Global Scientific JOURNALS

GSJ: Volume 9, Issue 10, October 2021, Online: ISSN 2320-9186

www.globalscientificjournal.com

WEATHERING OF COMMONLY USED AUTOMOBILE LUBRICATING OIL IN NIGERIA.

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ABSTRACT: The weathering of lubricating oil samples was investigated by monitoring the changes in the physico-chemical parameters and microbial counts over a period of 28 days. There was a decline in the TPH from 974.0300ppm to 802.345ppm for the engine oil, 939.090ppm to 819.936ppm for the gear oil and 1043.060ppm to 877.733 for the spent oil. A decrease in the pH of the samples was observed. The viable bacterial and fungal counts (TVC) indicated higher Total Heterotrophic Bacterial (THB) counts than total fungal (TF) counts. Characterization and identification tests revealed that a bacterial consortium comprising of the following genera; *Bacillus, Pseudomonas, Proteus, Escherichia, Micrococcus, Arthrobacter, Enterobacter* and *Citrobacter* were encountered in the oil samples, with the engine oil sample showing the greatest increase in total THB counts. Fungal genera encountered included *Aspergillus, Cladosporium, Penicillium, Fusarium,* and *Mucor.* Amongst the bacterial isolates, *Pseudomonas sp* had the highest frequency of occurrence of 35% and 45% in engine oil and spent oil, respectively. However, *Flavobacterium sp*. had the highest frequency of occurrence, 32% in the gear oil. The results of the study revealed that engine oil showed the greatest degree of weathering while the spent oil was the least susceptible to weathering.

Key words: Weathering, Lubricating Oils, Engine oil, Gear oil, TPH, TOC, Alkalinity, pH, Viscosity, TFC, THBC.

1. INTRODUCTION

With increase in the world's population, there has been a corresponding increase in the demand for petroleum products which can be in the form of fuel, oils and other products used in different capacities to meet the needs of the growing population. Industrialization has brought forth different machineries and automobiles which require diverse petroleum products for functionality. Petroleum-based products are a major source of energy for industry and daily life (Kvenvolden and Cooper, 2003). Petroleum lubricating oils are hydrocarbon compounds, containing combinations of hydrogen and carbon with various molecular forms. They are derived from naturally occurring petroleum deposits and are applied between contact surfaces to reduce friction between moving parts of machine systems. These oils are used in the lubrication of various internal combustion engines which include road vehicles such as cars and motorcycles, heavier vehicles, etc. While the main function is to lubricate moving parts, motor oil also cleans, inhibits corrosion, improves sealing, and cools the engine by carrying heat away from the

moving parts (Klamann, 1984). Although these set of products may seem so valuable and indispensable, they can get to the environment and cause pollution of land and water. When crude oil or petroleum products are released to the environment, they are immediately subject to a wide variety of changes in physical and chemical properties known as "weathering".

In the short term after a spill, evaporation is the single most important and dominant weathering process. (Antwi-Akomeah, 2011). For the lighter petroleum products particularly, evaporation has a great effect on the amount of oil remaining on water or land (Zhendi *et al.,* 2005). Biological weathering involves the breakdown of petroleum hydrocarbon compounds by living organisms into simpler compound compounds which could be recycled back into the nutrient pool through mineralization (Odokuma and Dickson, 2003). Due to the poor biodegradation rates observable for mineral oils, however, lubricating oils may persist in the environment and have the potential to cause harm to both man and his environment. (Balba *et al.,* 1988; Odokuma and Dickson, 2003)

Thousands of litres of waste lubricating oil are generated daily from mechanical workshops and are discharged carelessly into the environment (Adegoroye, 1997; Adelowo *et al.,* 2006). United States Environmental Protection Agency (USEPA), 1996, stated that only one litre of such waste lubricant is enough to contaminate one million gallons of freshwater. Waste lubricants are discharged into the environment, causing contamination of land and water (Holliger *et al.,* 1997; Nilanjana & Preethy, 2010).

Environmental contamination caused by these oils is a threat to humans, animals, and vegetation leading to significant public health and socio-economic hazard (Okerentugba and Ezeronye, 2003; Edewor *et al.,* 2004). This study therefore investigates the weathering (Physical, chemical and biological breakdown) of these oils in the environment.

AIM OF STUDY

This study is aimed at investigating the weathering of selected automobile lubricating oils.

1.2 OBJECTIVES OF STUDY

- To monitor the changes in microbial counts of organisms isolated from both spent and unused oil samples during weathering.
- To observe the changes in some physicochemical properties of automobile lubricating oils over a period of 28 days.
- To determine the microbial diversity of the microbial population in the lubricating oil samples.
- To compare the weathering of unused to used lubricating oil exposed to natural weathering processes.

2. Materials and methods

2.1 Collection of oil samples

Three different lubricating fluid samples were used for this study.

- Crown type A economy automatic transmission fluid (gear oil)
- Forte heavy duty motor oil (engine oil)

- Spent (used) oil from a mechanical workshop

The different lubricating fluid types and spent lubricating fluid were got from retail shops and from a mechanics workshop located near the University of Portharcourt, all in Rivers State, Nigeria. The Crown type A economy automatic transmission fluid was manufactured in the USA while the Forte heavy duty motor oil was manufactured in Nigeria. The samples were exposed to the environment (sunshine, air, and sunlight) in sterile containers for a period of 28 days.

2.2 ENUMERATION OF TOTAL BACTERIAL COUNTS.

The method used was adapted from APHA (2015). Bacterial enumeration was carried out on nutrient agar plates. One milliliter of the oil sample was first diluted by a 10-fold serial dilution (up to 10^{-6} dilution) using normal saline as a diluent. Appropriate dilutions (0.1ml) of oil samples were inoculated into their respective plates in duplicates using the spread plate method (APHA, 1998). The inoculums were gently and generously spread using a sterile bent glass rod. A control plate was prepared alongside, but without inoculation. The plates were incubated in an inverted position in the incubator at 35^0C for 48 hours. After incubation, the bacterial colonies formed were counted and the colony forming units per milliliter (cfu/ml) of the samples were calculated. Colonies were randomly picked and purified by sub-culturing onto nutrient agar plates using the streak plate method. The isolated colonies which were obtained from the sub-cultured plates were transferred onto nutrient agar slants in labeled bijou bottles and stored as stock cultures for further tests. For fungi isolation, the same procedure carried out for the bacterial isolation above was employed, however, modified Potato Dextrose Agar (PDA) medium (containing lactic acid in the ratio of 1000:1) was used in place of the nutrient agar medium. The plates were then incubated for about 3-5 days at room temperature. After incubation, a little portion of each fungal colony was picked using sterile forceps and aseptically sub-cultured onto fresh PDA plates and the developed sub-cultured colonies were used for fungi identification tests.

2.3 CHARACTERIZATION AND IDENTIFICATION OF THE ISOLATES

Identification of bacterial and fungal hydrocarbons utilized were accomplished on the basis of their cultural morphological characteristics and by Gram staining. The isolates were further subjected to a series of biochemical tests for identification and using the determination scheme of Holt *et al.;* (1994). Similarly, moulds were identified through their cultural and microscopic features.

2.4 Physicochemical analysis

Physicochemical parameters of the samples analyzed were pH, alkalinity, TPH, Viscosity, and TOC and were determined using methods adopted from Jamie and Richard (1996).

3.0 Results

There was a decrease in the TOC with time in all samples as presented in (Figure 1). Spent oil exhibited the highest organic carbon content of 10.59% as opposed to engine oil with 2.13% after 28 days. The percentage change in TOC of the samples revealed that 9.23% remained in the engine oil after 28days and thus exhibited the greatest change over the period of analysis. Changes in pH of the different oil samples analyzed during a period of 28 days at 7 days' intervals are represented in Figure 1. The results revealed that there was a decline in the pH from 5.90 to 4.80 for the spent oil, 8.99 to 6.57 for the gear oil, and 7.85 to 5.65 for the engine oil. The changes in viscosity of the samples are presented in Figure 3. with engine oil showing the lowest viscosity of 10.20cst as opposed to spent oil which had the highest viscosity of 13.43cst after 28 days. The alkalinity of the different oil samples over the same period are represented in Figure 4: which revealed that there was a decline in the alkalinity from 19.92 mg/KOH/g to 12.60 mg/KOH/g for the spent oil, 26.65 mg/KOH/g to 10.20 mg/KOH/g for the gear oil and 26.85 mg/KOH/g to 7.65 mg/KOH/g for the engine oil.
Table 1 shows the changes in the sum of the Total Petroleum Hydrocarbons in the samples and showed the different Petroleum Hydrocarbons and their amounts in the samples. The results revealed that there was a decline in the TPH from 974.03ppm to 819.937 for the engine oil, 939.09ppm to 819.937ppm for the gear oil and 1043.06ppm to 877.733ppm for the spent oil.

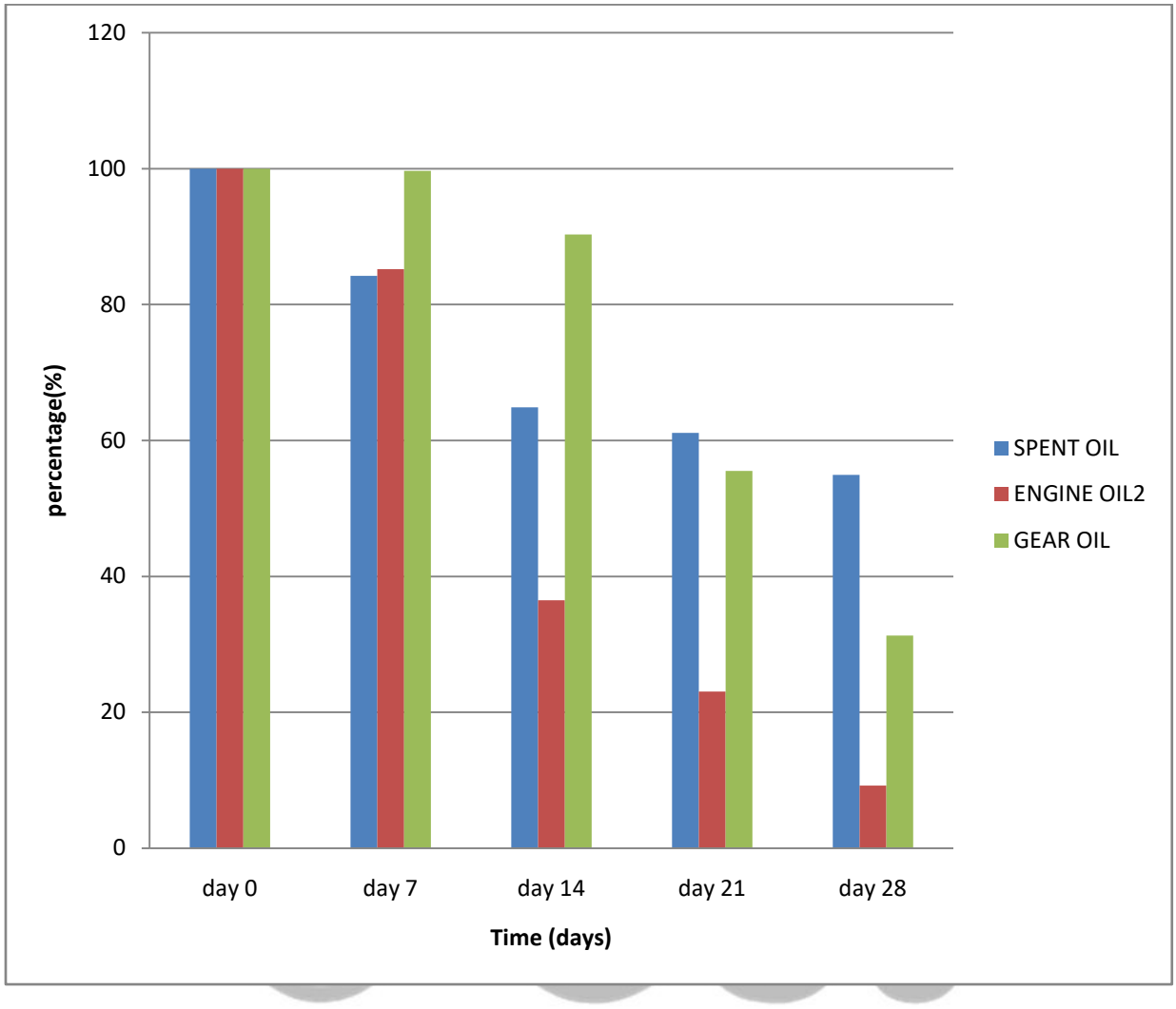

Figure 1: Percentage change in the TOC of the samples.

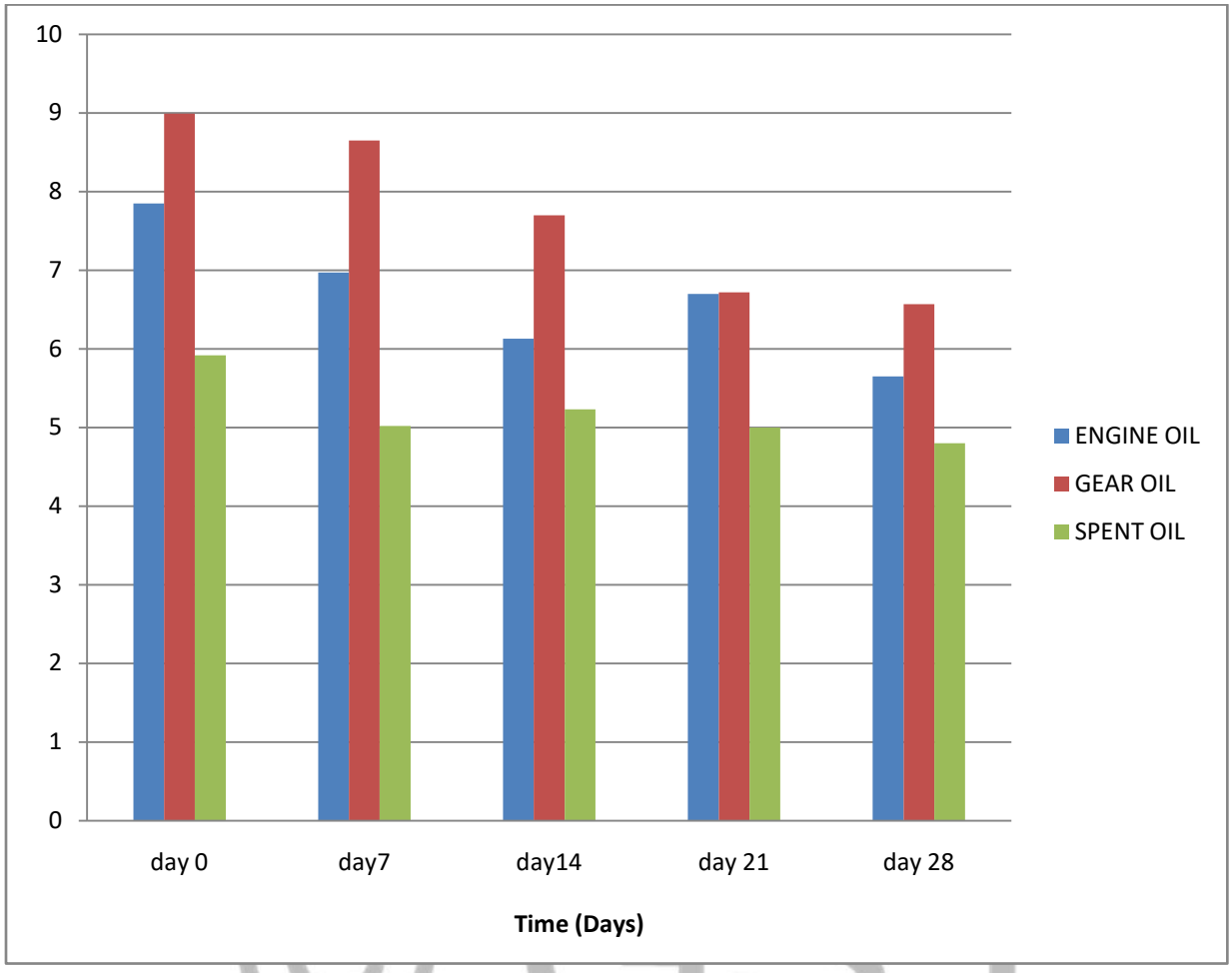

Figure 2: pH of the samples

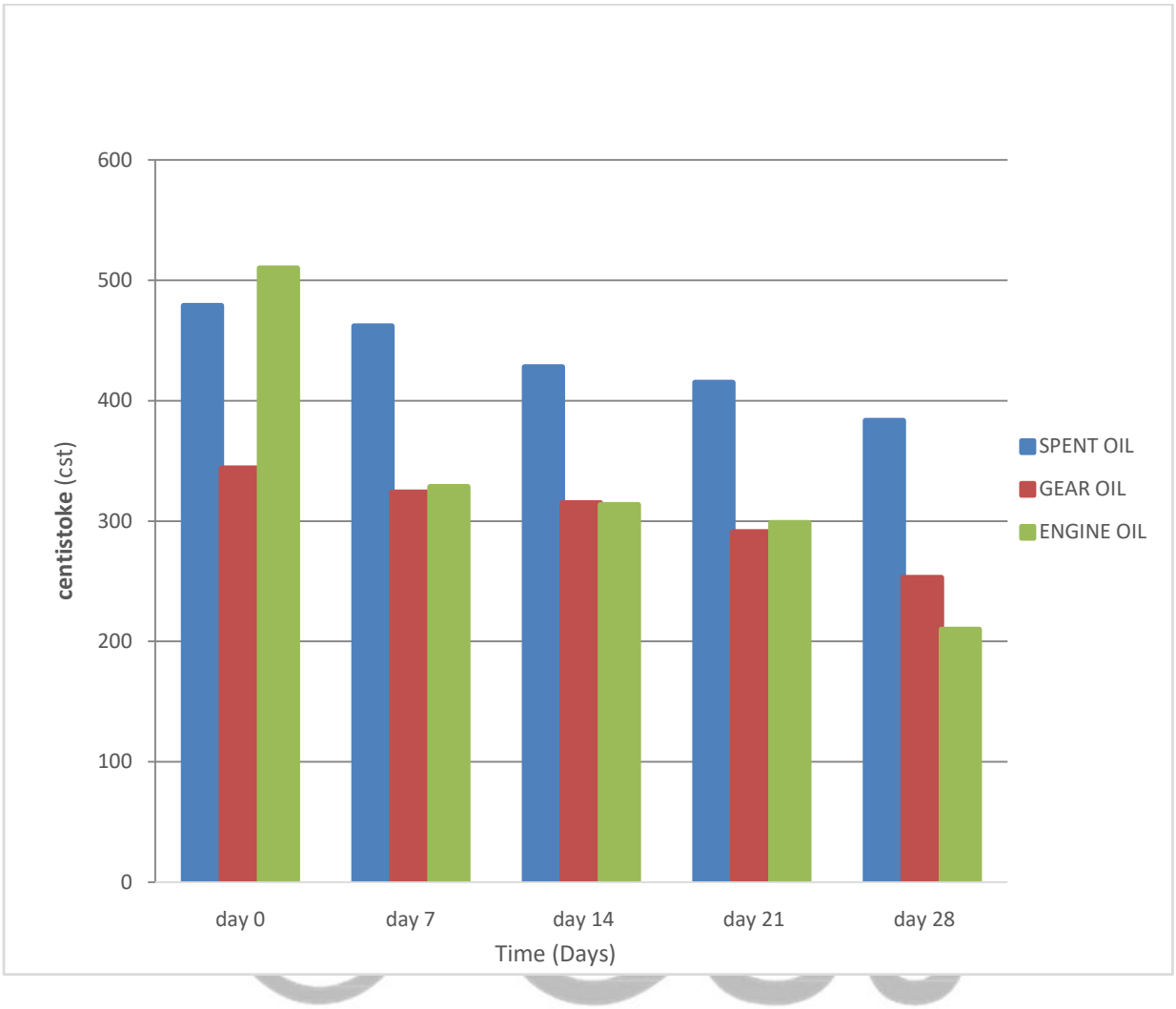

Figure 3: Viscosity of different oil samples

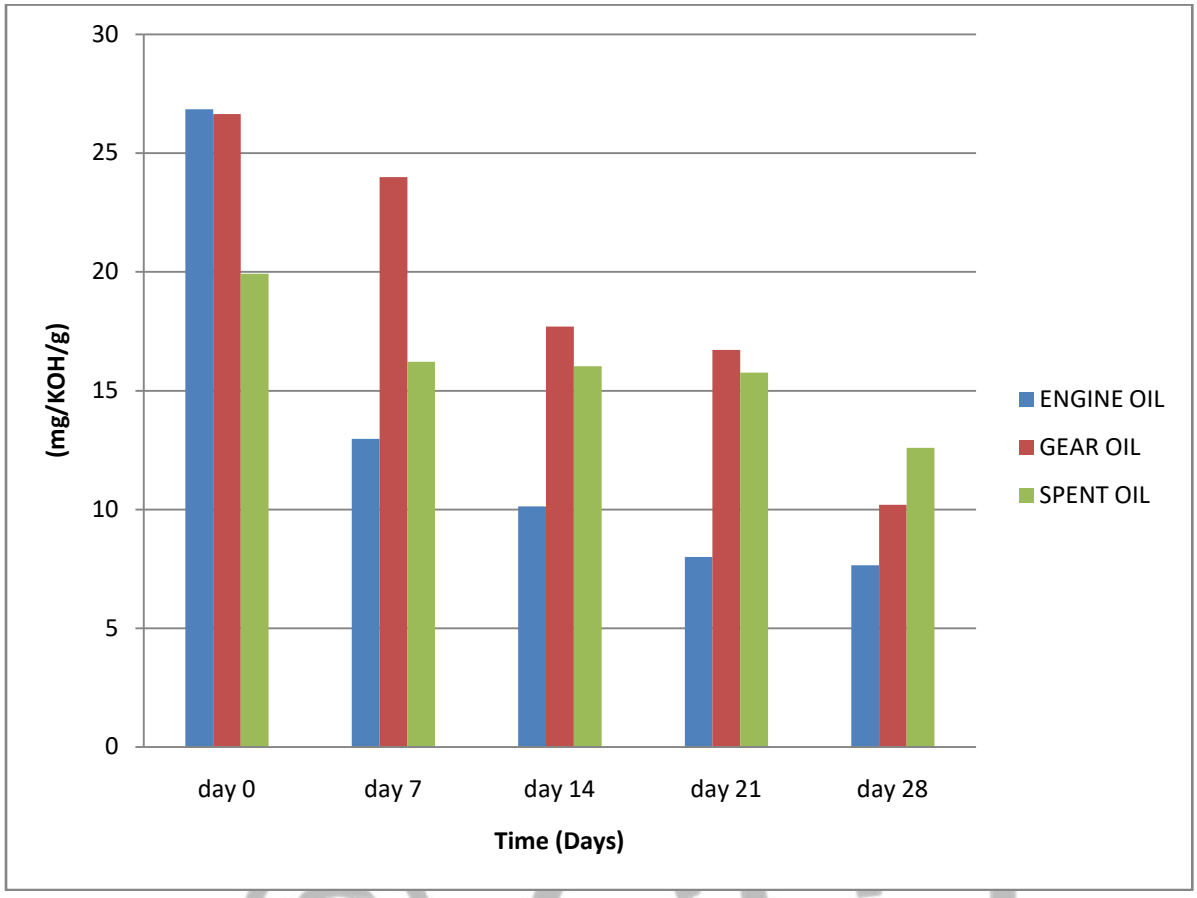

Figure 4: Alkalinity of the samples.

Table 1: Total Petroleum Hydrocarbons in the different samples

Sample	Day 0	Day 28	Percentage decrease in TPH
Engine oil	974.031**(ppm)**	802.345**(ppm)**	17.63%
Gear oil	939.090**(ppm)**	819.937**(ppm)**	12.69%
Spent oil	1043.06**(ppm)**	877.733**(ppm)**	15.85%

Table 2: Different petroleum hydrocarbons in the lubricating oil and amount (ppm) at 0 days

s/N	**Hydrocarbon**	**Engine oil**	**Gear oil**	**Spent oil**
1	n-octane	22.69528	-	18.13192
2	n-nonane	-	-	-
3	n-decane	-	-	-
4	n-undecane	-	-	-
5	n-dodecane	-	-	-
6	n-tridecane	-	-	-
7	n-tetradecane	-	-	-
8	n-pentadecane	-	-	-
9	n-hexadecane	16.79368	9.09194	-
10	n-heptadecane	8.48495	4.63408	-
11	n-octadecane	1.25694	1.25694	2.21040
12	n-nonadecane	10.78802	6.93715	11.27116
13	n-Eicosane	11.73585	7.88438	11.83037
14	n-heneicosane	12.11978	8.26891	11.46196
15	n-docosane	14.38116	30.53029	39.33103
16	n-tricosane	15.66920	111.81833	13.35477
17	n-tetracosane	39.60303	165.75216	53.39478
18	n-pentacosane	18.17005	14.31918	18.47486
19	n-hexacosane	17.84220	13.99133	28.15853
20	n-heptacosane	38.31886	34.46799	40.22544
21	n-octacosane	25.90932	22.05845	15.85641
22	n-nonacosane	30.77722	26.92635	27.29091
23	n-triacontane	57.25704	53.40617	56.06660
24	n-hentriacontane	42.99085	39.13998	95.92287
25	n-dotriacontane	31.65975	27.80880	67.20742
26	n-tritriacontane	167.26360	13.41273	154.04759
27	n-tetratriacontane	363.31382	159.46295	378.82638
	TOTAL	**974.031ppm**	**939.090ppm**	**1043.06340ppm**

Table 3: Different petroleum hydrocarbons in the lubricating oil and amount (ppm) after 28 days

s/N	**Hydrocarbon**	**Engine oil**	**Gear oil**	**Spent oil**
1	n-octane	35.81228	-	38.86275
2	n-nonane	27.87094	-	30.92141
3	n-decane	69.93222	-	52.78034
4	n-undecane	49.72987	-	72.98269
5	n-dodecane	17.84516		22.63232
6	n-tridecane	-	-	-
7	n-tetradecane	-	-	-
8	n-pentadecane	-	-	-

9	n-hexadecane	19.58185	41.86441	20.87563
10	n-heptadecane	25.68011	66.53385	14.30506
11	n-octadecane	17.09719	62.26152	28.73058
12	n-nonadecane	53.79112	21.87332	56.84159
13	n-Eicosane	38.70741	110.84737	41.75788
14	n-heneicosane	26.52163	49.70412	29.57210
15	n-docosane	38.70741	51.29524	41.75788
16	n-tricosane	10.03185	113.13981	13.08232
17	n-tetracosane	14.03116	172.47124	17.18143
18	n-pentacosane	51.95371	-	57.79631
19	n-hexacosane	11.93089	-	14.72302
20	n-heptacosane	73.94891	-	76.24104
21	n-octacosane	42.16095	-	44.95308
22	n-nonacosane	35.05271	-	37.84424
23	n-triacontane	28.03327	-	30.82540
24	n-hentriacontane	18.96809	-	21.76022
25	n-dotriacontane	27.65336	-	30.44549
26	n-tritriacontane	39.07122	-	41.86335
27	n-tetratriacontane	36.20357	129.94577	38.99570
	TOTAL	**802.3450ppm**	**819.9367ppm**	**877.73264ppm**

1.0 MICROBIOLOGICAL ANALYSIS

Results for the total heterotrophic bacteria counts showed that there was an increase in bacterial counts in all samples with the engine oil supporting the highest increase in total bacterial count. The THB count increased from 8.5×10^4 cfu/ml to 2.24×10^5 cfu/ml for the engine oil, 7.50×10^4 to 1.82×10^5 for the gear oil and 6.8×10^4 to 1.74×10^5 for spent oil as shown in Figure 5.

The total fungal counts indicated that there was an increase in the fungal counts in all samples with the spent oil showing the greatest increase in fungal counts of 5.00×10^5 cfu/ml during the period of analysis as presented in Figure 6.

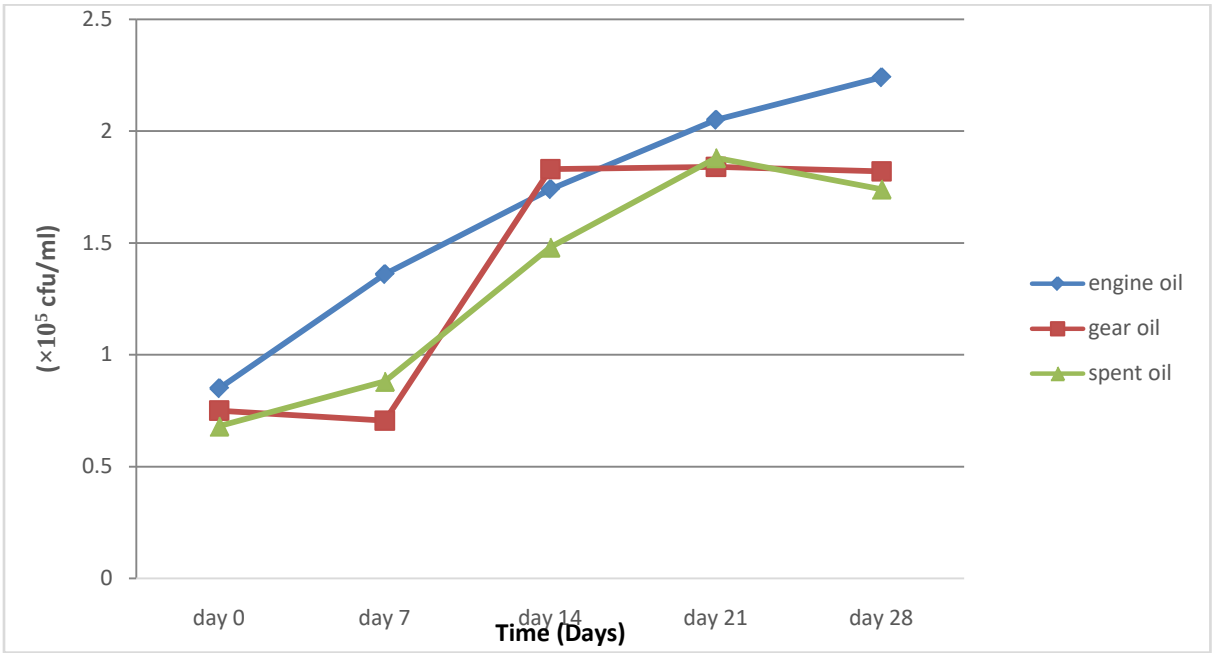

Figure 5: Total heterotrophic bacterial count of the samples

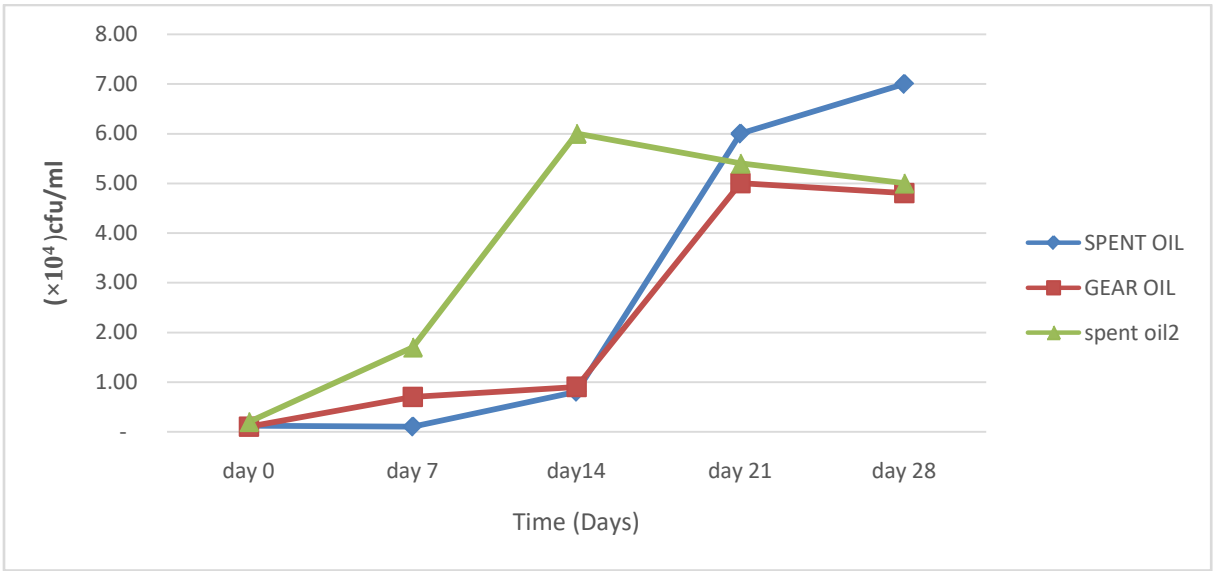

Figure 6: Total fungal count of different oil samples

4.1 Discussion

The results of the physico-chemical parameters analyzed over a period of 28 days and that of the total viable counts showed that engine oil was the most weathered relative to gear oil and spent oil, with gear oil being the least weathered. Microbial count results generated showed an increase in microbial numbers in the samples throughout the study. The physico-chemical parameters; Total Petroleum Hydrocarbons (TPH), pH, temperature, alkalinity, and viscosity measured to

ascertain the progress of the weathering of the oil contaminant proved to be good indicators for the investigation.

During the study, it was observed that the Total Organic Carbon content of the samples as presented in Figure 1 reduced significantly, which indicated that weathering actually occurred. The engine oil, however, showed a considerable reduction in TOC with time than the other samples. The gradual decrease in TOC corresponded with the gradual rise in the microbial population. The slow reduction in TPH, however, indicates that the biodegradation process and other weathering processes like volatilization and adsorption occurred. The importance of pH in the biodegradation of oil contamination as discussed by Dibble and Bartha (1976) suggests that the mineralization of hydrocarbons proceeds most rapidly at pH value between 6.5-8.5 It was observed from Figure 3 that the samples had gradual decrease in pH which could be due to the production of acidic metabolic products by the organisms. Additionally the Total Fungal count which was higher in the spent oil was as a result of relatively low pH.

The increase in the total viable count indicated that hydrocarbon pollution enriched the microbial population (Okpokwasili and Nnorom, 1990). However, a decrease in total viable count between day 21 and day 28 of the analysis indicated that increase in the number of microbes may deplete existing supplies of nutrients and may limit further growth of the microbial populations.

The results obtained from the characterization and identification of bacterial and fungal isolates reveal the following genera from gear oil; *Pseudomonas, Proteus, Citrobacter*, *Bacillus, Enterobacter*, *Norcardia* and *Corynebacterium* Bacillus, *Pseudomonas, Acnetobacter*, and *Flavobacterium,* were encountered in the engine oil and *Proteus, Nocardia*, *Pseudomonas, Corynebacterium, Arthrobacter and Serratia* were encountered in the spent oil. The fungal genera encountered in the gear oil were *Aspergillus, Rhizopus, Penicillium, and Cladosporium. Aspergillus, and Fusarium. Cladosporium, Penicillium,* and *Mucor* were encountered in the engine oil while *Aspergillus, Mucor,* and *Cladosporium* were encountered in the spent oil.

Most of these organisms encountered in this study have been implicated in earlier studies as being able to degrade car engine lubricating oil by Ekwenye and Ike (2007). They include *Bacillus, Pseudomonas, Micrococcus,* and *Citrobacter,* while the fungi include *Aspergillus, Cladosporium, and Penicillium.*

Recommendation: The quality of life on earth is linked inextricably to the quality of the environment. (Jain *et al.*, 2011). Approximately 85% of all lubricants presently being used in the

world are petroleum-based oils. These oils have been found to deteriorate the environment when they are mismanaged; leading to pollution of land and ground and surface water (Okpokwasili and Nnorom, 1990). In the case of used oils, priority should be given to preventive actions with the goal of generating zero used oil. In addition, biolubricants (plant-based lubricants) represent an opportunity to greatly reduce the environmental impact of lubricants and create green companies and jobs. Biolubricants, also known as biolubes and bio-based lubricants, apply to all lubricants that biodegrade rapidly and which are nontoxic for human beings, fauna, flora and aquatic habitats.(UNEP, 2015) The key advantages of biolubricants are rapid biodegradability, low toxicity in the environment, environmental friendliness, good lubricating properties, high viscosity index, longer equipment life, contribution to improved water quality, reduction of greenhouse gases, increase in economic security, better safety, since they have higher flashpoints, constant viscosity, and less oil mist and vapour emissions and reduction of oil dependence. **(**UNEP, 2015).

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