

Identification of compounds in spent oil contaminated soil

Abstract

The indiscriminate disposal of spent motor oil in the soil has led to the contamination of both land and water. The present study analyzes the compounds in spent motor oil from a car and that extracted from the soil from **two auto mechanic** workshops located in Gwagwalada area council of FCT, Abuja Nigeria. The samples were cold extracted in dichloromethane. The chemical compositions of the extracts were analyzed using GC/MS. The results revealed that extracts from SDP mechanical workshop (SMW), Dagiri mechanical workshop (DMW); and spent oil from a car (SCO) had carbon compounds that ranged from C_{15} – C_{35} ; C_{13} – C_{34} ; and C_{10} – C_{20} respectively. About thirteen polycyclic compounds, eleven monocyclic compound, five oxygenated compounds and eight hydrocarbons were identified. The results from this study give information on the toxic nature of spent oil, hence, informing and educating the auto mechanic engineers on the need to stop indiscriminate disposal of spent oil on soil. It also avails the government of the urgent need to enact and enforce policies on collection, management and treatment of spent oil before disposal.

Keywords: spent oil; mechanic workshop; GC/MS; polyaromatic hydrocarbons

Introduction

Motor oil is a complex mixture of heavy petroleum hydrocarbons and other organic compounds, including some organo-metallic constituents. It is used to lubricate parts of an automobile engine so as to smoothen engine operation [1]; cool the engine by carrying heat away from moving parts; prevent corrosion and degradation of oil; keep the pistons and ring, etc clean from sludge formation; reduce deposits in the combustion chamber; reduce friction and lubricate the engine parts [2]. Spent motor oil has been defined as any lubricating oil that has served its service properties in a vehicle, been withdrawn from area of application and considered not fit for its initial purpose because of contaminants [3-4]. This oil enters the soil environment via indiscriminate disposal at the mechanic workshop into gutters, water drains, open vacant plots and farmlands, a common practice by motor and generator mechanics [5-6]. Spent oil pollution also occurs when it is applied on gravel for dust control in rural areas; escapes into the environment from the engine during operation [2] and singeing the skin of animal. Oil improperly disposed of on land surfaces leaches into the soil and contaminates ground water [7].

Engine oil contains some additives such as: detergents and dispersants, zinc diaryl, molybdenum disulphate, zinc dithiophosphate, and other organometallic compounds [8]. These additives are added to increase and improve the properties of the oil such as: viscosity, anti-wear and anti-corrosion abilities, thermal and oxidation stability, for better efficiency and life span of the oil [2]. More so, antioxidants and corrosion and rust inhibitors protect the base oil and improve the base oil performance. When these additives undergo chemical and physical reactions in the engine, they form the chemical impurities found in spent oil [9]. When materials leak from the combustion chamber to the lubricating oil in the engine, contamination can also occur [2]. Orji *et al.* [5] and other authors [8, 10-11], have confirmed the presence of heavy metals in the spent oil contaminated soils. Most heavy metals such as: Pb, Zn, Cd, Fe, Cu, Al, Cr and Ni result from the wearing of engine parts during operation [2]. Bio-accumulation of these heavy metals in the ecosystem may result in high concentrations that may lead to heart, liver or kidney disease, possible damage to the bone marrow, and an increased risk of cancer and finally death of both plants and animals [5, 12]. In previous studies, [13-14], it has been established that PAHs are the most important environmental carcinogenic factors, impairing the structure of DNA and consequently leading to mutation. Therefore, this study is centred on assessing the chemical composition of spent oil extracted from two contaminated soil and a car, to ascertain the presence of mono and poly-aromatic hydrocarbons.

Materials and Methods

The spent oil contaminated soils were sampled from two auto mechanic workshops soils locate in Illorin and tipper garage in Gwagwalada Area Council Abuja of Nigeria. The sampling was carried out following the procedure of Orji *et al.* [5], as random samples were collected from depth of 0- 15 cm. Fresh spent oil from a car was also collected after the car servicing. The soil samples were air dried and cold extraction carried out via a modified method by Haque *et al.* [15] and Okop and Ekpo [16] using dichloromethane as solvent. The oil extracts were then subjected to GC/MS analysis.

Agilent technology 7890A GC system and Agilent technology 5975C VL MSD were used for the analysis. Details of the GC parameters include:
 Stationary phase: Length, 30 m; Diameter, 0.32 mm; Thickness of Column, 0.25 μ m; Oven Temperature, 60 °C for 5 min; 60 °C to 300 °C at 10 °C/min; Sample maximum Run Time, 30 min 50 sec. And Detector Mass spectrophotometer: Detector Temperature, 250 °C; Injection Temperature, 250 °C, Volume of Injection: 1 μ l

Results

The chromatogram of the extracts from DMW, SMW and SCO oil are shown on fig 2, 3, and 4 respectively.

Table 1 indicates the compound found in the extracts SMW. From the table, a total of seven compounds were identified. The peak area ranged between 2.05 and 45.86 %. 1,2-benzenedicarboxylic acid-2-butoxyethyl butyl ester had the highest area percentage of 45.86 %, followed by 2(1H)-naphthalenone, octahydro-4a, 5-dimethyl-3-(1-methylethyl)-, (3.alpha.,4a.beta.,5.beta.,8a.alpha.) at 9.12 % and then by 17-pentatriacontene at 6.01 %. However, 22.beta.-Acetoxy-3.beta.,16.alpha.-dihydroxy-13,28-epoxyolean-29-al, an alkanal, had the least area percentage of 2.05.

Only two hydrocarbon compounds were identified in SMW: 17-pentatriacontene (C₃₅H₇₀), at 6.01 % peak area and nonacos-1-ene (C₂₉H₅₈), at 4.53 % peak area. Therefore the total peak area percentage of hydrocarbons in the extracts of DMW was 10.53 %. There were five compounds that contained carbon, hydrogen, and oxygen while demecoline was the only compound containing nitrogen in addition to carbon, hydrogen and oxygen.

The compounds identified in the extracts of DMW are shown in Table 2. From the table, about thirteen compounds were identified in soil sample. These compounds include: *two alkanes*: cyclotetracosane and 2,6,10,14-tetramethyl-7-(3-methylpent-4-enylidene) pentadecane; *five alkenes* compounds: 1-docosene, 1-hexacosene, 1-nonadecene, 9-tricosene, (Z)- and 1-tricosene; *One alkanol*: 2-tetradecanol; *two halogenated compounds*: fumaric acid, 2,2-dichloroethyl tridecyl ester and 1,3-benzenedicarboxylic acid, 5-[[[(4-bromophenyl)sulfonyl]oxy]-, dimethyl ester; hexasiloxane, tetradecamethyl-, the only compound containing silicon; *One alkanoic acid* compound: 9-Octadecenoic acid (Z)-, 9-hexadecenyl ester, (Z)-; hexahydropyridine, 4-[4,5-dimethoxyphenyl]- was the only nitrogen-containing compound;

The peak area of these compounds identified in the extracts of DMW ranged between 0.10 % and 22.36 %. 1-docosene had the highest peak area of 22.36 %, seconded by 1-hexacosene at 15.11 % and then by 2-tetradecanol at 13.37 %. The compound with the least peak area was 1-tricosene with an area of 0.10 %. This implies that the concentration of hydrocarbons (%) was more than that of any other compound in the extracts of DMW.

The GC/MS analysis revealed about 21 compounds from the extracts of SCO, as specified on Table 3. The compounds identified in the extract include: 7-methyl-octadecane, the only alkane compound; hexadecanoic acid, methyl ester, the only falkanoate; 2-amino-5-isopropyl-8-methyl-1-azulenecarbonitrile, 7-Fluoro-3,4-dihydro-3-[4-(trifluoromethyl)phenyl]-1,9(2H,10H)-acridinedione and pyrimidin-4-ol,5-allyl-6-(2-hydroxyethylamino)-2-methyl- three nitrogen-containing compounds; 7-Fluoro-3, 4-dihydro-3-[4-(trifluoromethyl) phenyl]-1,9 (2H,10H)-acridinedione the only halogenated compound;

About eleven aromatic compounds were identified in the extracts of SCO. Among these were: one mono-aromatic, nine di-aromatic and one tri-aromatic compounds named phenanthrene, 2-methyl-

Some of the cycloalkenes identified include: [4.2.2]Propella-2,4,7,9-tetraene; 1,7-dimethyl-3-phenyltricyclo[4.1.0.0(2,7)]hept-3-ene and 1,5,6,7-tetramethyl-3-phenylbicyclo[3.2.0]hepta-2,6-diene

The peak areas of the compounds identified in SCO ranged between 16.70 and 1.41 %. One of the three nitrogen-containing compounds, 2-Amino-5-isopropyl-8-methyl-1-azulenecarbonitrile had the highest peak area of 16.70 %, seconded by 1,5,6,7-Tetramethyl-3-phenylbicyclo[3.2.0]hepta-2,6-diene at 12.21 % peak area and then, naphthalene, 1-methyl- with a peak area at 6.25 %. Meanwhile, pyrimidin-4-ol,5-allyl-6-(2-hydroxyethylamino)-2-methyl- had the least peak area of 1.41 %. Therefore, it implies that the extracts of SCO contain majorly complex aromatic compounds rather than simple compounds of hydrocarbon.

The difference in the peak areas of some identified compounds were less than 0.10 %. These included: naphthalene, 1-methyl- and naphthalene, 2-methyl- at 6.25 and 6.23 % peak areas respectively; 1,7-dimethyl-3-phenyltricyclo[4.1.0.0(2,7)]hept-3-ene and 1,1'-biphenyl, 2,2',5,5'-tetramethyl- at 5.55 and 5.50 % respectively. 1H-Indene, 1-methylene- and phenanthrene, 2-methyl-

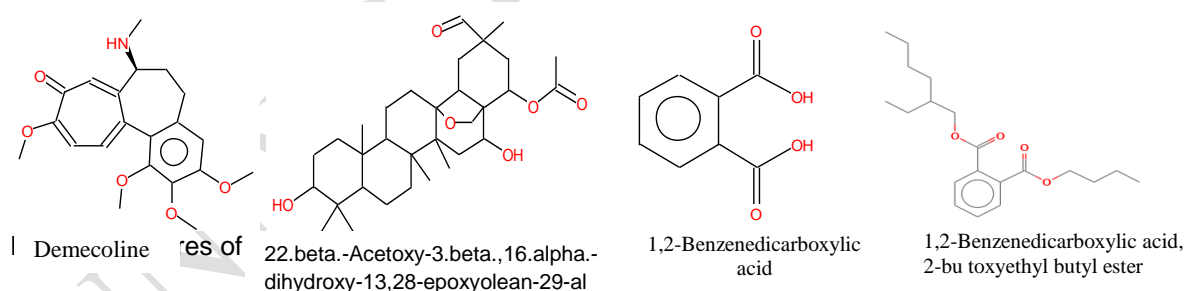
had peak areas of 3.46 and 3.38 % peak area respectively. Hence the difference in the concentration of these compounds mentioned did not exceed 0.10 %.

Compounds with very high molecular weights were identified in the extracts of SMW as shown in table 1. A compound, 22.beta.-Acetoxy-3.beta., 16.alpha.-dihydroxy-13, 28-epoxyolean-29-al, had the highest molecular weight of 530.74 g/mol and molecular formula of $C_{32}H_{50}O_6$. The second heaviest compound was 17-Pentatriacontene with molecular formula, $C_{35}H_{70}$ and weight of 490.93 g/mol and the third was nonacos-1-ene, with a molecular weight of 406.93 g/mol and formula, $C_{29}H_{58}$. The compound with the least molecular weight of 222.37 g/mol, in DMW soil oil extract was 2(1H)-Naphthalenone, octahydro-4a, 5-dimethyl-3-(1-methylethyl)-, (3.alpha., 4a.beta.,5.beta.,8a.alpha.), $C_{15}H_{26}O$.

From Table 2, showing compounds identified in extracts of DMW, the compound with the highest molecular weight of 504.89 g/mol was 9-Octadecenoic acid (Z)-, 9-hexadecenyl ester, (Z)-, $C_{34}H_{64}O_2$, seconded by Hexasiloxane, tetradecamethyl with molecular formula of $C_{14}H_{42}O_5Si_6$ and weight, 458.99 g/mol; and then 1,3-benzenedicarboxylic acid, 5-[[[4-(bromophenyl)sulfonyl]oxy]-, dimethyl ester with a molecular weight of 428.97 g/mol and formula, $C_{16}H_{13}BrSO_7$. Meanwhile, 2-Tetradecanol, $C_{14}H_{30}O$, had the least molecular weight of 214.39 g/mol.

Table 3 shows the compounds found in the extracts of SCO. From the table, 7-Fluoro-3, 4-dihydro-3-[4-[trifluoromethyl]phenyl]-1,9(2H, 10H)-acridinedione, a halogenated compound, $C_{20}H_{13}F_4NO_2$, had the highest molecular weight of 375.31 g/mol. This was seconded by Hexadecanoic acid, methyl ester with molecular weight of 270.45 g/mol and formula, $C_{17}H_{34}O_2$; and then 7-Methyl-octadecane, $C_{19}H_{40}$ with molecular weight of 268.52 g/mol. 1H-Indene, 1-methylene- and [4.2.2]propella-2,4,7,9-tetraene both had the least molecular weight of 128.17 g/mol and formula $C_{10}H_8$.

In all the samples studied, the compound with the highest molecular weight of 530.74 g/mol was 22.beta.-Acetoxy-3.beta.,16.alpha.-dihydroxy-13,28-epoxyolean-29-al, found in the extracts of SMW, followed by 9-Octadecenoic acid (Z)-, 9-hexadecenyl ester, (Z)- with a molecular weight of 504.89 g/mol from the extracts of DMW oil and then, by 17-Pentatriacontene, $C_{35}H_{70}$, with a molecular weight of 490.93 g/mol, found in SMW extracts. On the other hand, 1H-Indene, 1-methylene- and [4.2.2]Propella-2,4,7,9-tetraene with the same molecular formulas of $C_{10}H_8$ had the least molecular weight of 128.17 g/mol from the extract of SCO.



The extracts studied showed that for the extracts of SCO had: about 85.71 % aromatic compounds, 4.67 % alkanolic compound; 4.67 % alkane and 4.67 % alkene. For DMW, the extracts had about 15.38 % aromatic compounds; 38.46 % alkene; 7.69 % alkane; 23.07 % alkanooates; 7.69 % silaxane compound and 7.69 % alkanol.

However, the extracts of SMW had about 42.86 % aromatic compounds; 28.57 % alkene, 14.29 % alkanal and 14.29 % alkanooate

The compounds found in oil extracts of this study showed a range of C_{15} - C_{35} compounds for DMW; C_{13} - C_{34} compound for SMW and C_{10} - C_{20} compounds for SCO. What is the implication of these? However, Ogunbaya *et al.* [17] reported that their spent oil extract from their mechanic workshop soil had C_{21} - C_{25} carbon compounds. More so, the hydrocarbon from the different extracts revealed that: SMW had only two hydrocarbon constituents 17-Pentatriacontene, $C_{35}H_{70}$ and Nonacos-1-ene, $C_{29}H_{58}$ while DMW contained 4 hydrocarbon compounds: 2,6,10,14-Tetramethyl-7-(3-methylpent-4-

enylidene) pentadecane, $C_{25}H_{48}$; 9-Tricosene, (Z)-, $C_{23}H_{46}$; Cyclotetracosane, $C_{24}H_{48}$; 1-Tricosene, $C_{23}H_{46}$; whereas, SCO had three hydrocarbon compound namely: 7-Methyl-octadecane, $C_{19}H_{40}$, 1,5,6,7-Tetramethyl-3-phenylbicyclo[3.2.0]hepta-2,6-diene, $C_{17}H_{20}$; 1 and 1'-Biphenyl, 2,2',5,5'-tetramethyl-, $C_{16}H_{18}$. From all indications, it could also be added that DMW had more hydrocarbon than SMW and SCO. It could be that degradation of the spent oil in the soil had set in, leading to more hydrocarbon in DMW. Fig. 5 shows some hydrocarbons identified in the samples studied. Cyclotetracosane, found in the extract of DMW was the only cyclohydrocarbon identified in this study. The longest hydrocarbon chain identified was 17- pentatricotene, $C_{35}H_{70}$, seconded by nonacos-1-ene, $C_{29}H_{58}$ and then by 1- tricosane and 9-tricosane, $C_{23}H_{46}$. However, 1,5,6,7,tetramethyl-3-phenylbicyclo[3.2.0]hepta-2,6-diene , $C_{17}H_{20}$, was the only multiple branched hydrocarbon chain detected while 7-Methyl-octadecane, $C_{19}H_{40}$ and cyclotetracosane, $C_{24}H_{48}$, were the alkane compounds found in this study. Though there were about eight alkene compounds found, only five oxygenated compounds were identified as shown in Fig 6.

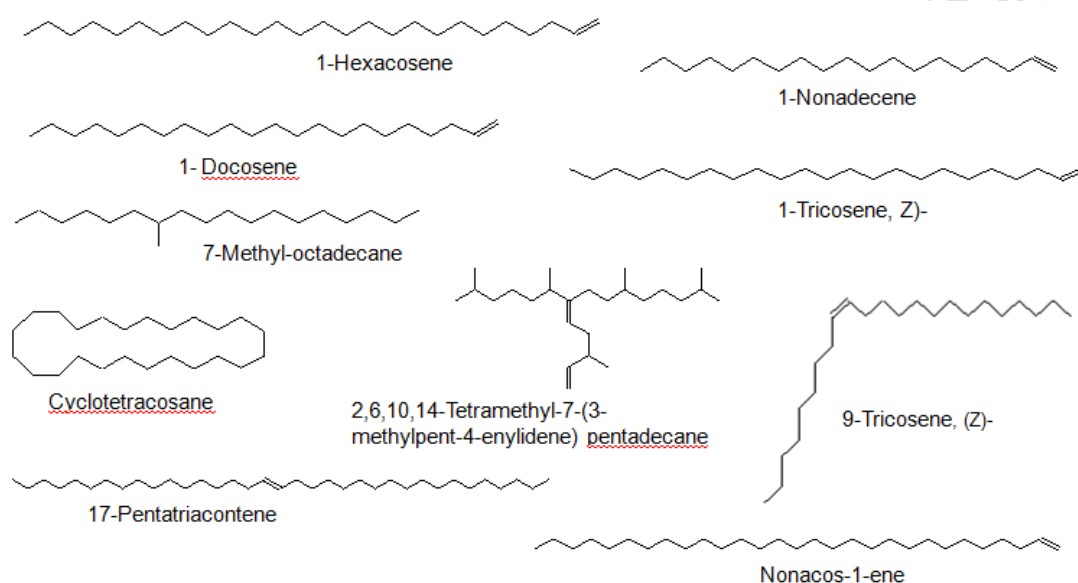


Fig 5: Some Hydrocarbons identified in the samples studied

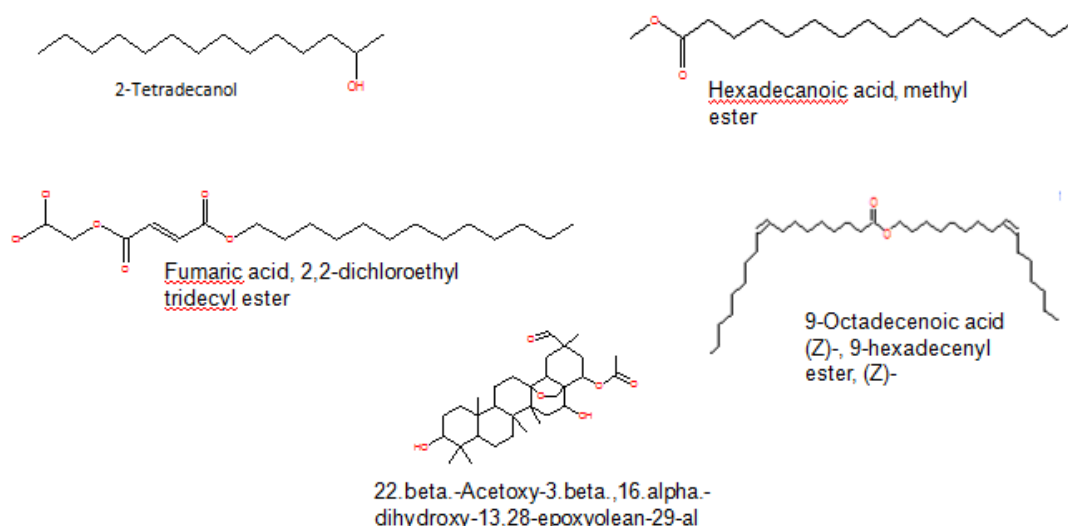


Fig 6: Some oxygenated compounds identified in the samples studied

About eleven (11) monocyclic aromatic compounds were identified in this study as shown in fig 7. Five of these compounds contain at least two oxygen atoms.

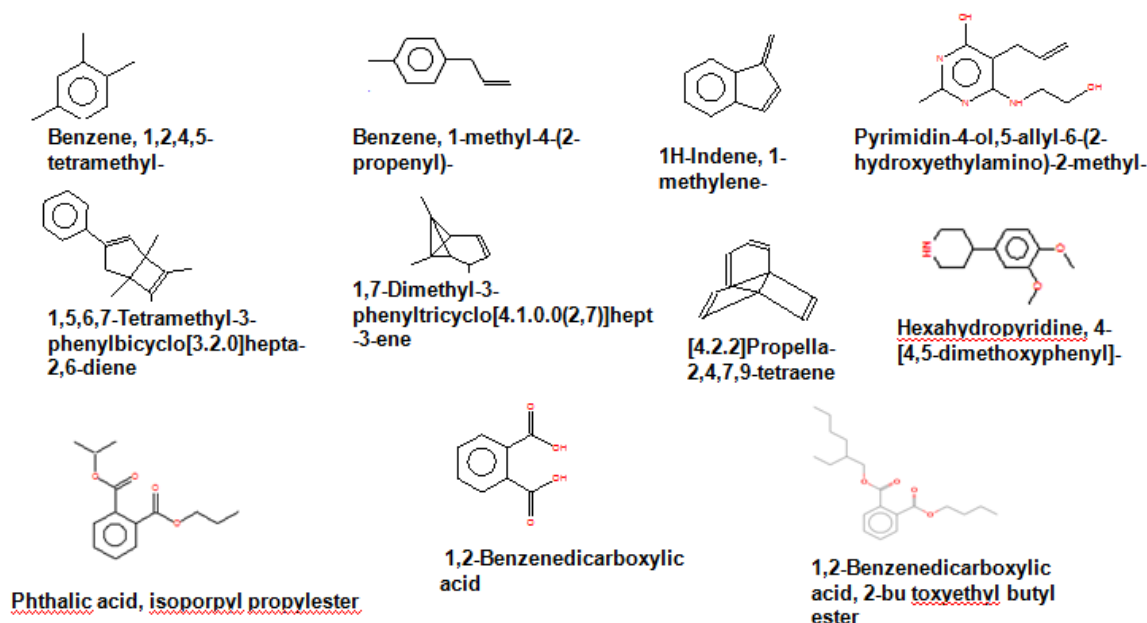


Fig 7: Some mono aromatic compounds identified in the samples studied

Thirteen **Polycyclic Aromatic Hydrocarbons** (PAHs) compounds were identified in SMW, DMW and SCO as shown in fig 8. Borah and Yadav [18] explained that PAHs have low water solubility and their presence in petroleum products could hinder the degradation of these products due to low or no accessibility of hydrocarbon degraders. A carcinogenic and polycyclic compound named 2-Amino-5-isopropyl-8-methyl-1-azulenecarbonitrile- $C_{15}H_{16}N_2$, was detected in the extract of SCO, with area of 16.70 %. Peng *et al.* [19] disclosed that 2-Amino-5-isopropyl-8-methyl-1-azulenecarbonitrile was among the most suitable volatile organic compounds (VOCs) for distinguishing between healthy subjects and patients suffering from a specific cancer. They reported that it was a common VOCs for colon cancer, breast cancer, prostate cancer and detection of lung, breast, colorectal, and prostate

cancers from exhaled breath using a single array of nanosensors. There was no phenol found in this study which also agrees with the study of Kupareva *et al.* [20] except for 2-tetradecanol, an alkanol. An increase in the concentration of both long-chain hydrocarbons, monocyclic and polycyclic aromatic compounds could result to bio-accumulation across the food chain and lead to health hazards and possibly death.

The presence of these compounds in contaminated soils could also slow down bio-degradation of the oil contaminants in the soil by the inherent micro-organisms in the soil. Apart from the reduction in the rate of bio-degradation, the polyaromatic hydro-carbons could poison the soil microbes, leading to their suffocation and death. After rainfall, the spent oil in contaminated soil could be washed into water bodies, thus, causing pollution. Therefore, these compounds in spent oil, which are carcinogenic in nature, could endanger the aquatic organisms and human beings when the contaminated water and aquatic organisms are consumed.

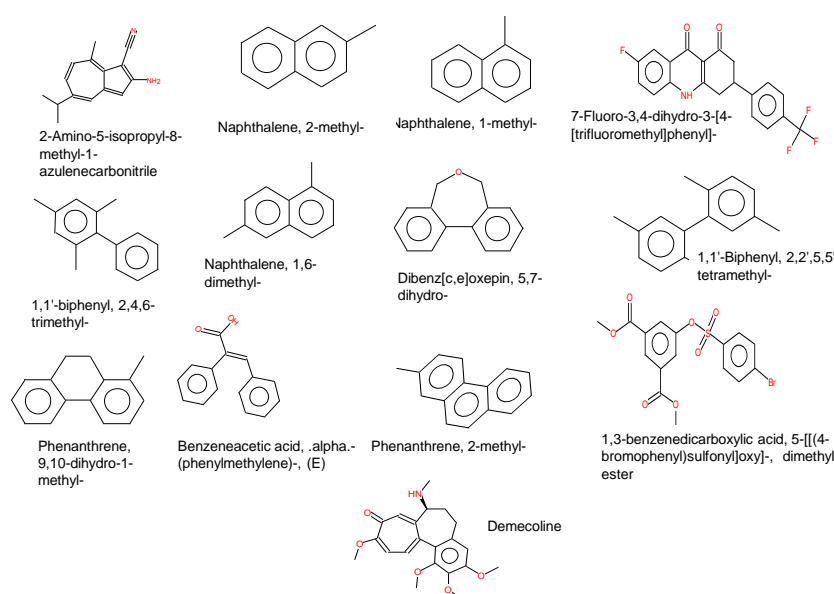


Fig 8: Some Poly-aromatic compounds identified in the extracts of the samples studied

Conclusion

The present work reveals the presence of long chain hydrocarbons, mono-aromatics and polycyclic aromatic compounds. The compounds found in oil extracts of this study showed a range of C_{15} – C_{35} compounds for SMW and C_{10} – C_{20} compounds for SCO. The aromatic composition of the extracts is in the order of SCO > SMW > DMW while the hydrocarbon content is in the order of DMW > SMW > SCO. About eleven monocyclic and thirteen polycyclic aromatic compounds were identified in the study. The most abundance compound was 1,2-Benzenedicarboxylic acid, 2-butoxyethyl butyl ester with peak area of 45.86 %, followed by 1-Docosene with area of 22.36 % and then 2-Amino-5-isopropyl-8-methyl-1-azulenecarbonitrile with area of 16.70 %. The bio-accumulation of these compounds in soil and across food chain could result to health hazards and possibly death. Consequently, the auto mechanic engineers should be educated on the presence of these toxic compounds inherent in the spent oil from cars. More so, the government should come up with policies on spent motor oil collection, possible management and treatment before disposal. Among all, strict enforcement and complete adherence of the made polices by the law enforcement agencies and the general public respectively should be ensured.

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Table-1 Compounds found in oil extract from SMW

S/NO	Compound Name	Molecular formula	Molecular wt (g/mol)	Area %
	1,2-Benzenedicarboxylic acid, 2-butoxyethyl butyl ester	$C_{20}H_{30}O_4$	334.45	45.86
	2(1H)-Naphthalenone, octahydro-4a, 5-dimethyl-3-(1-methylethyl)-, (3.alpha., 4a.beta.,5.beta.,8a.alpha.)	$C_{15}H_{26}O$	222.37	9.12
	17-Pentatriacontene	$C_{35}H_{70}$	490.93	6.01
	6-Octadecenoic acid, methyl ester, (Z)-	$C_{19}H_{36}O_2$	296.49	5.48
	Nonacos-1-ene	$C_{29}H_{58}$	406.77	4.52
	Demecolcine	$C_{21}H_{25}NO_5$	371.43	2.35
	22.beta.-Acetoxy-3.beta.,16.alpha.-dihydroxy-13,28-epoxyolean-29-al	$C_{32}H_{50}O_6$	530.74	2.05

Table 2: Compounds found in oil extract from DMW soil

S/NO	Compound Name	Molecular formula	Molecular weight(g/mol)	Area %
	1- Docosene	$C_{22}H_{44}$	308.58	22.36
	1-Hexacosene	$C_{26}H_{52}$	364.69	15.11
	2-Tetradecanol	$C_{14}H_{30}O$	214.39	13.37
	1-Nonadecene	$C_{19}H_{38}$	266.51	9.55
	Hexahydropyridine, 4-[4,5-dimethoxyphenyl]-	$C_{13}H_{19}NO_2$	221.30	8.97
	Cyclotetracosane	$C_{24}H_{48}$	336.64	6.71
	Fumaric acid, 2,2-dichloroethyl tridecyl ester	$C_{19}H_{32}O_4Cl_2$	394.9	5.17
	9-Tricosene, (Z)-	$C_{23}H_{46}$	322.61	4.64

1,3-benzenedicarboxylic acid, 5-[[[(4-bromophenyl)sulfonyl]oxy]-, dimethyl ester	$C_{16}H_{13}BrSO_7$	428.97	4.28
Hexasiloxane, tetradecamethyl-	$C_{14}H_{42}O_5Si_6$	458.99	4.15
9-Octadecenoic acid (Z)-, 9-hexadecenyl ester, (Z)-	$C_{34}H_{64}O_2$	504.89	3.80
2,6,10,14-Tetramethyl-7-(3-methylpent-4-enylidene) pentadecane	$C_{25}H_{48}$	348.65	1.78
1-Tricosene	$C_{23}H_{46}$	322.61	0.10

Table 3: Compounds found in SCO

S/NO	Compound Name	Molecular formula	Molecular weight (g/mol)	Area %
	2-Amino-5-isopropyl-8-methyl-1-azulenecarbonitrile	$C_{15}H_{16}N_2$	224.30	16.70
	1,5,6,7-Tetramethyl-3-phenylbicyclo[3.2.0]hepta-2,6-diene	$C_{17}H_{20}$	224.34	12.21
	Naphthalene, 1-methyl-	$C_{11}H_{10}$	142.20	6.25
	Naphthalene, 2-methyl-	$C_{11}H_{10}$	142.20	6.23
	Phthalic acid, isopropyl propylester	$C_{16}H_{18}O_4$	250.14	6.04
	1,7-Dimethyl-3-phenyltricyclo[4.1.0.0(2,7)]hept-3-ene	$C_{15}H_{16}$	196.29	5.55
	1,1'-Biphenyl, 2,2',5,5'-tetramethyl-	$C_{16}H_{18}$	210.31	5.50
	Naphthalene, 1,6-dimethyl-	$C_{12}H_{12}$	156.22	5.00
	Dibenz[c,e]oxepin, 5,7-dihydro-	$C_{14}H_{12}O$	196.25	4.75
	Benzeneacetic acid, .alpha.-(phenylmethylene)-, (E)	$C_{15}H_{12}O_2$	224.25	4.39
	Phenanthrene, 9,10-dihydro-1-methyl-	$C_{15}H_{14}$	194.27	3.56
	1H-Indene, 1-methylene-	$C_{10}H_8$	128.17	3.46
	Phenanthrene, 2-methyl-	$C_{15}H_{12}$	192.26	3.38
	7-Fluoro-3,4-dihydro-3-[4-(trifluoromethyl)phenyl]-1,9(2H,10H)-acridinedione	$C_{20}H_{13}FNO_2$	375.31	3.07
	[4.2.2]Propella-2,4,7,9-tetraene	$C_{10}H_8$	128.17	2.47
	1,1'-biphenyl, 2,4,6-trimethyl-	$C_{15}H_{16}$	196	2.32
	Hexadecanoic acid, methyl ester	$C_{17}H_{34}O_2$	270.45	2.22
	7-Methyl-octadecane	$C_{19}H_{40}$	268.52	1.96
	Benzene, 1-methyl-4-(2-propenyl)-	$C_{10}H_{12}$	132.20	1.86
	Benzene, 1,2,4,5-tetramethyl-	$C_{10}H_{14}$	134.22	1.67
	Pyrimidin-4-ol,5-allyl-6-(2-hydroxyethylamino)-2-methyl-	$C_{10}H_{15}N_3O_2$	209	1.41

Abundance

TIC: M1 nna oji h.D\data.ms

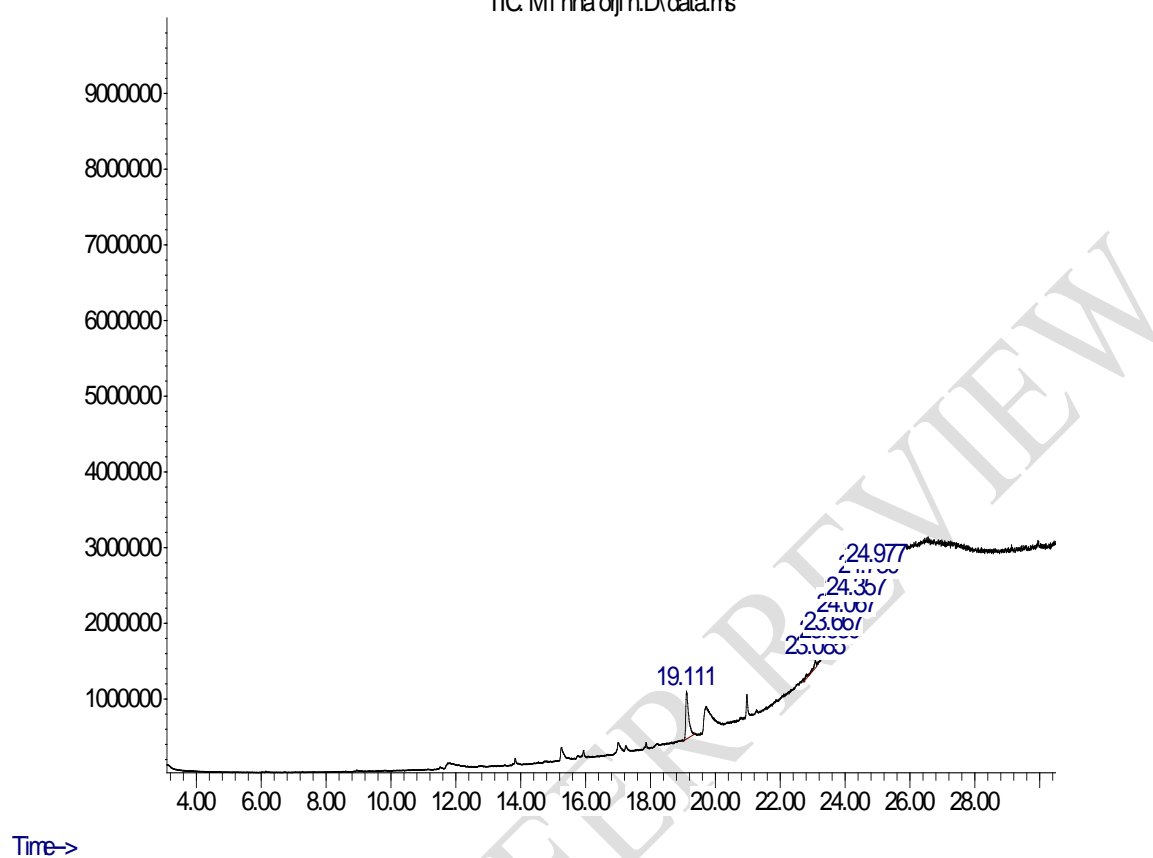
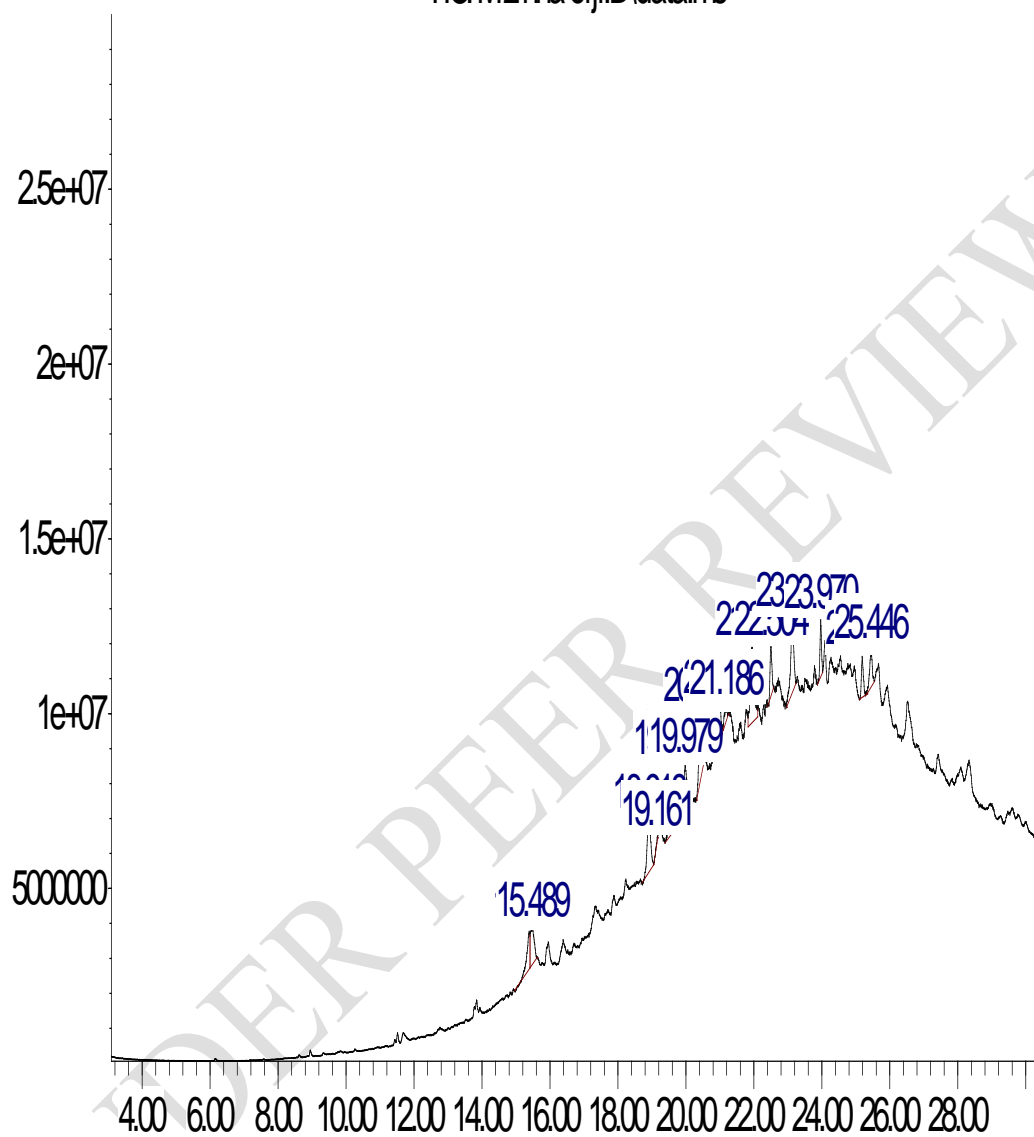


Fig. 2: Chromatogram for oil extracted from SMW soil.

Abundance

TIC: M2ma orji.D\data.ms



Time→

Fig 3: Chromatogram for residual oil extracted from DMW

Abundance

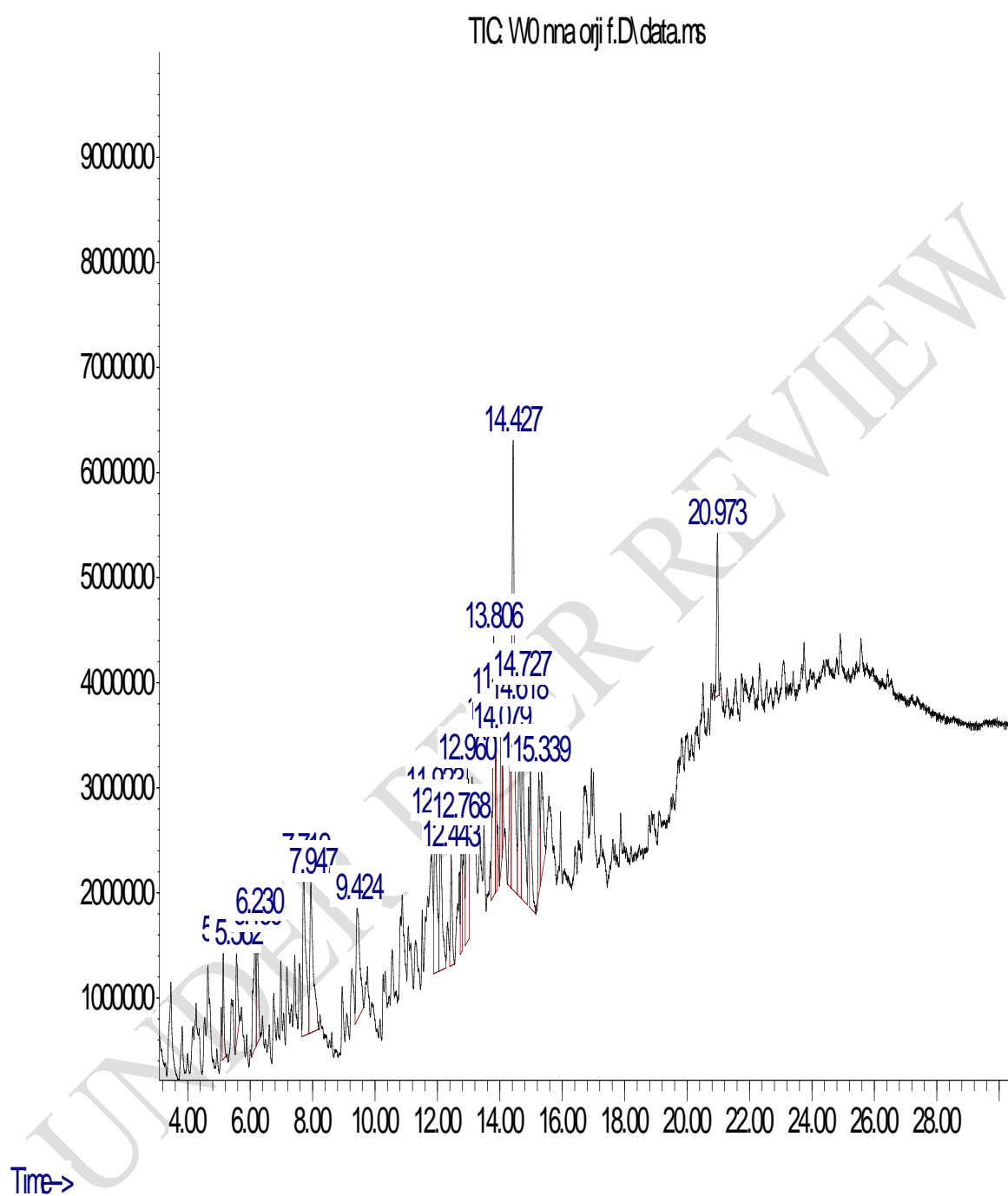


Fig 4: Chromatogram of extract from SCO