recycling used oil has been presented in this study. The result of characterization of the spent oil and recycled oil with different acids type namely H_2SO_4 , HCl, and HNO_3 is also presented in Table 1, while the yield of recycled oil with respect to each acid type is presented in Figure 1.

3.1 Viscosity Index

The results obtained as presented in Table 1 indicates that the viscosity index of spent oil is 91.2 which is lower than that of fresh oil (127) indicating that it cannot provide the much needed protection between the surfaces of its application. 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 (Udonne and Bakare, 2013). 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. The low value of viscosity of the spent oil was a result of oxidation, contamination and temperature change on the lubricant composition during usage with time which subsequently affects the efficiency and life expectancy of the lubricant. The results of viscosity index obtained when waste oil was treated with different acids is also presented in Table 1 which shows that, there was increase in viscosity with respect to each acid but the best improvement was obtained using sulphuric acid treatment with viscosity index of 125 which is close to that of fresh oil (127) and slightly higher than that (120) obtained in literature (Eman and Shoaib, 2012) as shown in Table 2. The result obtained shows that it was much higher and slightly lower than that obtained in literature (Udonne and Bakare, 2013; Hammaward *et al.*,

2013) as shown in Table 2. The variation in the viscosity index obtained with most literature values can be linked to the type of feed treated and method of treatment. Generally acid treatment method especially using sulphuric acid is the most favoured in terms of greater viscosity index recovery, an indication of better destruction and subsequent removal of all products of oxidation. Viscosity is a temperature, pressure and density function dependent. Inverse relationship exists between viscosity and temperature and when the temperature of the engine oil decreases the viscosity increases and reverse is the case. The measurements of viscosity testing can adequately predict the presence of contamination in used engine oil. The oxidized and polymerized products dissolved and suspended in the oil may cause an increase of the oil viscosity, while a decrease in the viscosity of engine oils shows fuel contamination (Hamawand *et al.*, 2013). Oxidation of base oils during use in an engine environment produces corrosive oxidized products, deposits, and varnishes which lead to an increase in the viscosity (Hamawand *et al.*, 2013). Kinematic viscosity for different samples at two temperatures measured for fresh, spent and recycled oil is presented in Table 1 and compared with other values obtained in literature as shown in Table 2. Results as presented indicates that kinematic viscosity of spent oil are 115.8 and 12 cSt respectively at 100 and 40 °C which shows that treatment of spent oil with acid equally influenced the kinematic viscosity of spent oil, however, sample treated with H₂SO₄ gave the best result with kinematic viscosity at the two temperature levels of 40 and 100 °C (159.60 and 18 cSt) being closer to that of the fresh oil (170 and 19 cSt). In comparison to other previous literature values it shows that the kinematic viscosity obtained for the recycle oil is lower than 169.5 obtained at 40 °C by Hammaward *et al.* (2013) and greater than some other literature values at the two temperatures (Owolabi *et al.*, 2013; Hammaward *et al.*, 2013; Isah *et al.*, 2013; Shakirullah *et al.*, 2006). The variation resulted from the use of different treatment approach, operating conditions and importantly the type of feed that differs in the extent of degradation.

3.2 Pour Point

Pour point of fresh, spent and treated oil were investigated and the result obtained is presented in Tables 1. It is an important property that determines the minimum temperature at which the lubricant will cease to flow. Most engine base oils

contain waxes and paraffin's that solidify at cold temperatures. Engine oils with high wax and paraffin content will have a higher pour point. Pour point is highly affected by oil's viscosity, and engine oils with high viscosity are characterized by having high pour points. The pour point of engine oil is an important variable, especially when starting the engine in cold weather. It is necessary for the oil to possess ability to flow into the oil pump such that it can be pumped to the various part of the engine especially at low temperatures. The result obtained in Tables 1 indicates that the pour point of the spent oil was higher than that of fresh oil an indication that some materials added as additive for the pour enhancement have degraded and treatment with different types of acid resulted in the decrease of pour point to -15 with H₂SO₄ and -18 using HCl and HNO₃. The extent to which each acid reduces the pour point of the spent oil in Table 1 indicates that H₂SO₄ has the strongest effect in lowering pour point than all other acids considered and the value obtained using H₂SO₄ was -15 closed to that of fresh oil (-12) which is the reference point. Table 2 shows further comparison with some literature values and the result shows that the pour point of -15 obtained in this work is higher than that of the most values obtained in literature (Udonne and Bakare, 2013; Hammaward *et al.*, 2013; Shakirullah *et al.*, 2006). This indicates that the pour point can be further improved when its paraffin and wax content can be further reduced. Also the variation may be as a result of operating variables such as the acid to oil ratio or the acid concentration.

3.3 Flash Point

Flash point of engine oil is the minimum temperature to which the oil must be heated under specified conditions such that it can give off sufficient vapour required to form mixture with air that can be ignited spontaneously by a specified flame. Lubricants find most of its application between moving parts where there is heat generation and therefore, it should be able to withstand higher temperature conditions. The flash point of engine oil determines the extent of the oil's contamination. A very low flash point of engine oil is a reliable measure of oil contamination with volatile products such as gasoline. In the presence of ≥ 3.5% fuel in used engine oils, the flash point may reduce to below 55 °C. The flash point provides identity of a particular petroleum product. The flash point

increases with increasing molecular mass of the oil and decreases as a result of oxidation resulting into the formation of volatile components. The reference in this case is the flash point of the fresh oil shown in Table 1 which in comparison to that of spent oil shows that there was decrease as a result of contamination with volatile components. However, treatment with different acids shows that H_2SO_4 is the most effective followed by HNO_3 . The flash point obtained in this work is higher than that obtained in most literatures (Udonne and Bakare, 2013; Hammaward *et al.*, 2013; Shakirullah *et al.*, 2006) and lower than that obtained by Shakirullah *et al.* (2006) in Table 2. This differences could be linked to the used of improved aluminium sulphate-sodium silicate acid base treatment method resulting into the enhance flash point. However, the variations in the flash points in literature can also be linked to the different feedstocks and treatment methods. The improvement in spent oil flash point from 220 to 232 °C can be made better when the process operating variables are optimized.

3.4 Specific Gravity

Specific gravity (specific density) parameter evaluated for the fresh, spent and treated oil is the ratio of the density of material to the density of equal volume of water at a given temperature of fresh oil, spent, and treated oil. The results obtained for fresh and other samples (spent and treated) are presented in Tables 1. Specific gravity which is temperature dependent property was measured at 40 and 15 °C for all samples and the spent oil specific gravity at these temperatures were 0.91 and 0.93. The difference in comparison to that of fresh oil is as a result of sludge present in the spent oil. Meanwhile, treatment with different acids type gives results that are in close range, though treatment with H_2SO_4 slightly gives smaller value of specific gravity of 0.90, hence a better spent oil treatment option. The specific gravity of the feed oil is higher than the re-refined oils in Table 1 and lower than the virgin base oil (0.895). The results for the feed and re-refined oils with H_2SO_4 , HCl and HNO_3 are 0.925, 0.90, 0.91 and 0.9070, respectively. The specific gravity of contaminated oil could be lower or higher than that of its fresh oil depending on the type of contamination. If the used oil was 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 or the re-

refined one (Eman and Shoaib, 2012). The result obtained shows that both water dilution and other contaminants were responsible for the change in the fresh oil specific gravity during operation and H_2SO_4 has shown close proximity to the fresh oil, followed by HCl. In comparison to literature results as shown in Table 2, the result of specific gravity of the most recycled oils had specific gravity lower (Hammaward *et al.* 2013; Isah *et al.* 2013; Eman and Shoaib, 2012) than that of the spent oil used in this work with exception of 0.932 obtained by Isah *et al.* (2013).

3.5 Water Content

Lubricants hygroscopic nature allows it to absorb moisture from the air and formed emulsion or suspended moisture which is a chemical contaminant. The engine block is made of aluminum, iron and lead, hence during combustion of fuel in the engine chamber, the wear of these metals in parts per million (ppm) are found in the used oil (Eman and Shoaib, 2012). In addition, the wear of these metals is due to the corrosion caused by the presence of water and aided by fuel dilution due to bad piston rings (Eman and Shoaib, 2012). The fresh oil analysis result in Table 1 shows the absence of moisture in the fresh sample and the spent oil of the same oil brand had water content of 10.99 wt %. This moisture content increase is as a result the hygroscopic nature of the oil and possibly combustion, oxidation, and neutralization of the oil within its service period is what resulted into the increase in the oil moisture content. The different acid treatment reduces the moisture content to virtually the same extent (< 10 %) and closely to that of fresh sample as 1.01, 1.04 and 1.02 wt % water using H_2SO_4 , HCl, and HNO_3 respectively. The greater water removal and observed higher sludge settlement in using HCl indicates its greater potentials in freeing impurities but the results of properties of recovered oil may favour sulphuric acid because of its potentials to mop out any impurity. The result obtained in comparison to previous works in literature (Udonne and Bakare (2013) shows that moisture content in the recycle oil in this work is the highest with some methods reported to have completely remove all moisture (Owolabi *et al.* (2013). This could be attributed to the moisture content in the spent oil and the effectiveness of the method used and variation in operating variables.

Table 1. Properties of Spent and Treated Oil

S/N	Test Parameter	Fresh	Spent oil	Values		
				H ₂ SO ₄	HCl	HNO ₃
1	Kinematic viscosity@ 40 °C (cSt)	170.00	115.80	159.60	132.20	140.80
2	Kinematic viscosity@100 °C (cSt)	19.00	12.00	18.00	15.00	16.00
3	Viscosity index	127.00	91.20	125.00	116.00	119.00
4	Flash point (°C)	245.00	220.00	232.00	225.00	227.00
5	Pour point (°C)	-12.00	-30.00	-15.00	-18.00	-18.00
6	Specific gravity @ 40 °C	0.895	0.91	0.885	0.895	0.8920
7	Specific gravity @ 15 °C (after correction factor)	8.90	0.925	0.90	0.91	0.9070
8	Water content (wt %)	-	10.987	1.011	1.036	1.023
9	Sulphur content (wt %)	-	0.80	0.046	0.20	0.350
10	Metals present	-	-	-	-	-
	Lead (Pb) (ppm)	-	0.401	0.123	0.147	0.389
	Zinc (Zn) (ppm)	-	16.33	8.020	8.400	12.93
	Copper (Cu) (ppm)	-	0.930	0.41	0.71	0.586
11	Total base number (TBN)Mg KOH/g	-	2.900	7.80	5.00	5.60

3.6 Sulphur Content

The sulphur content of the fresh, spent and recycled oil samples were tested and the result obtained in Table 1 shows that the total absence of sulphur in fresh sample is an indication of the fresh oil satisfactory requirement in reducing particulate emission during usage. The absence of sulphur in the fresh oil analysed sample could be attributed to the presence of inherent anti-oxidant. The spent oil sulphur content was 0.80 wt %, a level considered unacceptable as it can cause a serious engine wear and corrosion. The treated oil sulphur content was reduced to different wt % depending on the acid type and the least sulphur content was obtained using H₂SO₄ followed by HCl with 0.05 and 0.20 wt % sulphur content respectively. This reduction did not fall within the acceptable limit of sulphur of 0.01 wt% in lubricating oils (Abdulkareem *et al.*, 2014). The reduction in sulphur content in this work was much better in comparison to most reported values in literature (Udonne and Bakare, 2013; Eman and Shoaib, 2012) as shown in table 2. This can be attributed to the effectiveness of the method and appropriate choice of operating parameters in addition to the nature of the sample.

3.7 Metals Present

The metal contents in lubricating oil are usually heavy metals like lead, iron, aluminum and copper; and indiscriminate disposal of these metals into the environment could lead to the pollution of ground water. The potentials of the

method to remove these metals from spent oil was also investigated and the result in Table 1 shows that contrary to that of fresh oil, the presence of metals in spent oil and the extent to which it has been removed using different acids treatment indicates the potentials of each acid treatment. The metal content in the spent oil are actually picked by the oil during usage because some amount of these metals are available in most metallic surfaces for which the lubricant was meant function. Metals analysed for fresh, spent and recycled oil were lead, zinc and copper and were found to be present in the spent oil as 0.40, 16.33 and 0.93 ppm respectively. After spent oil treatment, the metals were reduced more with H₂SO₄ followed by HCl for lead and zinc only. However, nitric acid removes more copper than HCl and this could be attributed to its ability to greatly oxidize the metals picked by the oil. The recycle oil in this work has shown to have the least metal content when compared with the available literature (Eman and Shoaib, 2012) values in Table 2. Though, there were few reported values of metal content of most recycled lubricating oils. This indicates probably that the variables such as acid concentration and other operating variables favoured more oxidation of these metals to form compounds that can be removed easily as contaminants.

3.8 Total Base Number (TBN)

Table 1 also shows the result of total base number (TBN) or neutralization number for fresh, spent

and treated oil samples which is the determined quantity of potassium hydroxide (KOH) needed to neutralize the acid present in a lubricant. The result presented in Table 1 indicates that Lubcon fresh oil has total base number of 8.9 mg KOH/g, which is high. From the result presented, spent oil has the lowest TBN of 2.9 mg KOH/g, which is dangerous to the engine because it has little or no power to neutralize the acid formed during combustion, it means oil change is required. Meanwhile result presented further indicates that the sample treated with H₂SO₄ gave the best TBN

value of 7.80 mg KOH/g which falls within the standard range of fuel engine lubricant TBN of 7-10 mg KOH/g. Therefore, H₂SO₄ is the best acid for this treatment followed by HCl and HNO₃ with 5.0 and 5.60 mg KOH/g respectively. The later values obtained for HCl and HNO₃ are not within standard acceptable range of TBN. The result of TBN obtained in this work compared favourably to the standard in the case of H₂SO₄ and with most reported literature values of recycled lubricating oil as shown in Table 2.

Table 2. Properties Comparison of Treated Oil

S/ N	Test Parameter	Values											
		Spent Oil	Treated Oil	A	B	C	D	E	F	G	H	I	J
	Kinematic viscosity@ 40 °C (cSt)	115.80	159.60	-	108.1	169.5	61.5	53.16			92	90	91
	Kinematic viscosity@100 °C (cSt)	12.00	18.00	-	-	16	7.8	-			7.7	7.2	7.5
	Density (g/cm ³)	-	-		1.091	-	-	-	-	-	0.885	0.88	0.88
	Viscosity index		125.00	88.6	-	97.07	89	-	120	128	-	-	4
	Flash point (°C)	91.20	220.00	173	-	222	220	-			240	180	185
	Pour point (°C)	-30.00	-15.00	-13	11	-10	-9.8	-			-11	-9	-9
	Specific gravity @ 40 °C	0.91	0.885	-	-	0.882	0.869	0.932	0.699	0.820			
	Specific gravity @ 15 °C (after correction factor)	0.925	0.90	0.876	-	-	-	-	1	2			
	Water content (wt %)	10.987	1.011	0.48	Nil	-	-	-	-	-	-	-	-
	Sulphur content (wt %)	0.80	0.046	0.041	-	-	-	-	0.81	0.42	-	-	-
	Metals present												
	Lead (Pb) (ppm)	0.401	0.123	-	-	-	-	-	-	-	-	-	-
	Zinc (Zn) (ppm)	16.33	8.020	-	-	-	-	-	-	-	-	-	-
	Copper (Cu) (ppm)	0.930	0.41	-	-	-	-	-	117.8	52	-	-	-
	Total base number (TBN)Mg KOH/g	2.900	7.80	-	-	-	-	0.54	0.055	0.052	-	-	-

A-Udonne and Bakare (2013); B- Owolabi *et al.* (2013); C&D-Hammaward *et al.* (2013); E- Isah *et al.* (2013); F&G- Eman and Shoaib (2012); H, I&J- Shakirullah *et al.* (2006).

3.9 Yield

The yield of treated oil samples in this work was also determined and the result presented in Figure 1. The result further shows that sulphuric acid is the best not only in terms of parameter characterization but also in terms of yield followed by nitric acid and lastly HCl. The yield obtained for sulphuric acid was higher than 63.7 %

obtained by Eman and Shoaib (2012) that used acid treatment and clay. The variation could be linked to the differences in the feedstock and treatment conditions applied. Therefore, this suggests optimization of the process to establish conditions that can suit all formulations of waste oil.

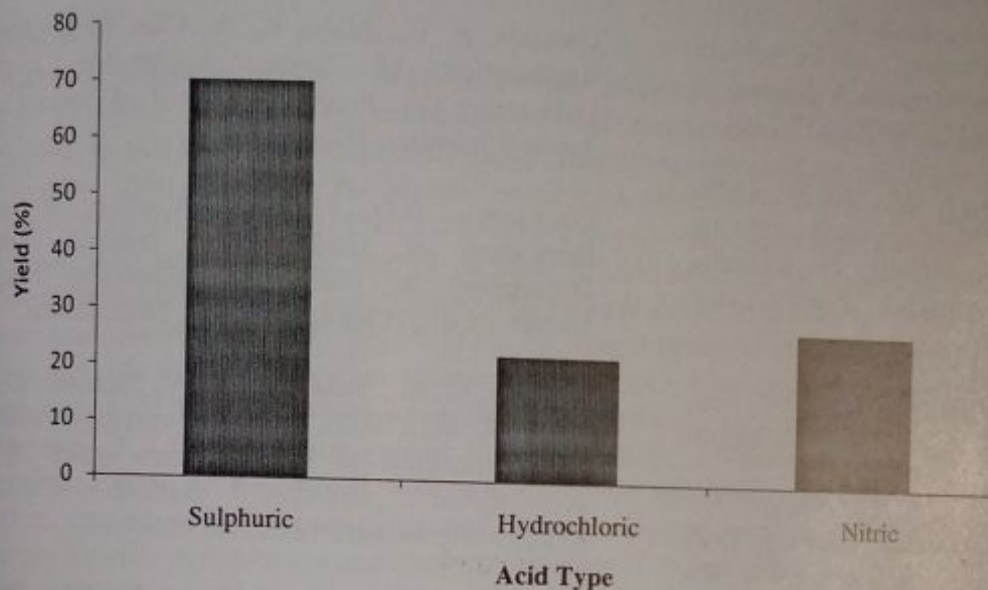


Figure 1: Effect of various acids on the yield of recycled lube oil

Conclusively, the result obtained entirely shows that sulphuric acid is the best in acid treatment of spent lubricating oil, however not all parameters investigated in this work for the treated oil are adequately within the acceptable range but further optimization and probably co-acid treatment may likely enhance the treatment efficiency. Also, nitric acid is the second best in terms of yield and property parameter assessment expect for copper metal removal which HCl was the best. The performance of H_2SO_4 can be linked to its greater potential to mop out impurities contained in the spent oil. This suggests that the mopping potential of sulphuric acid has greater influence than the freeing ability of the sulphuric acid in spent lubricating oil recycling.

4.0 CONCLUSION

Used lubricant oil was treated using acid treatment method. Effects of acid types on the qualities of treated oil such as kinematic viscosity at $40^\circ C$ and $100^\circ C$, viscosity index, flash point, pour point, specific gravity at $15^\circ C$, water content, sulphur content, total base no (TBN) and metals

present (Pb, Zn and Cu) were investigated. Based on the results obtained, acid treatment method effectively improved the qualities of treated oil. Treatment efficiency of used oil with H_2SO_4 gave the best results compared to that of HNO_3 and HCl; this is evident by the 70 % yield of treated oil. The result obtained suggests optimization of operating parameters and study of synergistic effect of HCl and H_2SO_4 for the best recovery of the spent oil. The quality of treated oil is adequately suitable for direct used as lubricant as most parameters fall within acceptable limits but additional treatment may be required for quality improvement.

ACKNOWLEDGEMENT

Support received from Tertiary Education Trust Fund (TETFUND/FUTMINNA/2014/28) is highly appreciated.

REFERENCES

Abdul-Jebbar, N.M., Alzubaidy, E.A.H, Mehrvar, M., (2010). Waste lubricating oil treatment by adsorption process using different adsorbents. *World*

Academy of Science, Engineering and Technology, 62, 9-12.

Abdulkareem A.S, Afolabi A.S, Ahanonu S.O, Mokrani T. (2014). Effect of treatment methods on used lubricating oil for recycling purposes. *Energy Sources, Part A* 36:966-973.

Base Oil Handbook retrieved online from www.engnetglobal.com on the 26th May, 2015.

Durrani, H.A., Panhwar, M.I., Kazi, R.A., (2011). Re-refining of waste lubricating oil by solvent extraction. *Mehran University research journal of Eng. & tech.*, [ISSN 0254-7821], 30 (2), 237-246.

Emam, A. E. and Shoiab, A. M. (2012). *ARNP Journal of Science and Technology* on Re-refining of Used Lube Oil. Department of Refining Engineering and Petrochemicals, Suez Canal University, Egypt. p1-8.

Garthe, J.W. (2005); Recycling used oil, Agricultural and Biological Engineering, College of Agricultural Sciences, Penn State. Pg 1-2

Gorman, W.A., (2005). Recovering base oils from lubricants. *Petroleum Technology Quarterly*, 4, 85-88.

Hamad, A., Alzubaidy, E., and Fayed, E.M. (2005). Used lubricating oil recycling using hydrocarbon solvent. *J. Environ. Manage.* 74:153-159.

Hamawand, I., Yusaf, T., Rafat, S. (2013). Recycling of Waste Engine Oils Using a New Washing Agent. *Energies*, (6), 1023-1049.

Isah, A.G, Abdulkadir, M., Onifade, K.R., Musa, U., Garba, M.U., Bawa, A.A., Sani, Y. (2013). Regeneration of used engine oil. In *World Congress on Engineering*.

James, R.G. (1967). Reclamation of used lubricating oil 180-280, USA.

Jhanani, S., and Joseph, K., (2011). Used oil generation and management in the automotive industries. *International Journal of Environmental Sciences*, 2(2), 638-648.

Jilner, D. A. (1997). Method of analysis and testing of petroleum related products. *J. Mater. Process. Technol.*, 7: 15-21.

Josiah, P. N., Ikiensikimama, S.S., (2010). Effect of desludging and absorption ratios on recovery of low

pour fuel oil (LPFO) from spent engine oil. *Chemical Engineering Research Bulletin*, 14, 24-28.

Kamal a., Khan, F., (2009). Effect of extraction and adsorption on re-refining of used lubricating oil, *Oil and Gas Science and Technology*, 64(2), 191-197

Ogbeide, S.O. (2010). An investigation to the recycling of spent engine oil. *Journal of Engineering Science and Technology Review* 3(1) (2010) 32-35

Owolabi, R. U., Alabi, K. A., Oke, O. B (2013). Reclamation of Spent Automobile Engine Lubricating Oil. *Fountain Journal of Natural and Applied Sciences*, 2(1), 11 – 16.

Oluwaseyi, E.O (2014). Recycling of used lubricating oil. Undergraduate research project. Department of chemical Engineering, Federal University of Technology Minna, Unpublished.

Rahman, M. M., Siddiquee, T. A., Samdani, S. and Kabir, K. B., (2008). Effect of operating variables on regeneration of base-oil from waste oil by conventional acid-clay method. *Chemical Engineering Research Bulletin* 12, 24-27.

Shakirullah, M., Ahmad, I., Saeed, M., Khan, M.A., Rehman, H., Ishaq, M., Shah, A.A (2006). Environmentally Friendly Recovery and Characterization of Oil from Used Engine Lubricants. *Journal of the Chinese Chemical Society* (53), 335-342.

Toolingu University Press, (2013). Mechanical system trainings and bearing applications210. Retrieved from <http://www.toolingu.com/definition-560210-86370-journal-bearing.html> on 15th March, 2013

Udonne J.D (2011). A comparative study of recycling of used lubrication oils using distillation, acid and activated charcoal with clay methods. *Journal of Petroleum and Gas Engineering* Vol. 2(2), p12-19

Udonne J.D, Bakare O.A (2013). Recycling of Used Lubricating Oil Using Three Samples of Acids and Clay as a Method of Treatment. *International Archive of Applied Sciences and Technology IAAST*, 4 (2) 8- 14

Zambiri S. (1988). Regeneration of used lubricating oil. Chemical Engineering Department ABU Zaria.