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Analysis of Acid Options in the Purification of Spent Motor Engine Oil Using Acidified Clay

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Abstract: This research is centered on the purification of Spent Shell Helix (HX3 20W-50) lubricating oil using acidified clay treatment with three acids options: mineral sulfuric acid (H_2SO_4), organic acetic acid (CH_3COOH), and organic citric acid ($C_6H_8O_7$), (a novel washing agent). The clay mineral, spent, purified, and fresh lubrication oil samples were characterized to determine their physicochemical properties. The clay mineral was characterized using Energy-Dispersive X-Ray Fluorescence (EDXRF). The result indicated that the clay was bentonite clay and it was successfully acidified. The result also indicated that properties of the spent lubricating oil such as Kinematic viscosities at 40 ºC and 100 ºC increased from 118.9 cSt and 13.9 cSt for the spent oil to 169.7 cSt and 15.2 cSt, 178.2 cSt and 16.3 cSt, 183.4 cSt and 16.8 cSt for the oil samples purified with $C_6H_8O_7$, CH_3COOH and H_2SO_4 respectively Also, the flash point increased from 106 ºC for the spent oil to 133 ºC, 180 ºC, 189 ºC for the oil samples purified with $C_6H_8O_7$, CH_3COOH and H_2SO_4 respectively. The FTIR analysis of the spent oil revealed the presence of primary oxidized products, absent in spent engine oil purified with mineral sulphuric acid (H_2SO_4), organic acetic acid (CH_3COOH), and fresh oil. Acidified clay effectively purified the spent engine oil, bringing its physicochemical properties closer to those of fresh oil. However, the effectiveness of the treatment varied depending on the specific acid used.

Keywords: Spent oil, purification, shell helix, acidified clay, viscosity

I. Introduction

Lubricating oils, its derivatives, and lubricants exhibit significant chemical complexity [1]. Lubricating oil is essential for various internal combustion engines, it goes beyond simply smoothing friction between moving parts. It also cleanses components, guards against corrosion, enhances sealing, and cools the engine by transporting heat away. Due to its widespread use in a diverse range of machinery, from cars and trucks to airplanes and industrial equipment, lubricating oil stands as one of the most crucial products derived from crude oil [2]. Lubricating oils are viscous liquids that are used to minimize friction and wear between parts rubbing together in a variety of combustion engines, automobiles, and machines [3].

Used lubricating oil includes engine oil, transmission oil, hydraulic oil, and cutting oil that have been used for a period of time. Over time, the original oil components degrade and mix with contaminants like metals, ash, carbonaceous residue, water, varnish, gums, and asphaltic substances from the engine's wear. This contamination makes the oil less effective, so it must be regularly changed and removed from vehicles, machinery, and equipment to prevent damage [4].In 2008, The United States Environmental Protection Agencies (EPA) set a standard for used oil, defining it through a three-part approval process.

Firstly, the oil must be derived from either Petroleum-based sources or synthetically derived materials, excluding animal and vegetable oils. Secondly, the oil must have been used for lubrication, hydraulics, heat energy transfer, or similar purposes. Unused oils, such as those from tank cleanouts or spills, are excluded. Additionally, products like antifreeze, kerosene, and those used solely for cleaning or solvent purposes are not considered used oil. Finally, as a result of being used, oil inevitably becomes contaminated with various physical and chemical impurities. These contaminants, generated from handling, storing, and processing the oil, can include physical materials like metal shavings, sawdust, or dirt, as well as chemical substances such as solvents, halogens, or saltwater [5].

Improper disposal of used lubricating oil can pose significant threats jeopardizing environmental sustainability and economic stability. Discharging it onto land, water, or even burning it as fuel leads to serious pollution. These practices pollute the environment with harmful metals and other contaminants [6]. Generally, Engine oil wears out for two key reasons:

i. Accumulation of impurities in the oil

ii. Oil chemical alterations.

These effects compromise the oil's critical properties, hindering its effectiveness in use [7].

To combat this issue, we must prioritize sustainable alternatives like recycling, which is currently considered the most effective approach to addressing the detrimental effects of improper used oil disposal [8]. Beyond environmental benefits, re-refining used lubricating oil offers a more sustainable approach to producing base oil compared to using crude oil. This process requires significantly less raw material and utilizes up to 90% less energy, minimizing environmental impact [9].

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Certain large-scale industrial users of lubricants around the world have adopted on-site oil regeneration. This process utilizes physicochemical methods to clean used oil, effectively removing contaminants. The regenerated oil is then either blended with virgin oil for use in internal combustion engines (ICEs) or directly repurposed for industrial applications like gear lubrication, cutting oils, and lubricants for metal rolling [10].

Despite a depth of comprehensive research, several studies have explored the potential of acidified clay for purifying used engine oil. The Table 1 summarizes some of these investigations.

Table 1: Application of Acidified Clay technique in the purification of Used Engine Oil

S/N	Author	Sources Of UEO	Acidification Method	Purification Parameters	Application
1.	Ugwele and Chime, 2019	Used Mobil oil	Acid/clay, acid/charcoal and acid/clay/charcoal method using sulfuric acid, acetic acid and hydrochloric acid	Acid Concentration = 30ml Bleaching Temp = 110^0C for 10 minutes	To be used in combustion engines of cars, industries and factories
2.	Afolabi *et al.*, 2016	Used Total Quartz 5000 20W-50	Acid/clay method using H_2S0_4	Acid Concentration = Varied Conc Bleaching Temp = Preheated for 10 minutes	To be used in combustion engines of cars, industries and factories
3.	Shabanzade *et al.*, 2018	Waste Engine Oil	Acid/clay method using H_2SO_4, HCl and CH_3COOH	Acid Concentration = Varied Conc Bleaching Temp = 100^0C for 1hour	To be used in combustion engines of cars, industries and factories
4.	Mintesinot, 2021	Used Engine Oil	Acid/clay method using Hydrochloric acid and Acetic acid	Acid Concentration = 10 ml Bleaching Temp = 250^0C	To be used in combustion engines of cars, industries and factories

[3,8,15,17]

II. Materials and Methods

Materials

This research exclusively relied on locally sourced materials, they include the used (Shell Helix HX3 20W-50) Crankcase oil from Honda civic 2008 model, Clay from the back of talba market in Minna Niger State, at a depth of about 30 cm, Conventional laboratory reagents from Panlac Chemicals along Keteren Gwari Road, Minna Niger State. Apparatus and Equipment from National Board for Technology Incubation David Mark Road, Minna Niger State.

Methods

A 1000 cm^3 round-bottom flask was used to mix thoroughly 200 g of clay particles with a size of 74 μm with 400 cm^3 of 1M H_2SO_4. The mixture was subjected to heating at 200 °C using a magnetically stirred hot plate for a duration of 3 hours [11]. Afterwards, the clay was subjected to washing with distilled water to eliminate remaining precipitates, stones, and impurities until a neutral pH was reached as indicated by a pH indicator. The clay was then filtered and baked for about 6 hours at 200 ºC to remove water and stored in dry place to be used later in the purification process. The obtained dry and clean clay is called activated clay because it is active chemically and electrostatically as reported by [12].

To eliminate contaminants like micro impurities, metal chips and sand from used lubricating oil, a filtration process was carried out. The filtration process involved the use of a buchner funnel and filter paper. The filter paper was fitted into the buchner funnel and the used oil was then filtered by passing it through the set-up. Following a 24-hour settling period, the oil underwent preheating at 45 °C for 30 minutes. This preheating step promotes the degradation of specific additives, reducing the workload of the acid [13].

Three separate beakers were then used to accurately measure three samples of pre-treated engine oil each measuring 300 cm^3. Then Separate aliquots of each acid (30 cm^3 each) were individually transferred into 50 cm^3 beakers. To prepare for the reaction, the regulated hot plate was activated, and the beaker containing 300 cm^3 of used engine oil was placed on the hot plate. The oil was heated for approximately 5 minutes until it reached a temperature of 45 °C. Following this heating step, each acid was carefully added to its respective beaker containing the preheated oil. The first beaker 1M H_2SO_4, Second beaker 1M CH_3COOH and third beaker 1M $C_6H_8O_7$. The whole mixture was then stirred consistently for 10 minutes. Following the acid treatment, the acid-treated oils were left undisturbed for 24 hours. This allowed sedimentation of insoluble materials, forming a residue (acidic sludge) at the bottom of the beakers. Subsequently, the acidic mixtures were carefully decanted using mesh filters into separate 500 mL beakers. This process effectively separated the treated oil from the settled sludge, which was then discarded properly [3].

The Acidified clay (30 g), grounded to a particle size of 74 μm, was packed into a Buchner funnel equipped with filter paper. This setup was then connected to a vacuum pump. Subsequently, the sulfuric acid-treated oil was passed through the clay bed using the

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vacuum. This filtration process effectively removes impurities from the oil and was repeated for each of the acid-treated oil samples [14].

To neutralize the acid in the three oil samples, 100 cm^3 of 10 % sodium hydroxide (NaOH) solution was added to each oil sample. The mixture was then left undisturbed for 24 hours to allow for phase separation between the oil and the aqueous layer. Following this settling period, the oil layer was carefully decanted into a clean beaker, and the remaining residue at the bottom was discarded appropriately. Finally, the pH of the decanted oil was measured and recorded to verify the effectiveness of the neutralization process [3]. Finally, the resulting purified lubricating oil was obtained and ready for analysis as reported by [11].

III. Results and Discussion

The characterization of the clay sample was carried out using EDXRF analysis so as to determine the soil composition and type of clay sample used for the experiment, Afterwhich the spent, purified and fresh engine oil samples (Shell Helix HX3 20W-50) were characterized.

Characterization of Clay Sample using Energy-Dispersive X-Ray Fluorescence Spectroscopy (EDXRF) Analysis

Table 2: Result of Characterization of Clay Sample using Energy-Dispersive X-Ray Fluorescence spectroscopy (EDXRF) Analysis

S/N	Element	Weight % (Concentration)
1.	Fe_2O_3	7.399
2.	MgO	2.350
3.	Al_2O_3	24.460
4.	SiO_2	49.819
5.	K_2O	1.215
6.	TiO_2	0.927
7.	CaO	0.742
8.	BaO	O.512
9.	SrO	0.730
10.	L.O.I	11.846

L.O.I = Lost on Ignition

The result obtained in the analysis of the clay sample in Table 1 indicates that the sample is primarily composed of Alumina (Al_2O_3) and silica (SiO_2) as the two main constituents. It also contains Iron (III)oxide (Fe_2O_3) in large proportions while other constituents such as Magnesium oxide (MgO), Barium oxide (BaO), Titanium oxide (TiO_2), Calcium Oxide (CaO), Potassium oxide (K_2O), and Strontium oxide (SrO) are present in noticeable contents.

The result in Table 1 above indicates that the percentage ratio of Alumina (Al_2O_3) to Silica (SiO_2) is approximately 0.5 which can be compared to the basic composition of bentonite clay which is > 0.3 [15].

Characterization of Spent, Purified and Fresh Engine Oil Samples Using Quality Test Procedures Kinematic Viscosity:-

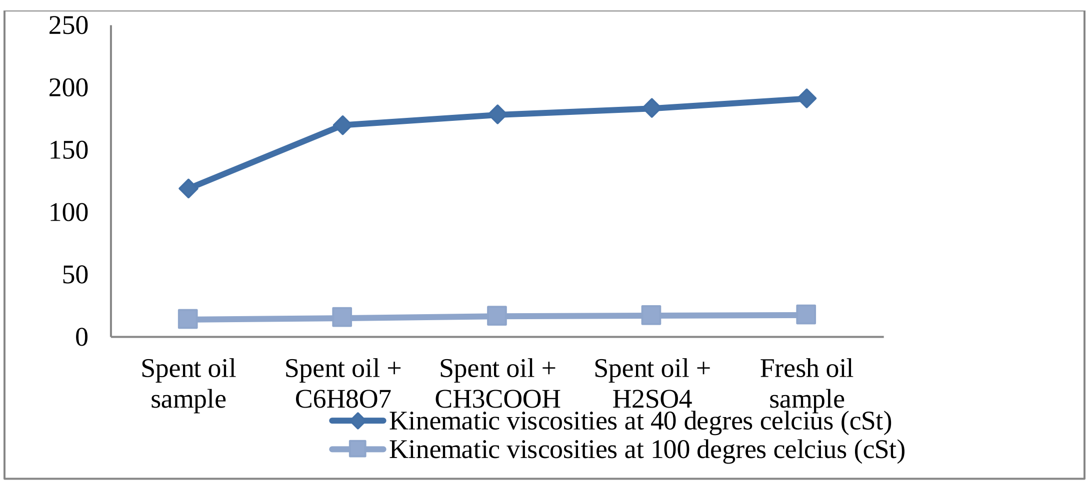

Figure 1: Effect of Kinematic Viscosities of Engine oil Samples at 40 ^{O}C and 100 ^{O}C

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Several factors contribute to an increase in engine oil's viscosity, making it flow less easily, factors such as dirt, glycol and soot while fuel dilution and the breakdown of special molecules in some oils can make it thinner [16]. The thicker the oil (higher viscosity), the stronger the protective film it forms on engine parts [3]. Figure 1 above indicates that the viscosity is greatly influenced by the type of acids and clay spent for the purification of the used engine oil. The kinematic viscosity of the spent oil was obtained as 118.9 cSt and 13.9 cSt which is in agreement with the work reported by [17]. While the kinematic viscosities of the purified lubricating oil samples were much higher than that of the spent engine oil and comparable to that of the fresh oil. The kinematic viscosity obtained for the fresh oil sample is in agreement with 192 cSt and 18.3 cSt reported by [18]. While used engine oil is typically thinner due to impurities, the purification process using acids can convert and remove these contaminants, resulting in thicker, higher viscosity oil after filtration. [19]. The result from figure 1 above indicates that the spent oil samples purified with Organic acetic acid (CH_3COOH) and Mineral sulphuric acid (H_2SO_4) solutions gives kinematic viscosities that are very close and comparable to that of the fresh oil sample which means that the purification of spent engine oil using this two acids with acidified clay is very effective in removing contaminants however purified engine oil using acidified clay method with sulphuric acid as a purifying agent has advantage over other techniques [3].

Specific Gravity:-

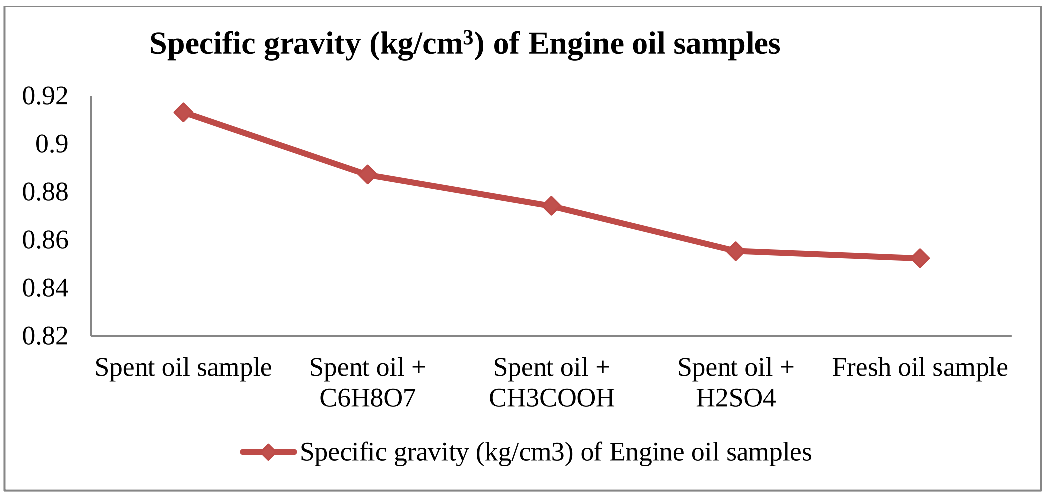

Figure 2: Effect of Specific Gravity on Engine oil Samples

Specific gravity compares how tightly packed a substance is (its density) to a standard, often water and the more solid particles there are in the used oil, the higher its specific gravity gets [20]. Figure 2 shows the effects of the specific gravity obtained for the spent, purified and fresh engine oil samples using the acidified clay treatment procedure. The result for the spent engine oil is in-line with 0.902 reported by [15], 0.900 obtained for used mobile oil reported by [3] and 0.91 reported by [21]. While the results of the regenerated oil samples are less than that of the spent engine oil and in agreement with 0.8759 reported by [22] and 0.849 to 0.898 reported [23]. [24] reported that used engine oils thickens (higher specific gravity) with use due to the buildup of impurities like worn metals and degraded oil components during operation. From the result obtained, The acidified clay purification method using mineral sulphuric acid improved the specific gravity of the engine oil and gives values that are equivalent to that of the fresh engine oil.

Sulphur Content:

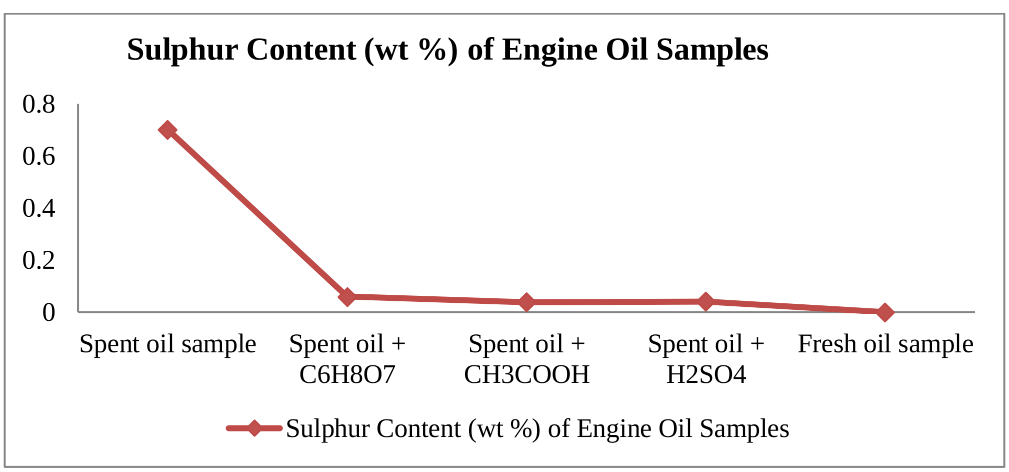

Figure 3: Effect of Sulphur Content on Engine oil Samples

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High sulfur content in used oil indicates potential engine wear. As engine parts rub against each other, tiny metal particles contaminate the oil. Sulfur in the oil reacts with these metal fragments, forming compounds with low melting points. Furthermore, in engines using refined oils, leftover sulfur from fuel can oxidize (break down) into acidic compounds that contribute to engine corrosion [14]. Figure 3 indicates that the spent engine oil has a high sulphur content as compared to the purified engine oil samples which shows low values of sulphur content, the fresh engine oil shows a total absence of sulphur content indicating that sulphur was introduced into the spent engine oil due to engine parts rubbing against each other [24]. The result obtained is in agreement with the results reported by [3,11 ,14]. The result shows that the spent engine oil sample purified with organic acetic acid gives the lowest sulphur content of 0.038 (wt%) which is comparable to the sulphur content of the fresh engine oil sample.

Flash Point:

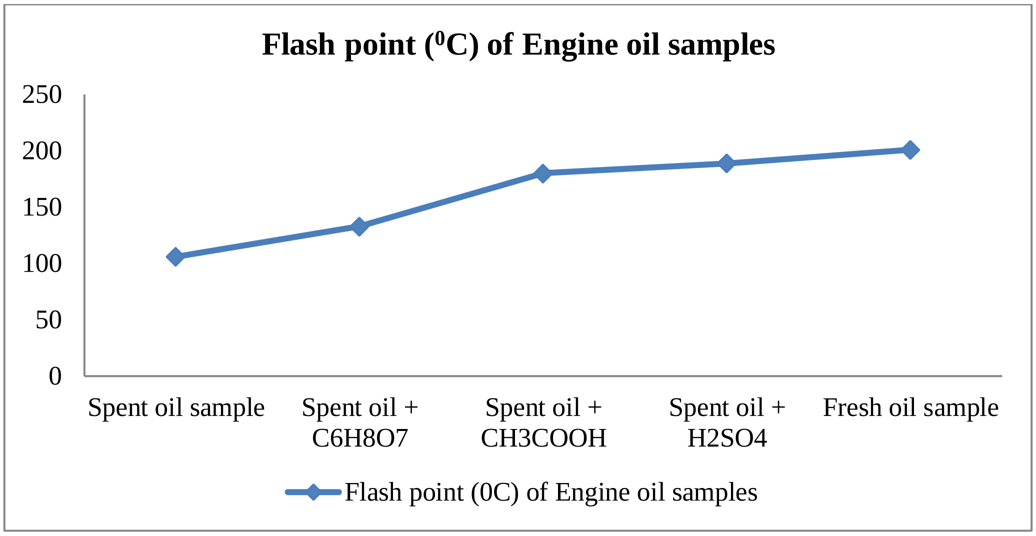

Figure 4: Effect of Flash point on Engine oil Samples

Engine oil's flash point indicates the minimum temperature at which its vapors become flammable, highlighting the importance of using oil with a higher flash point as it signifies the lighter, more volatile components have evaporated, reducing fire risk [25]. A significantly low flash point in engine oil warns of potential contamination with volatile substances like gasoline [19]. From figure 4 the result obtained for the flash points of the purified engine oil and fresh engine oil samples are higher than that of the spent engine oil and are in agreement with the results of the research work reported by [17,13,6]. Also, the results obtained for the purified engine oil samples are higher than 130 ᵒC which is the minimum ASTM limit (ASTM D93) for the regeneration of spent engine oil [26].

The flash point obtained from the purification process using Mineral sulphuric acid (H_2SO_4) is close to and comparable to that of the fresh engine oil.

Pour Point:

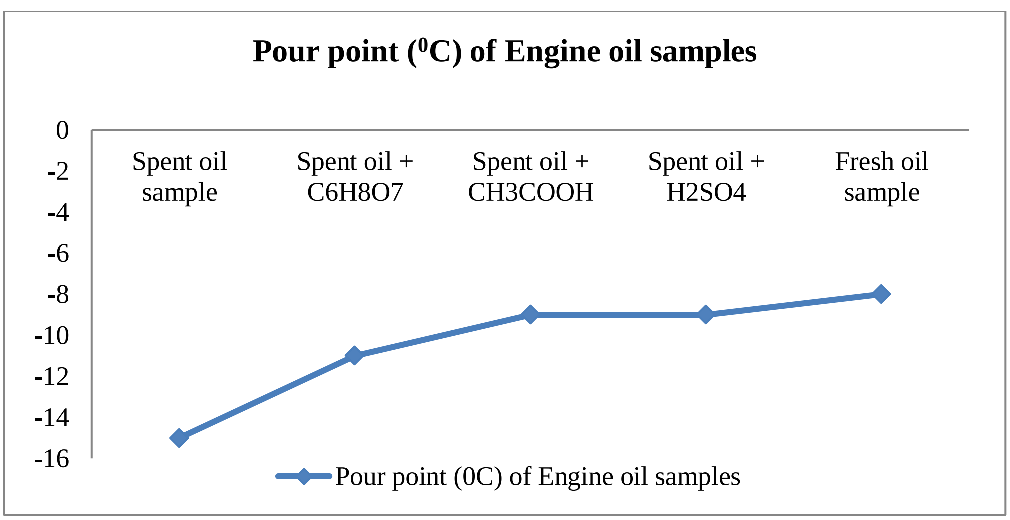

Figure 5: Effect of Pour point on Engine oil Samples

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Engine oil's pour point indicates the temperature at which it becomes too stiff to flow freely. This matters most in cold weather. When the oil is too stiff due to a high pour point, it struggles to reach engine parts during startup [23]. Figure 5 shows the result acquired for pour point of the spent engine oil, purified engine oil samples and the fresh engine oil. The values obtained for the purified oil samples are within the range of -11 ºC to -9 ºC and are higher than that of the spent engine oil which is -15 ºC. This result is in-line with the research work reported by [23, 24]. The acidified clay treatment using Organic Acetic acid (CH_3COOH) and Mineral Sulphuric acid (H_2SO_4) as washing agents gives pour point values that are very close and comparable to that of the fresh engine oil, which indicates that they are very effective in the purification of spent/contaminated engine oil.

Characterization of Engine oil Samples using Fourier-Transformed Infrared Spectroscopy (FTIR) Analysis

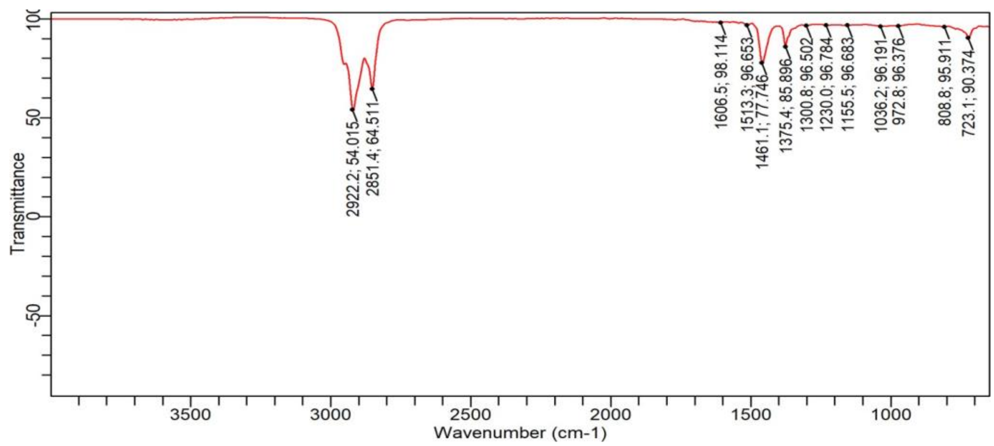

Figure 6: FTIR Analysis of Spent Engine Oil Sample

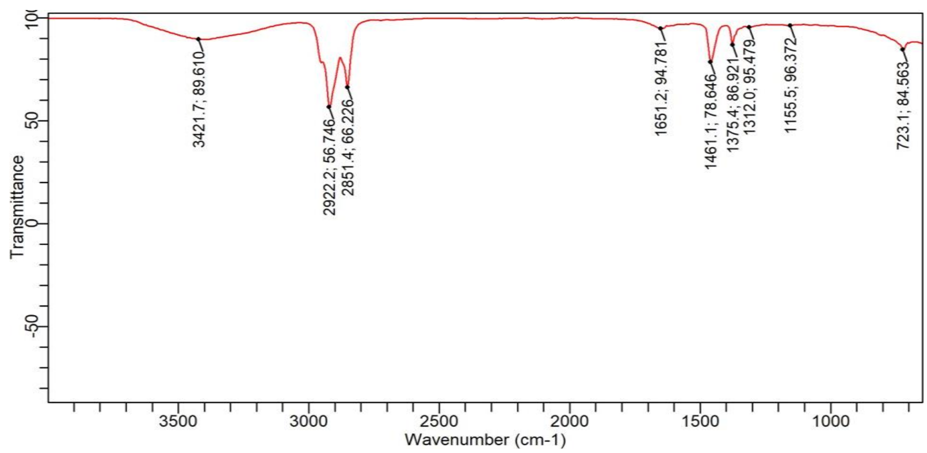

Figure 7: FTIR Analysis of Spent Engine Oil Purified with Organic Citric Acid

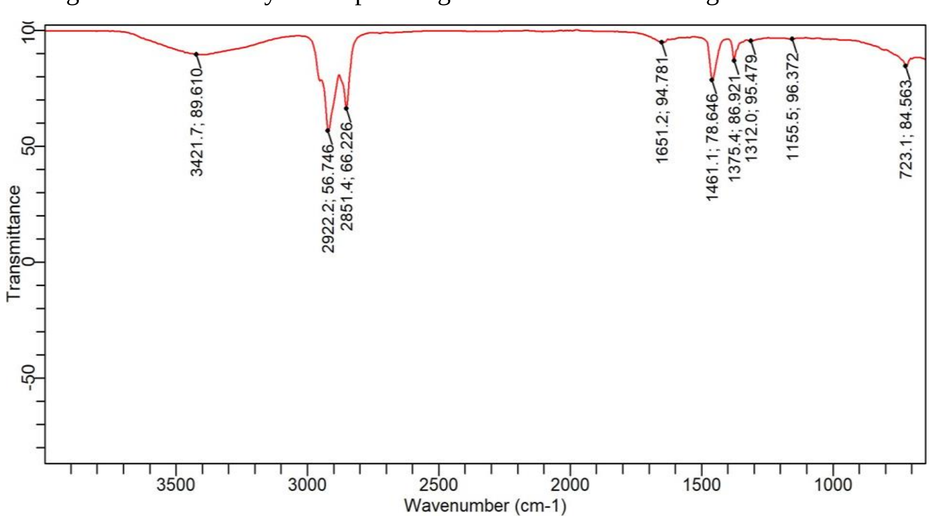

Figure 8: FTIR Analysis of Spent Engine Oil Purified with Organic Acetic Acid

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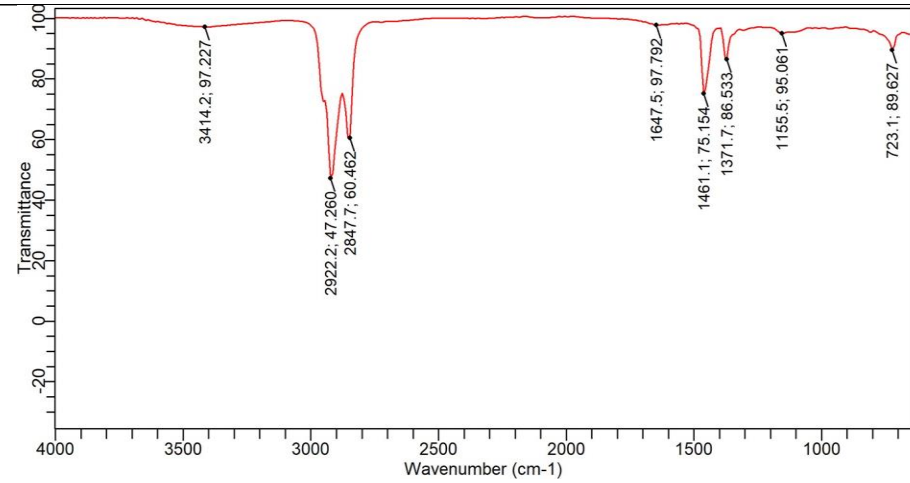

Figure 9: FTIR Analysis of Spent Engine Oil Purified with Mineral Sulphuric Acid

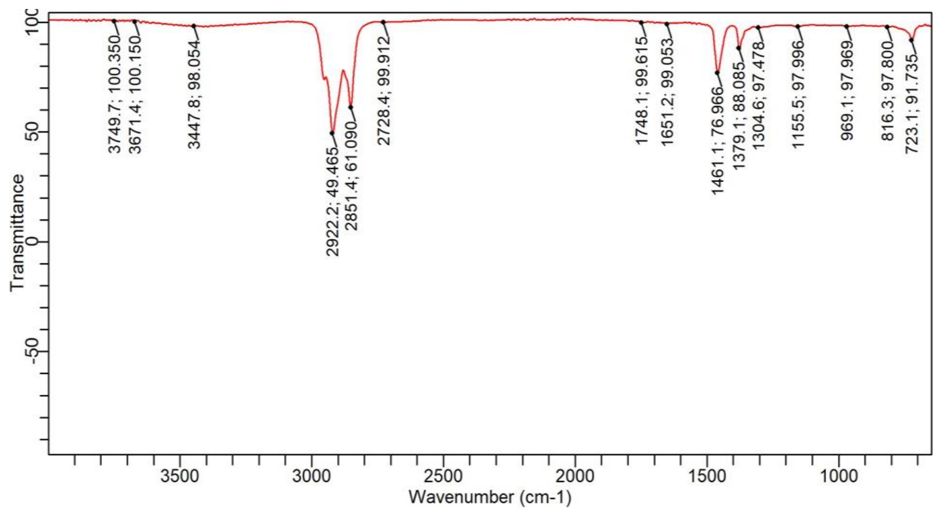

Figure 10: FTIR Analysis of Fresh Engine Oil Sample

FTIR analysis is a technique used to identify the presence of contaminant-attracting molecules, like carboxyl and hydroxyl groups, within a substance [8]. FTIR analysis is a versatile technique capable of examining various sample types, including liquids, solutions, pastes, powders, films, fibers, and gases. It can even analyze materials present on the surfaces of other objects [27]. FTIR spectrometers act as powerful, multi-purpose tools for monitoring oil health. Their ability to create a unique "fingerprint" of the oil's molecules allows for comprehensive analysis of its condition [19]. The FTIR spectra of the spent, purified and fresh oil samples are presented in Figures 6 to 10 respectively. Figure 6 represents the spectra of the spent engine oil. The spectra points at 1230.0 cm^{-1}, 1155.5 cm^{-1} and 1036.2 cm^{-1} shows the presence of primary oxidized products at high temperatures with c-o stretch [27]. They indicate the presence of carboxylic acids. The strong band at 1606 cm^{-1} indicates the presence of nitration products [15] because it falls within the range of 1600-1650 cm^{-1}. Figure 7 represents the spectra of the purified engine oil using acidified clay with organic citric acid ($C_6H_8O_7$). The stretching bands at 1155.5 cm-1 and 1080.9 cm^{-1} shows the presence of primary oxidized products at high temperatures. The presence of nitration products was also noticed at spectra peak of 1651 cm^{-1}. There is also a broad stretching band at 3410.5 cm^{-1}suggest the presence of water, glycol contamination and antioxidant additives because it fell within the absorbance region of 3600-3400 cm^{-1} reported by [11]. Figure 8 represents the spectra of the purified engine oil using acidified clay with organic acetic acid. The spectra peak at 1651.2 cm^{-1} indicate the presence of nitration products as reported by [11] because it falls within the range of 1650-1600 cm^{-1}. Also, a broad spectrum was observed at 3421.7 cm^{-1} which may indicate the presence of antioxidant additives or water. The spectra peak noticed at 723.1 is related to aromatic compounds because it falls within the region of 570-800 cm^{-1} [28]. This method proved to be very effective because it those not show the presence of carboxylic acid and aldehyde at 2359.56 cm^{-1} [6]. Figure 9 represents the spectra of the purified engine oil using acidified clay with mineral sulphuric acid (H_2SO_4). The spectra band at 1647.5 cm^{-1} indicates the presence of nitration products, a broad stretching spectra was also observed at 3414.2 cm^{-1} which suggest the presence of antioxidants additives or water. The acidified clay mineral sulphuric acid treatment indicates the absence of carbonyl compound, aldehyde, primary oxidized products at high temperatures and fuel residue in the spectra and it shows peaks which are equivalent to the peaks of the fresh lubricating oil. This is in line with the result reported by [11] which proved its effectiveness in the purification process.

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IV. Conclusions

The potential of acidified clay with 1M concentration of different acid options was investigated to determine its effectiveness in the purification of spent engine oil**.** The acid options used were mineral sulphuric acid (H_2SO_4), organic acetic acid (CH_3COOH) and organic citric acid ($C_6H_8O_7$) as a novel washing agent, The effectiveness of these acids in the purification process was determined using different characterization techniques such as Specific gravity, Kinematic viscosities at 40 ^oC and 100 ^oC, Pour point, Flash point, Sulphur content and FTIR analysis to determine the functional groups present in the oil samples. The clay sample used for the purification process was also characterized using EDXRF to determine the type and group of clay used. Based on the analysis carried out, the following conclusions were arrived at;

i. The clay soil was analysed and found to be bentonite clay which belongs to Smectite group of clay. Therefore, bentonite clay can be said to be an effective adsorbent for the acidified clay purification technique using mineral sulphuric and organic acetic acids. This is because they give results which are equivalent to that of the fresh lubricating oil in the purification process.

ii. The different characterizations carried out on the spent, purified and fresh engine oil samples indicates that the use of acidified clay treatment with mineral sulphuric and organic acetic acids are very effective for the purification process as compared to the use of acidified clay with organic citric acid. This is observed in the results obtained for the specific gravity, kinematic viscosities at 40 ^oC and 100 ^oC,sulphur content, flash point and pour point. The result showed a substantial improvement in the quality of the spent lubricating oil after going through the purification technique.

iii. The FTIR analysis carried out indicated the absence of contaminants such as carbonyl group in the spectra of the purified engine oil using mineral sulphuric acid (H_2SO_4) and organic acetic acids (CH_3COOH), only nitration products were noticed in their spectra. The FTIR spectra of the spent and purified engine oil using acidified clay with organic citric acid showed the presence of carboxylic acid and nitration product.

iv. From the points ii and iii above it can be concluded that the purification of used engine oil using mineral sulfuric acid (H_2SO_4) and organic acetic acid (CH_3COOH) appears to be more effective than using organic citric acid ($C_6H_8O_7$). Additionally, mineral sulfuric acid (H_2SO_4) demonstrated a slight advantage over organic acetic acid in terms of purification efficacy, effectively treating spent engine oil and producing a comparable quality to fresh engine oil suitable for reuse in combustion engines for lubrication purposes.

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