

Comparative Study of Regeneration of Used Lubricating Oil Using Sulphuric and Oxalic Acids/Clay Treatment Process

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Abstract

This research focused on treatment of used crankcase lubricating oil using sulphuric [H_2SO_4] and oxalic [$H_2C_2O_4$] acids with clay. The effect of these treatments on the properties of fresh, used and regenerated oil tested for were kinematic viscosity at $40^\circ C$ and $100^\circ C$, viscosity index, flash point, pour point, specific gravity, sulphur and metal (Cu, Zn, Fe and Pb) contents, as well as Fourier-transformed infrared spectroscopy [FTIR], and Gas chromatography-mass spectroscopy [GC/MS] analyses. The result showed that viscosity at $40^\circ C$ and $100^\circ C$ increases from 160.94cSt and 13.58cSt for used oil; 265.64cSt and 20.42cSt for oil regenerated with H_2SO_4 and 181.93cSt and 15.54cSt for oil regenerated with $H_2C_2O_4$ as compared to 271.13cSt, 20.9cSt for fresh oil, while flash point increased from $95^\circ C$ for used oil to $186^\circ C$ and $125^\circ C$ for oils regenerated with H_2SO_4 and $H_2C_2O_4$ respectively, as compared to $190^\circ C$ for fresh oil. Similarly, viscosity index increased from 73.0 for used oil to 89.2 and 84.3 for oil sample regenerated with H_2SO_4 and $H_2C_2O_4$ acids respectively, compared to 91.8 of the fresh oil. The FTIR analysis of used oil reveals the presence of oxidized products such as acid, aldehyde and also glycol dilution which were absent after treatment with sulphuric acid/clay. The GC/MS analysis of used oil indicated the presence of benzene based compound, organic acids, esters which were absent in the fresh oil. The results reveal that the quality of lubricating oil was affected by usage due to oil degradation and contamination and that acid/clay regeneration greatly improved the quality of the used oil. The extent of the regenerated oil quality improvement varies with acid type.

Keywords: used oil, regeneration, acid/clay treatment, flash point, viscosity, GC/MS analysis.

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Introduction

Used lubricating oil refers to transmission oil, engine oil, hydraulic and cutting oils after certain

period of application. It also refers to the degradation of the fresh lubricating oil components that become contaminated by metals, ash, carbon residue, water, varnish, gums, and other contaminating materials, in addition to asphaltic compounds which result from the bearing surface of the engines. When these additives and foreign substances, are mixed with lubricating oil, aging, degrading and failure will likely occur, leading to mechanical fault and degraded performance. In such cases, these oils must be replaced from the automobiles, machineries or utility equipment after certain period of application [1].

These used lubrication oils have significant impact on both economic and environmental aspects, when discharged on land, water or even burnt as a low grade fuel, may cause serious pollution problems, releasing harmful metals and other pollutants into the environment [1].

During usage lubricating oil deteriorates and loses its efficiency due accumulation of foreign matters such as metal powder, filings, as well as additives, becomes oxidized, increases its acid value generating precipitate, oil sludge, and varnish. If these materials deposit on the surface of the engine and the flow passage cause malfunctions of the machine reduce its overall productive efficiency. Generally lubricating oil becomes unfit for further use for two main reasons: accumulation of contaminants in the oil and chemical changes. These effects interfere with the basic properties of lubricating oil which are peculiar for their effective performance during application [2].

The concentration of oil contamination these impurities depends purely on the application to which the particular oil is put to. Generally, the contaminants from the engine are usually as a result of metallic particles resulting from wear of the engine (Fe, Cu and Al); carbonaceous particles (soot and carbon originate due to incomplete fuel combustion); water as a product of fuel combustion or from leakage of the cooling system; metallic oxides present as corrosion products of metals and fuel or fuel additives or their by-products, which might enter the crankcase of engines [3].

During operation of engine, lubricating oil temperature builds up and degrades the oils leading to decline in important properties such as viscosity, specific gravity and flash point. Aside this dirt particle and worn out metal parts from engine surfaces are deposited into the oils. With the passage of time, these engine oils lose their functional properties and must be replaced with fresh lubricant. Considering the fact that huge quantities of lubricating oils are used on a daily basis, the frequent dumping of lubricating oils has become a source of serious concern. It is on this premise that most countries of the world are presently paying serious attention to the menace of environmental degradation caused by the disposal of waste or used lubricating oils [4].

Material and Methods

Sample collections: A plastic container [5 litres] was used to collect the sample of used Oando Oleum Super motor oil SAE 40 from Toyota Corolla salon car after covering a distance of 3500km using Drain Stream method [5]. While its corresponding virgin oil was procured from Oando Filling Station opposite NNPC Mega Station in Sokoto metropolis, Sokoto State. Clay sample was collected from Kwakwalawa area of Usmanu Danfodiyo University Sokoto, Sokoto state.

Clay Preparation: Untreated clay sample [200g] was grounded and mixed with distilled water [500cm³]. The slurry was then kept in an oven to dry up [at 105°C for 6hrs]. The dried clay was thereafter grounded and sieved into very fine particle size using 74µm mesh sieve [6].

Activation of Clay: Clay [200g of 74µm sized] was mixed thoroughly with 400cm³ of 1M H₂SO₄ in a round bottom flask [1000cm³]. The suspension was heated on a magnetically stirred hot plate at 200°C for 3 hours. The resulting slurry was poured into a Buchner funnel to separate the acid and clay. The residual clay was washed severally with distilled water until neutral point was obtained, which was then dried in an oven at 105°C for 8 hours and reground to the 74µm size [6].

Pre-treatment of used oil: Filtration of the used oil was carried out to remove impurities such as sand, metal chips, micro impurities that

contaminated the used 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. The used oil was filtered and was then allowed to settle for 24 hours, pre-treated at 45°C for 30 mins to degrade some of the additives and reduce the work load of the acid [7].

Acid/clay treatment of pre-treated oil: Each of the pre-treated oil [300cm³] was measured into two different beakers and acid [30 cm³] each was introduced; the first and second beakers were treated with sulphuric and oxalic acid respectively. Each beaker was shaken thoroughly to ensure homogenous mixing of the acid, and the sample was allowed to stand for 30 minutes. The oil samples were transferred into two different separating funnels fixed to a retort stand and left undisturbed for 24 hours to allow the acid sludge to settle. The acid sludge was discharged at the bottom of each separating funnel and 30g of acid activated clay was packed in a Buchner funnel, with a filter paper inserted in it. This was plugged to a vacuum pump, allowing the sulphuric acid treated oil to pass through the bed of the clay, the procedure was repeated for oil treated and oxalic acid. 100cm³ solution of 10% NaOH [caustic soda] was added each to neutralize the acid of the two oil samples, the oil was allowed to sediment for 24 hours and was decanted into a beaker while the residue at the bottom was discarded. The regenerated lubricating oil was obtained for analysis [7].

Results and Discussion

The results of the characterization of the used, fresh and regenerated lubricating oils (SAE40 Oleum Super) are presented in Table 1 below

TABLE 1: Results of the Physicochemical Properties of Fresh, Used and Regenerated Oil.

S/N	Test	Fresh Oil	Used Oil	Oil Regenerated with Acid/Clay	
				H ₂ SO ₄	H ₂ C ₂ O ₄
1	Kinematic Viscosity @ 40 [cSt]	271.13	160.94	265.64	181.93
2	Kinematic Viscosity @ 100 [cSt]	20.9	13.58	20.42	15.54
3	Viscosity Index	91.8	73	89.2	84.3
4	Pour Point [°C]	-6	-11	-6	-8
5	Flash Point [°C]	190	95	186	125
6	Specific Gravity Kg/cm ³	0.825	0.8624	0.8261	0.8423
7	Sulphur Content [wet%]	N/A	0.958	0.052	0.06
8	Metal Content [ppm]				
i	Zinc [Zn]	3.840	22.75	6.173	9.238
ii	Iron [Fe]	0.059	1.765	0.497	1.425
iii	Lead [Pb]	0.071	2.237	0.141	0.894
iv	Copper [Cu]	0.003	0.99	0.005	0.031

Kinematic Viscosity

Viscosity testing can indicate the presence of contamination in used lubricating oil. Oxidation of motor oils during usage in an engine environment produces corrosive oxidized products, deposits, and varnishes which lead to an increase in the viscosity [8]. Result of kinematic viscosity of the fresh, used and treated oil presented in Table 1. The kinematic viscosity at 40^oC and 100 ^oC of fresh oil are 271.13cSt and 20.9cSt respectively; used oil is found to be 160.94cSt and 13.58 cSt respectively, while the oil sample treated with H₂SO₄ and H₂C₂O₄ gave a kinematic viscosity of 265.64cSt, 20.42cSt and 181.93cSt, 15.54cSt at 40 and 100 ^oC respectively. The result for kinematic viscosity of the used oil is higher than 136.6cSt and in line with 13.5cSt reported by Hamawand et al.[1]. The kinematic viscosity of the regenerated oil, on the other hand, are higher than 169.5cSt and 16cSt at 40^oC and 100 ^oC respectively, reported by Hamawand et al. [1]. The types of acid and clay used for the treatment greatly influenced the viscosity of the treated oil [1]. Hence, the sample recycled with H₂SO₄ gives a viscosity that is very close to that of fresh oil indicating that the method is effective in removing the oxidized products, deposits, and varnishes from the used oil [8].

Viscosity Index

Viscosity index [VI] is the viscosity-temperature relationship and is an arbitrary number indicating the degree of change of viscosity of a lubricant with change in temperature [9]. The results in Table 1 above indicate that the viscosity index of fresh oil is 91.8, that of the used oil is 73.0, while that of the regenerated oil are 89.2 and 84.3 for oil sample treated with H₂SO₄ and H₂C₂O₄ respectively. However, the result of the used oil treated with H₂SO₄/clay is in agreement with the result (89.11) reported by Hamawand et al. [1]. This is an indication that the used oil has lost its viscosity due to oxidation, thermal degradation, contamination, corrosion and wearing during usage [10]. Thus, the used oil has lost its resistance to flow. The result obtained for the regenerated oil is in line with 88.6-80.2 range reported by Udonne and Bakare, [11], which shows that oil sample treated with H₂SO₄ gave a higher VI value is close to the fresh oil and is comparable to the result obtained for oil sample treated with H₂C₂O₄ .

Flash Point

The flash point of engine oil is the lowest temperature to which the oil must be heated under specified conditions to give off sufficient vapour to form a mixture with air that can be ignited spontaneously by a specified flame. A substantially low flash point of engine oil is a reliable indicator that the oil has become contaminated with volatile products such as gasoline and increases with

increasing molecular mass of the oil [12]. The flash point of fresh oil is 190°C, which could be due to different additives that improve its flash point. The results obtained for flash point of the used oil is 95°C is below the 130°C minimum specification of SAE-40 lube oil, as a result of contamination with fuel and oxidation products [12]. Similarly the flash point of sulphuric and oxalic acid/clay treated oils are 186°C and 125°C respectively, which are lower than 120°C reported by Udonne [13] and 158°C reported by Hamawand *et al.* [1] for used oils. However, the result of sulphuric acid/clay treated oil is higher than 173°C reported by Udonne and Bakare [11] the 130°C minimum standard specification of SAE-40 lube oil indicating a high influence of the sulphuric acid in regenerating used oil.

Specific Gravity

Specific gravity is the ratio of the density of a substance to the density of a reference substance and it increases with increase in the amount of solids in the used engine oil [14]. The results of the specific gravity of the fresh and used lubricating oils were 0.8250 and 0.8624 respectively, while those of the oils treated with acid/clay treatment methods were 0.8261 for sulphuric acid and 0.8423 for oxalic acid treatment. The values obtained for used oil in this research was lower than 0.9261 reported by Hamawand *et al.* [1], while the values obtained for the oils treated with sulphuric and oxalic acid/clay treatment methods are lower than 0.86 reported by udonne [13]. The Result obtained for the used oil treated with sulphuric acid /clay improves the specific gravity of the oil and makes it comparable to the specific gravity of the fresh oil.

Pour Point

Pour point is a measure of the temperature at which the oil ceases to flow under service conditions of a specific system [13]. The result obtained for the pour point of the fresh oil was -6°C and that of the used oil was -11°C which are lower than -5°C reported by Hamawand *et al.* [1], while the result of the sulphuric and oxalic acid/clay treated oil samples gave pour point values of -6°C, and -8°C respectively, which are higher than -11°C reported by udonne [13]. This result shows that the H₂SO₄/clay treatment proves to be more effective method for used oil regeneration than the organic acid.

Sulphur Content

Result from Table 1 shows the absence of sulphur in the fresh oil, while used oil contains sulphur of

0.958 wt.% which could be as a result wears caused by moving parts. The sample treated with H₂SO₄ and H₂C₂O₄ acid /clay gave sulphur content of 0.052 and 0.060 wt.% respectively. The result of the used oil is higher than 0.8 wt % reported by udonne,[13] and the results of regenerated oil are higher than 0.04 wt% reported by udonne,[13]. The mineral acid/clay method proves to be a better a method with the lowest sulphur content of 0.052 wt.%.

Result of the Metal Content by AAS

In this research the concentrations of four (4) metallic elements was determined [Cu, Zn, Fe and Pb]. The wear of these metals is due to the corrosion caused by the presence of water and fuel dilution due to bad piston rings and also due to oil contamination [15]. The Result of wear metal contamination is presented in Table 1 above. From the table no wear metals were found in the fresh oil because it has not been used in engine combustion.

Zinc (Zn)

Zinc is introduced to base oil in the form of additives package as anti-oxidant, corrosion inhibitor, anti-wear, detergent and extreme pressure tolerance in the , such as Zinc diethyldithiophosphate (ZDDP) [16]. Table 1 results show the concentration of Zinc in fresh oil is 3.840ppm which is as a result of additives present in the oil. A high concentration of Zinc [Zn] was observed in the used oil with value 22.750 ppm, and concentrations of 6.173 ppm and 9.238 ppm were observed in mineral acid (H₂SO₄) and organic acid (H₂C₂O₄)/ clay treated oil respectively. The used oil concentration is in line with 13.0-73.6ppm range reported by Friday *et al* [17]. However, the results of the acid/clay treated oil are lower than 1050ppm reported by Hamawand *et al.*,[1]. The increase in the zinc concentration in used oil is due to the wearing of the galvanized piping as well as the decomposition of anti-oxidant, anti-wear additives whose basic element is zinc. Re-refining of oil with H₂SO₄ is most effective because H₂SO₄ reacts with zinc more to produce precipitated compounds than oxalic acids. . It is to notes that the order of removal by the acid is H₂SO₄ (73%) > H₂C₂O₄ (59%).

Iron (Fe)

Iron [Fe] in used oil usually comes from various engine such as liners, camshafts and crank shaft, pistons, gears, rings, and oil pump. Iron concentration in engine oil depends on the bearing conditions inside the engine[18] The concentration of iron [Fe] in fresh and used oil is 0.059ppm and 1.765ppm respectively while that of the acid/clay

treated oil are 0.497ppm and 1.425ppm for sulphuric and oxalic acid respectively. The concentration of used oil is in line with [1.5-5.4ppm] range reported by Friday *et al* [17]. The results from the acid/clay treatments tends to support the report of Udonne [13] (0ppm) and Henry [19] (2.6ppm) who reported small removal of Fe from engine oil using clay/charcoal/acid treatments. However the Fe content the provision of SAE, stipulates that Fe contents in used oil should not be greater than 100-200ppm [5]. Oil sample treated with H₂SO₄ acid reduces iron concentration followed by oil treated with H₂C₂O₄ acid.

Lead (Pb)

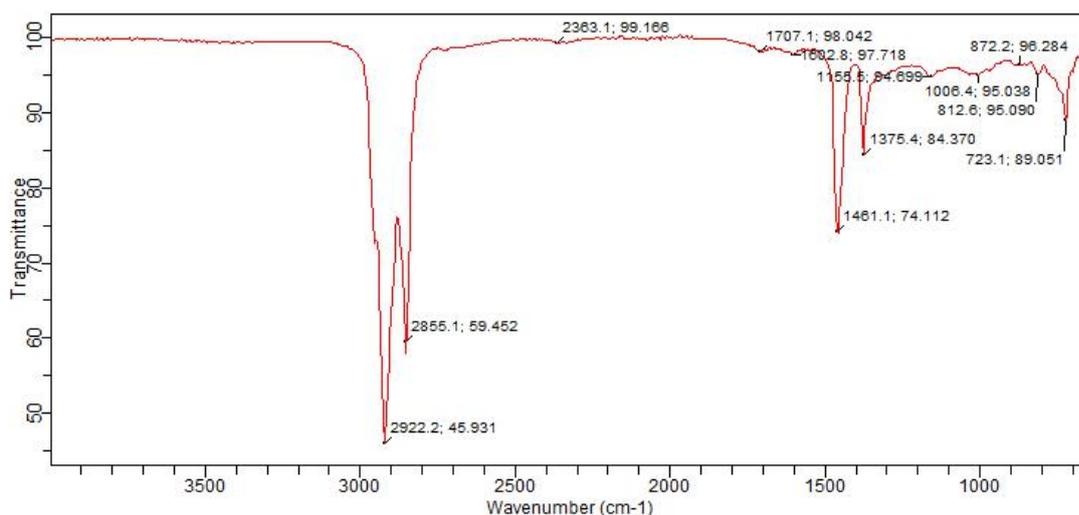
Lead (Pb) is associated with bearing wear, fuel source (leaded gasoline), and contamination due to the use of galvanized containers [20]. Table 1 shows lowest concentration of Pb in fresh oil (0.071ppm) and result indicates high concentration of Pb in the used oil (2.237ppm) compared to re-refined oils (0.141ppm and 0.894ppm) to suggest that all acids reacted with lead ion to form salts. The results obtained in this work fall within the range reported by Zajac *et al.* [21] (2.2-4.3ppm) and is lower than 8.0ppm reported by Casap [22]. Results of the acid/clay treated oil are lower than 1.86ppm

reported by Henry [19]. Overall, re-refined oil, with H₂SO₄ prove more effective in reducing lead concentration in the oil.

Copper (Cu)

Copper (Cu) is introduced to engine oils after use from bearings, wearing and valve guides. Engine oil coolers can also be contributing to copper content along with some oil additives [23]. The amount of Cu metal presented in the fresh oil, used engine oil and in the refined products (Table 1) is a trace amount compared to Fe, Zn or Pb which suggest that it may have come from decomposition of additives introduced in smaller quantities or from wearing of piston and valve guides. Cu concentration in fresh oil is 0.003ppm and used oil contains highest concentration of copper with 0.990 ppm, the concentration of 0.005 ppm and 0.031 ppm was observed in sulphuric and oxalic acid/clay treated oil respectively. The used oil result is lower than 2.4ppm reported by Friday *et al.*, [17] and also result of the acid/clay treated oil is higher than 0.00ppm reported by Hamawand *et al.*, [1]. Oil treated with mineral acid [H₂SO₄] proves to be more effective in reducing the copper concentration followed by oil sample treated with organic acid [H₂C₂O₄] because sulphuric acid is more reactive with copper than oxalic acid and forms a precipitate.

Figure 1 FTIR Spectroscopy of Used Oando Oleum Super [SAE 40] oil



primary oxidized products at high temperatures, while peaks at 1707.1 and 1802.8 cm^{-1} indicative short-chain compounds with carbonyl groups from esters, ketone or acids [25], which are compounds commonly formed in motor oil via chemical oxidation processes [26].

Figure 3 indicates that the oil treated with sulphuric acid-clay method shows no carbonyl compounds. Figure 4 shows that the oil treated with oxalic acid-clay method indicated some oxidized component at

2359.4 cm^{-1} indicating the presence of carboxylic acid and 3444.13 cm^{-1} which suggests the presence of water, glycol contamination and antioxidant additives because it fell within the absorbance region of 3600-3400 cm^{-1} reported by Toms[27]. Therefore oil treated with sulphuric acid-clay method gives the best result showing peaks closer to the peaks of the fresh oil and also result obtained is in line with that reported by Hamawand et al.,[1] which also shows no aldehyde and no carboxylic acid at 2359.56 cm^{-1} .

Table 2 GC-MS Spectrometry of Used [SAE40] Oando Oleum Lubricating Oil.

S/N	Retention	Peak	Name of Compound	Molecular	MW	Quality
1.	5.372	0.60	Mesitylene	C9H12	120	91
2.	5.835	1.98	O-Cymene	C10H14	134	94
3.	6.119	1.10	1,2,4,5,tetra methyl benzene	C10H14	134	95
4.	6.688	3.43	Naphthalene	C10H8	128	91
5.	7.613	1.18	2-methyl naphthalene	C11H10	142	95
6.	5.835	1.98	4-ethyl-1,2-dimethyl	C10H14	134	94
7.	28.172	74.87	Octadecadienal	C18H32	264	95
8.	5.372	0.60	1,2,3,trimethyl benzene	C9H12	120	94
9.	5.550	2.30	1-methyl-3propyl benzene	C10H14	134	81
10.	7.115	1.71	2,3-dihydr-4,7-dimethyl-1H-	C11H14	146	81
11.	7.506	1.48	1-methyl naphthalene	C11H10	142	94

Table 3 GC-MS Spectrometry of Fresh [SAE40] Oando Oleum Lubricating Oil.

S/N	Retention	Peak	Name of Compound	Molecular	MW	Quality
1.	5.835	2.62	Undacane	C11H24	156	93
2.	7.257	1.51	Tridacane	C13H28	184	95
3.	7.471	0.53	2-methyl naphthalene	C11H10	142	93
4.	7.933	1.54	Tetradecane	C14H30	198	96
5.	8.645	3.06	Pentadacane	C15H32	212	93
6.	9.143	2.37	Cyclopentadecane	C15H30	210	93
7.	9.427	4.14	Hexadecane	C16H34	226	95
8.	10.352	6.30	Heptadecane	C17H36	240	97
9.	11.454	6.55	Octadecane	C18H38	255	97
10.	12.806	6.25	Nonadecane	C19H40	268	96
11.	14.407	8.06	Eicosane	C20H42	282	98

Table 4 GC-MS Spectrometry of Used [SAE40] Oando Oleum Lubricating Oil Treated with Sulphuric Acid/Clay

S/N	Retention	Peak	Name of Compound	Molecular	MW	Quality
1.	8.396	0.12	Dodecane	C12H26	170	55
2.	9.463	0.11	Hexadecane	C16H34	226	95
3.	10.388	0.13	Heptadecane	C17H36	240	96
4.	12.877	0.15	Nonadecane	C19H40	268	96

5.	14.478	0.22	Eicosane	C20H42	282	98
6.	32.404	95.11	Octacosane	C28H58	395	93

Table 5 GC-MS Spectrometry of Used [SAE40] Oando Oleum Lubricating Oil Treated with Oxalic Acid/Clay

S/N	Retention	Peak	Name of Compound	Molecular	MW	Quality
1.	5.408	0.48	1,2,3 trimethyl benzene	C9H12	120	94
2.	5.657	1.50	2-ethyl -1, 4 dimethyl	C10H14	134	93
3.	6.191	1.31	1,2,4,5,tetra methyl benzene	C10H14	134	95
4.	8.431	1.63	1,6- dimethyl naphthalene	C12H12	156	96
5.	8.716	0.68	Pentadecane	C15H32	212	92
6.	9.072	0.55	Naphthalene	C10H8	128	90
7.	9.534	1.04	Heptacosane	C27H56	380	97
8.	10.459	0.69	Heptadecane	C17H36	226	92
9.	11.633	0.69	Heptacosane	C27H56	380	97

Gas Chromatography/Mass Spectroscopy [GC/MS]

GC/MS is a separation technique that is applied to the light ends of a lubricant to separate portions of base stocks, as well as many of the smaller additives [28]. When linked to the mass spectrometer detector, the GC/MS can also identify the components being separated and can increase the detectability of the compound separated [29]. GC/MS can measure the concentrations of the additives, such as antioxidants, as low as 10ppm [30] and enables the analyst to identify both the types of molecular species present and their concentrations and accurately determine the concentrations of certain contaminants, especially fuel and glycol- in used oil sample [28]. The GC-MS analysis was carried out in order to determine the hydrocarbon compounds and other oxidation product present. Table 2 to 5 above represented the GC/MS analyses of both fresh, used and treated oil.

GC/MS analysis of the fresh oil in (Table 3) did not reveal the presence of any oxidative products and carboxylic acid. However, the spectrum of naphthalene-based compound which is 2-methyl naphthalene was also detected. Other hydrocarbons compound such as undecane, tridecane, tetradecane, pentadecane, cyclopentadecane, hexadecane, heptadecane, octadecane, nonadecane and eicosane were also detected in the fresh lubricating oil.

GC/MS spectrum of the used engine oil (Table 2) revealed numerous new compounds such as mesitylene, o-cymene, 2,3-dihydro-4,7-dimethyl-1H-indene. The carboxylic acid identified in the GC/MS spectra of the used oil was methoxyacetic acid 3-tetradecyl ester. It also identified the prevalence of benzene-based and naphthalene related compounds in the used oil; some of the compound identified are 1,2,4,5-tetramethyl benzene, 4-ethyl-1,2-dimethyl benzene, 1,2,3-trimethyl benzene, 1-methyl-3-propyl benzene while the naphthalene related compound include naphthalene, 1-methyl naphthalene, 2-methyl naphthalene. This result is in agreement with the work of Dabai [31] who identified 1,2,3-trimethyl benzene, 1-ethyl -2-methylbenzene, propenylbenzene, 1-btenylbenzene and also 1-methyl-2-[2-prpenyl]-benzene in used oil and the work of cotton *et al.* [32], who detected the presence of naphthalene, acenaphthalenes, dinaphthenoanthracenes, benzo[α]pyrene, benzo[α]anthracene in used oil samples. Furthermore, other hydrocarbon compound such as octadecadienal was detected in the used engine oil, which is partly in agreement with the work of Scapin *et al.*, [33] who reported the abundance of hexadecane, tridecane and eicosane in used engine oil.

GC/MS analysis of the used oil treated with sulphuric acid presented in (Table 4) above indicated the absence of new compound detected in used oil spectra and carboxylic acid was also not

detected in the spectra, while other hydrocarbon such as dodacane, hexadecane, heptadecane, nonadecane and eicosane was detected in the spectrum.

GC/MS analysis of the used oil treated with oxalic acid in (Table 5), identified the prevalence of benzene-based and naphthalene related compounds in the treated oil; the benzene based compound identified were 1,2,4,5-tetramethyl benzene, 2-ethyl-1,4-dimethyl benzene, 1,2,3-trimethyl benzene, while the naphthalene related compound include naphthalene, 1,6-dimethyl naphthalene, Other hydrocarbon such as pentadecane, heptacosane, heptadecane, were also detected in the spectra of the treated oil..

Conclusions

Based on the results obtained in this research the following conclusion can be arrived at:

- i. It can be inferred that using mineral acid and clay treatment method effectively improved the quality of the oil.
- ii. Used oil treated with mineral acid [H₂SO₄] gave a better results compared to that of organic acid (H₂C₂O₄). AAS Result also show that metals such as Zn, Fe ,Pb and Cu present in the used oil were reduced after treatment with mineral acid/clay.
- iii. FTIR analysis indicated that primary oxidized product, carbonyl compound and fuel residue present in the used oil spectra were absent after treatment with mineral acid/clay (H₂SO₄).GC/MS spectrum of used oil reveals the presence of numerous new compound and organic acid, ester but was found absent after treatment with mineral acid/clay (H₂C₂O₄).
- iv. Hence, the treatment oil can be reuse for lubricating purpose. Mineral Acid/clay proved to be a good adsorption that can be used to regenerate used engine oil.
- v. Based on the result presented above the GS/MS analysis of used oil treated with sulphuric (mineral) acid shows result that is comparable to the GC/MS result of the fresh oil and therefore proves to be the best

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