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INVESTIGATION OF SOIL CONTAMINATION IN A LUBRICANT RETAIL MARKET IN IKOKU, PORT HARCOURT, NIGERIA.

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ABSTRACT

The physicochemical properties, total polycyclic aromatic hydrocarbons (PAH) concentration as well as that of lead, zinc, chromium, cadmium and arsenic in soil obtained from a lubricant retail market were determined. The PAHs, lead, chromium and zinc concentrations were found to be higher in comparison to the soil samples obtained from a typical residential area 2 km away. The average values for total PAH concentration in the sample soil were 3.76 /4.70 mg kg^{-1} (topsoil/subsoil). The values for the control (topsoil/subsoil) were 0.189/ 0.286 mg kg^{-1}. The average concentrations of lead, zinc, chromium and cadmium in the sample (top-soil/ sub-soil) were 3.70/ 3.85, 9.7/9.69, 197.29/82.18 and 7.42/3.35 mg kg^{-1} respectively while in the same order, their values in the control gave 0.75/0.94, 8.86/9.40, 103.66/59.50 and 4.08/0.87 mg kg^{-1}. The sample soil also showed an increase in some physicochemical parameters such as Total Organic Carbon (TOC), sulphate and nitrate contents as well conductivity both in the topsoil and the subsoil.

Keywords: Soil contamination; Lubricant retailing market; Polycyclic aromatic hydrocarbons; Heavy metals.

INTRODUCTION

Lubricants are substances placed between the contact surfaces of solid moving parts, for the primary purpose of reducing friction and wear. They also carry out other functions such as removal of heat, corrosion prevention, transfer of power, providing a liquid seal at moving contacts and suspension and removal of wear particles (Dorinson and Ludema, 1985; Boyde, 2002; Nowak *et al*. 2019). Most lubricants are 95% oil-based which may be natural, mineral or synthetic depending on their applications, (Lu and Kaplan, 2008; Stachowiak and Batchelor, 1993); However, a lubricant may be graphite, or any substance- gas, liquid, semisolid, or solid that permits free action of mechanical devices and prevents damage by abrasion and seizing of metal or other components through unequal expansion caused by heat.

The increased use of machinery in various areas of human activities has led to an increase in the demand and production of lubricants and consequently, an increased contribution to environmental pollution. Due to its low cost and a wider range of applications, mineral oil-based lubricants are readily available. Contaminants resulting from the use of lubricants are either inherited from the crude oil feedstock or were components of the lubricant formulations. Lead naphthenates, for example, is employed as an extreme pressure additive while mild corrosion of components of certain machinery components can be the source of metal contaminants such as copper, cadmium etc. (Stachowiak and Batchelor, 1993). Lubricants, therefore, pose similar environmental problems similar to hydrocarbons when substantial quantities are deposited in the biota (Vazquez-Duhalt, 1989; Wang *et al*., 2000; Syahir *et al*., 2017).

Like most products of crude oil, the mineral oil used for the production of lubricating oils contains compounds such as aromatic and polycyclic aromatic hydrocarbons (PAHs), heteroatomic compounds, paraffins, naphthenes and heavy metals which could act as pollutants once their permissible range in an ecosystem is exceeded. Their sources of entry into the environment have been identified as spills, deliberate disposal, leakages at the retail outlets and mishandling of fresh and used products (Nespeca *et al*., 2018). It can also be from the operation of devices and machinery that use lubricants, such as two-stroke engines, open chain saws and agricultural equipment (Betton 2010; Nowak *et al*., 2019). Betton (2010) specifically stated that only 32% of total lubricants produced in Europe is ‘successfully disposed of’ by burning and recycling. He concluded that the greater percentage enters the environment in one way or the other in the course of ‘consumption’.

Fresh and used lubricating oils contain a variety of aliphatic, aromatic and polycyclic aromatic hydrocarbons (PAHs). PAHs are classes of persistent organic pollutants made up of multiple carbon ring structures. These compounds have become a global concern owing to their persistence in the environment and have been listed as priority pollutants by both the US Environmental Protection Agency (EPA) and European Union (EU) due to their carcinogenic and mutagenic properties (Tang, et al, 2005; Aichner *et al*., 2007). Polycyclic aromatic hydrocarbons are perhaps the most common contaminant that has been associated with both used and unused lubricating oils which has been investigated by several researchers (Wang et al 2000; Obini *et al*., 2013; Rengarajan et al., 2015; Ekanem and Ogunjobi 2017; Wu et al., 2017; Mao et al., 2020).

Several studies have profiled the negative impact of lubricating oil in both plants (soil) (Vwioko *et al*., 2006; Agbogidi, 2009; Stephen *et al*., 2013; Ekanem and Ogunjobi, 2017; Hazim and Al-Ani, 2018; Nyarko *et al*., 2019; Nwachukwu *et al*., 2020) and aquatic ecosystems.

Although different strains of bacteria have been identified to appreciably degrade contaminants associated with lubricating oils or the source crude oil (Lopes *et al*., 2010; Ramadan, 2012; Ron and Rosenberg 2014; Raju et al 2017), none is 100% efficient as they only reduce the contaminants without guaranteeing a reduction in toxicity (Saterbak *et al*., 2000). More so, some of the products of biodegradation may yield harmful secondary contaminants that are more eco-toxic than their precursors (Plaza *et al*., 2005; Aruyor and Ori-jesu, 2009). Soroldoni and his co-workers (2019) in particular, reported the extent of toxicity of spent lubricant oil-contaminated soil to *Eisenia andrei* after 22 months of bioremediation.

From the foregoing, it is evident that the most adequate method of protecting the environment from the harmful effects of lubricants is by avoiding or minimising spills, disposals and leakages.

This study aims at investigating the soil contents of a local lubricant retailing market; Ikoku, Port Harcourt, Nigeria, for pollutants and comparing same with a typical residential area. It is believed that the activities in this market have impacted negatively on the surrounding soils and may eventually sip to underground water or nearby farmlands.

MATERIALS AND METHODS

The Study Area: Ikoku lubricant retail market is one of the commercial establishments in Port Harcourt, Nigeria, and has been in operation for more than 30 years. Although with lock-up shops, several drums of lubricating oils both filled and empty are always stacked in the available open spaces. The marketers make transfer of the liquid contents in the open with attendant spills. Some auto spare parts (both new and fairly used) shops are also located within this vicinity.

The study area lies within longitude 6^0 58’ 30” E through longitude 7^0 1’ 30” E and Latitude 40 43’ 30” N through latitude 40 54’ 30” N (Figure 1).

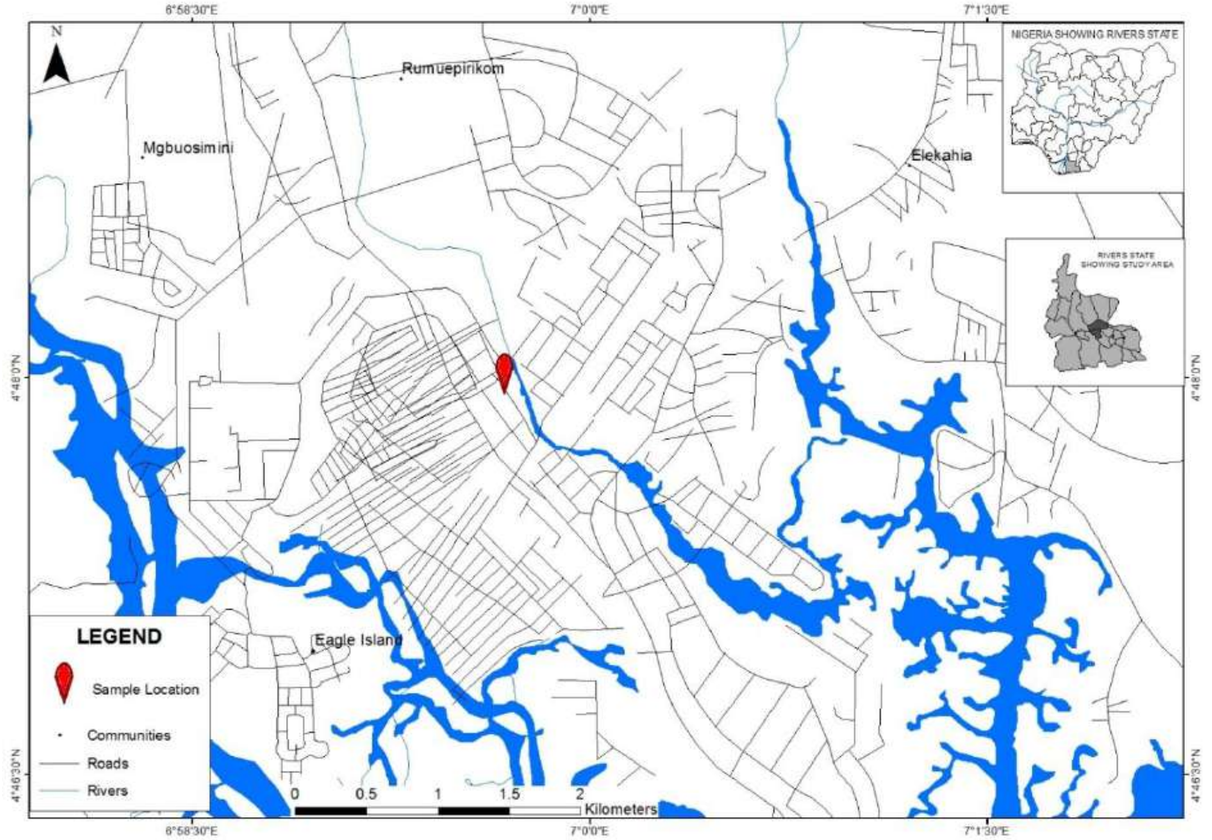

Figure 1: Sample location map

Soil Sampling and Pretreatment

Twelve samples were collected using an improvised auger from the three corners of a virtual triangular mapping of the two sites (six samples from each); the lubricant market and a residential area that is 2 km away. Each corner was analyzed and random points picked. Two composite samples from each site were prepared; one, by mixing the three samples from the surface or topsoil of the three corners and the other by mixing the subsoil (7-15cm depth) of the three corners. The composite samples were collected in polyethylene bags and labelled S_T and S_S representing the topsoil and the subsoil respectively from the lubricant market site and C_T and C_S representing that from the control site. Samples were air-dried by spreading in an open airy space for 3 days with occasional stirring to expose fresh surfaces for proper drying. Before analysis, samples were further demagnetized, ground and sieved.

Instrumentation

Jenniary Digital pH, Hama Instruments Hi 83200 multiparameter bench photometer, GBC sensAA Atomic Absorption Spectrophotometer (AAS) and Agilent Technologies 7890A Gas chromatograph.

Physicochemical Parameters of Soil

The pH, conductivity, moisture, nitrate, sulphate and phosphate contents were determined at the Chemistry Department of the University of Port Harcourt using standard laboratory procedures (reference). TOC, the concentration of heavy metals and PAHs were conducted at International Energy Services Limited, Port Harcourt.

The samples were prepared for AAS by weighing the dried (2 g) into a digestion flask and adding an acid mixture (20 ml) containing concentrated nitric acid (HNO_3) (650 ml), Perchloric acid ($HClO_4$) (80 ml) and concentrated sulphuric acid (H_2SO_4) (20ml). The flask was then heated until a clear digest is obtained. Distilled water (50ml) was added to dilute the digested sample and then filtered using No. 42 Whatman filter paper into a 250 ml flask and made up to the mark with distilled water. Afterwards, appropriate dilutions were made for each element. The sample is thoroughly mixed by shaking and 100 ml was transferred into a glass beaker of 250 ml volume. The sample is aspirated into the oxidizing air-acetylene flame. When the aqueous sample is aspirated, the sensitivity for 1% absorption is observed in a GBC sensAA Atomic Absorption Spectrophotometer (AAS)

For the PAHs determination, each sample (5 g) was extracted with 100 ml of solvent (Hexane & dichloromethane 1:1 v/v) in a Soxhlet apparatus. After 16 hours of extraction, the contents of the flask were filtered and evaporated by a rotatory vacuum evaporator. The dried residue was resuspended into 2.5 ml of methanol and passed through a 5 cm anhydrous sodium sulphate column to remove moisture. The PAH contents in each soil sample were measured by reverse phase HPLC analysis.

Briefly, the 20 µl of the methanol soil extracts were injected into an HPLC system fitted with a 5 µm particle diameter C18 column. The elution gradient was programmed as follows [shown as percent (by volume) of methanol in water, acidified with 0.76 ml of H_3PO_4 per liter]: 50% for 2min, linear gradient to 80% at 5% per min, holding at 80°C for 16 min. The flow rate was kept at 1 ml/min. Peaks were measured at 254 nm. Concentrations of PAHs were calculated by comparing peak areas of sample chromatogram with that of peak area of standard chromatogram.

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RESULTS AND DISCUSSION

Physicochemical Analysis

The physicochemical parameters of the samples are summarized in Table 1. The mean pH values for the site under study are 6.3 for the topsoil (S_T) and 5.6 for the subsoil (S_S). While that for the control soils are 6.6 for the topsoil (C_T) and 6.2 for the subsoil (C_S), indicating a slight increase in acidity of the soil from the lubricating oil market. The lower pH of 5.6 in the Ss sample is an indication of a mildly acidic soil. In their separate works, Njoku *et al.*, (2008) and Johan *et al.*, (2020) had linked decrease in soil pH to oil pollution.

Table 1: Physicochemical parameters of soil samples

S/N	PARAMETERS	C_T	C_S	S_T	S_S
1	PH	6.6	6.2	6.3	5.6
2	CONDUCTIVITY (µs/cm)	71	89	152	209
3	NITRATE (mg/kg)	18.5	19.4	32.5	47.6
4	PHOSPHATE(PO_4^{3-}) (mg/kg)	43.5	22.5	14.7	19.5
5	PHOSPHORUS(P) (mg/kg)	14	7.5	4.8	6.4
6	PHOSPHATE(P_2O_5) (mg/kg)	32.5	17	11	14.6
7	SULPHATE (mg/kg)	0	0	40	20
8	MOISTURE CONTENT (%)	3.2	4.4	3.6	8.6
9	ORGANIC CARBON (%)	8.9	7.93	10.52	10.16

C_T = Control TopSoil; C_S = Control SubSoil; S_T = Sample TopSoil; S_S = Sample SubSoil

Increased mean conductivities for the samples from the site under study was observed (152µs/cm and 209µs/cm respectively for the topsoil and the subsoil) when compared to their respective values of 71µs/cm and 89us/cm for the control soil samples. The trend is the same for TOC, 10.52% and 10.16% for the soil under investigation, against 8.9% and 7.93% for the control samples. However, according to Arshi and Khan (2018), both the conductivity and TOC values in the control and site samples are higher than the standard values (2.0 µs/cm and 1.0% respectively) required for normal plant activities.

The mean nitrate contents of the soil samples from the lubricating oil market are 32.5 mg/kg for the topsoil and 47.6mg/kg for the subsoil. These are high values compared to the mean values for the control soil samples which are 18.5mg/kg for the topsoil and 19.4mg/kg for the subsoil. This is contrary to an expected increase in nitrate concentration due to the hydrocarbon-degrading activities of indigenous micro-organisms as observed in a work with crude oil (Devatha *et al.*, 2019). However, it was established in another investigation, that the percentage of biodegradation of hydrocarbons is less in lubricating oils (used or unused) when compared with kerosene and diesel. Also, the higher nitrate level in the contaminated soil could be due to a high concentration of the contaminants which can be inhibitory to microorganisms by toxic effects (Ijah and Antai, 2003; Abioye *et al.*, 2012)

The mean moisture contents for the samples from the site under study are 3.6% for the topsoil and 8.6% for the subsoil, which is high compared to that of the control soil samples (3.2% and 4.4% for the topsoil and the subsoil respectively). These values are shown pictorially in Appendix A. Some related earlier investigations that have to do with crude oil contamination reported decreases in moisture content with increasing crude oil contamination (Ghouse *et al.*, 1980; Devatha *et al.*, 2019). The variation could be attributed to not only the other additives in lubricating oils but to the fact that the site of investigation is full of human activities, enough to thoroughly homogenize the oil and water content of the soil unlike in those reported cases. As such, the oil films will form barriers that would rather reduce the natural evaporation and sipping-down of the soil water.

The mean phosphate ($PO_4\ 3^-$)/Phosphorus (P)/ Phosphate (P_2O_5) for the soil samples from the Ikoku market are 14.7/4.8/11mg/kg respectively for the topsoil, 19.5/6.4/14.6mg/kg for the subsoil. These are much lower than the control samples collected from a location that is 2 Km away which are 43.5/14/32.5mg/kg for the topsoil and 22.5/7.5/17mg/kg for the subsoil. The decrease may be attributed to microbial metabolism as posited by Margesin and Schinner (2001).

The mean sulphate values for the site under study are 40mg/kg for the topsoil and 20mg/kg for the sample subsoil compared to the controls soils which null values for both the top and subsoils. This could be attributed to the high level of contamination of the soil with lubricating oil. Mangas *et al.*, 2014) had posited that lubricating oils deteriorate as they become contaminated and undergo chemical changes by oxidation, with acidic materials as the major products.

Heavy Metals

Table 4.2 shows the values for the concentration of heavy metals for the sampling locations. The mean concentrations for Lead (Pb) are 3.70ppm and 3.85ppm for the sample topsoil and subsoils respectively. While the mean concentration of Lead (Pb) for the control samples were lower at 0.75ppm and 0.94ppm for the control topsoil and subsoils respectively.

Table 2: Heavy Metal Content of Soil Samples

S/N	Metals	Concentrations (mg kg^{-1})			
		C_T	C_S	S_T	S_S
1	Lead	0.75	0.94	3.70	3.85
2	Zinc	8.86	9.41	9.71	9.69
3	Arsenic	0.00	0.00	0.00	0.00
4	Chromium	103.66	59.5	197.29	82.18
5	Cadmium	4.08	0.87	7.42	3.35

Chromium and cadmium also exhibited higher levels of concentration in the site sample 197.29/82.18 mg kg^{-1} and 7.42/ 3.35 mg kg^{-1} respectively for the topsoil/subsoil. Their respective values in the control are 103.66/ 59.5 for chromium and 4.08/ 0.87 mg kg^{-1} for cadmium. Zinc also exhibited higher levels, with 9.71 mg

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kg^{-1} and 9.69 mg kg^{-1} for the sample top soil and subsoil respectively, while the control showed lower levels of 8.86 mg kg^{-1} and 9.41 mg kg^{-1} for the control topsoil and subsoils respectively. Arsenic showed zero concentration for all the samples. The respective World Health Organization target value concentrations in soil for lead, cadmium, zinc and chromium is 85, 0.8, 50 and 100 mg/Kg (W.H.O., 1996). The lubricant retail market can therefore be said to be contaminated with respect to cadmium and chromium.

Appendix B is an illustration of the concentration values of the metal with a histogram.

PAH

The total PAH levels for the soil samples collected at the lubricant retailing market was 3.757mg/kg for the topsoil and 4.696mg/kg for the subsoil which in turn is higher than those of the control samples which are 0.189mg/kg for the topsoil and 0.286mg/kg for the subsoil (Figure 1). This indicates an increase in the level of poly aromatic hydrocarbons, which in turn is due to the improper handling and disposal of lubricants in the market. The European classification system of soil contamination graded total PAHs concentration of 0.6–1 mg/kg as moderately polluted and over 1 mg/kg as heavily polluted (Maliszewska-Kordybach, 1996).

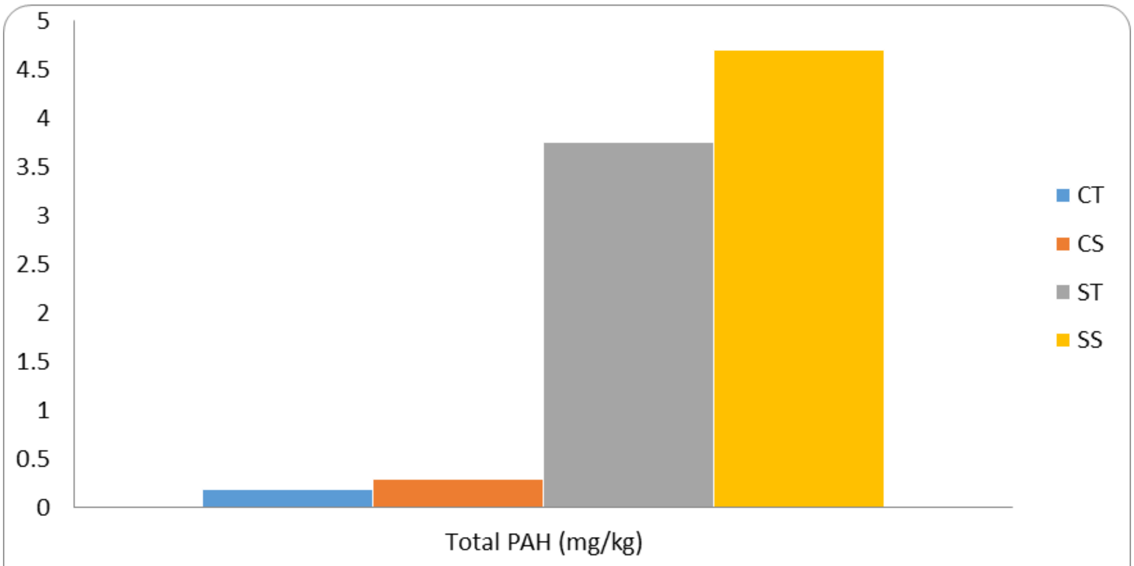

Fig 1: A histogram illustrating the total levels of PAH in the sample and control soils

The various PAHs and their concentrations are shown in Figure 2. Flouranthene showed the highest concentration of PAHs in all the soil samples. Flourene, Anthracene Benzo (a) Pyrene and Acenaphthene also showed high presence while Benzo (g,h,i) Perylene, Indeno (1,2,3-cd) Pyrene and Dibenzo (a,h) Anthracene made negligible contributions to the total PAH concentration.

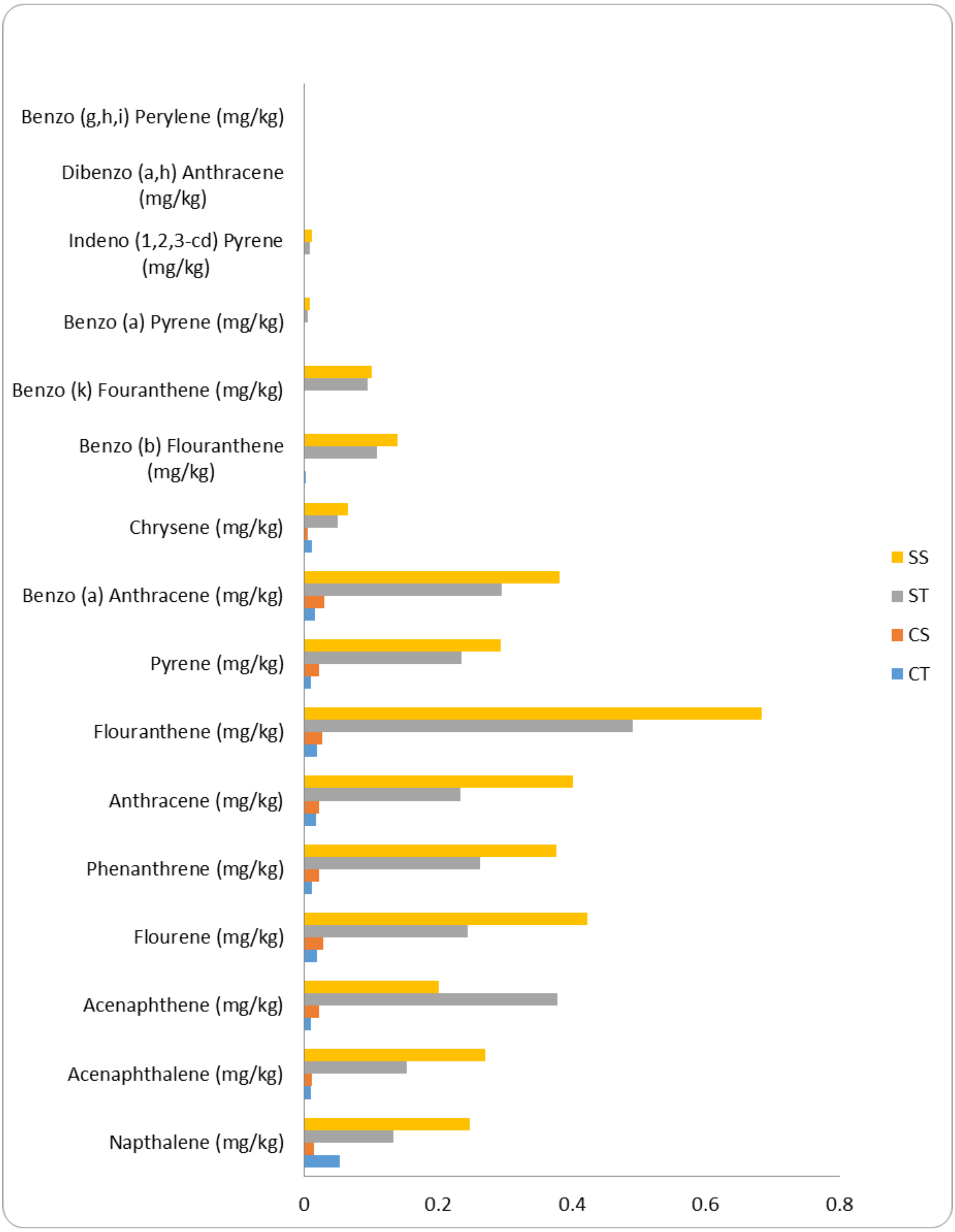

Figure 2: Various PAH Contents of the Soil Samples

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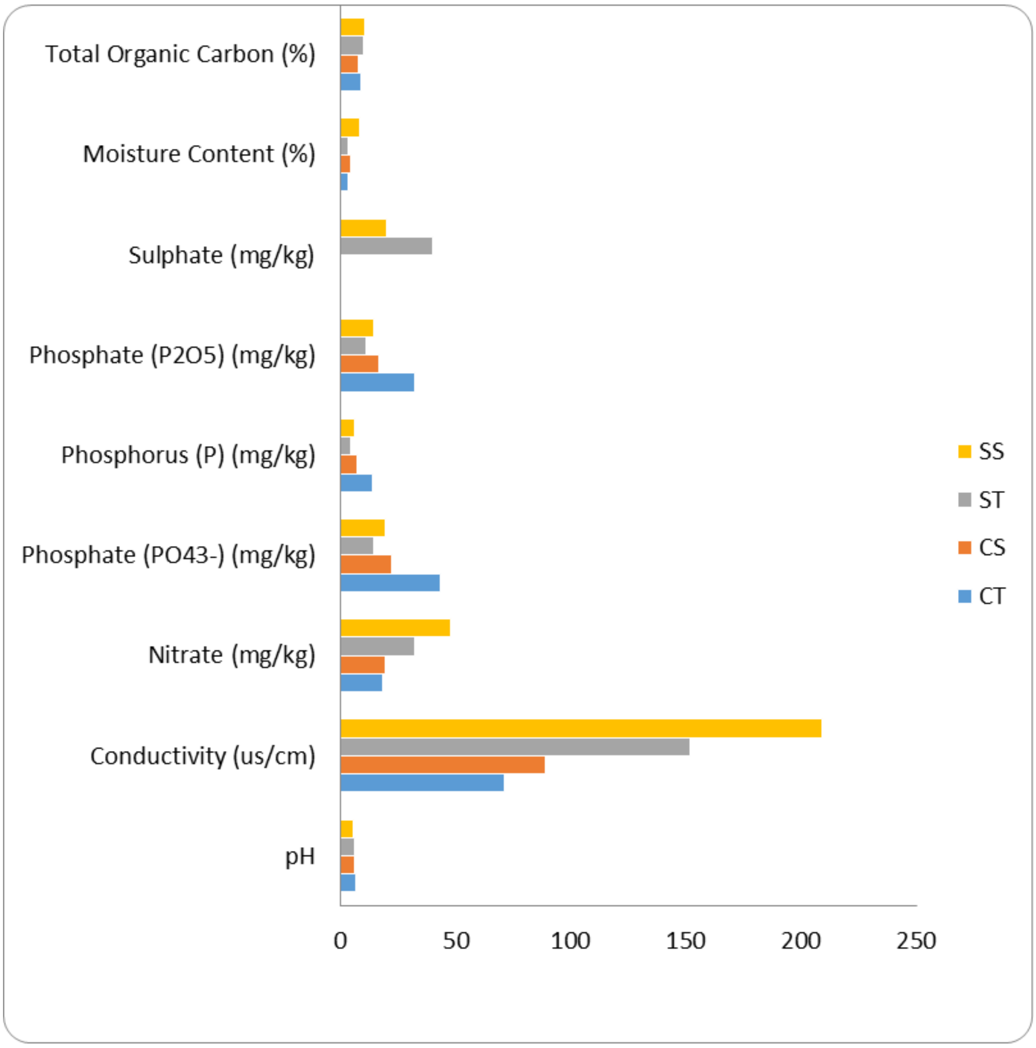
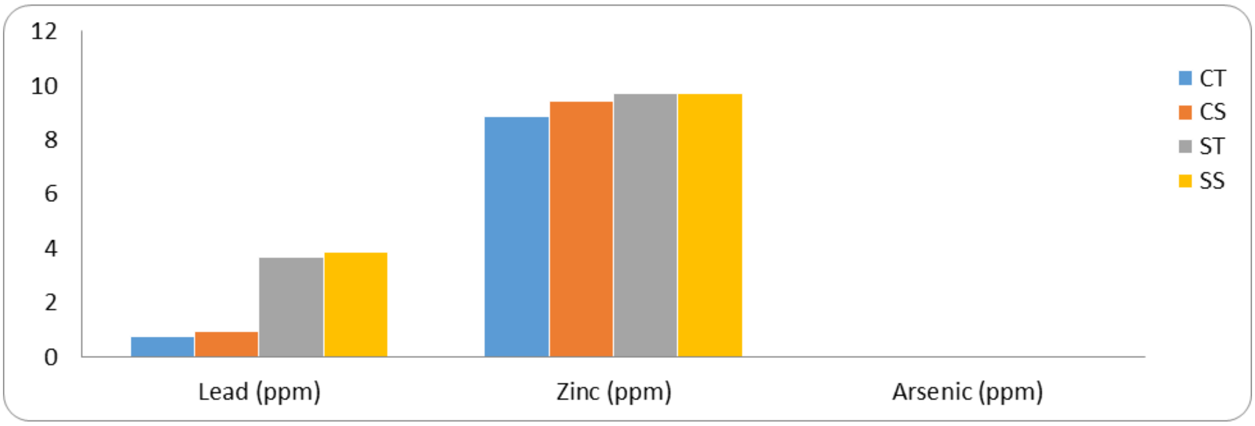
APPENDIX A: A Histogram illustrating the physicochemical properties and their level in both the sample and control soils



APPENDIX B: A Histogram illustrating levels of heavy metals in the sample and control



CONCLUSIONS

The physicochemical properties of soil sampled from Ikoku lubricant market indicate some alterations. Increased levels are seen in the conductivities, TOC nitrates and sulphate contents which are typical of hydrocarbon contamination. Also, the recorded decreases were seen in the pH and phosphate contents when compared with the control sample. Neither the increased nor the decreased properties seemed desirable for normal plant life. The total PAH concentration, the values cadmium and chromium are all indicators of polluted soil. The activities in the lubricant retailing market are the most likely source of the differences in the values of these parameters in the site and control soil samples. The implication is that these toxic compounds and elements could be washed off to nearby farmlands or sipped down to underground water with the attendant consequences.

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