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Measurements of Physicochemical, Elemental, and Total Petroleum Hydrocarbon Contents as Pollution Indicators of Soils in Petroleum Products Retailing Station Surrounding Areas

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Soils around petroleum products retailing stations were analyzed for their physicochemical, elemental, and total petroleum hydrocarbon contents with a notion to assess the contamination level of the soils. Samples were collected using standard analytical procedures and the elemental contents of the soils were determined using bulk scientific atomic absorption spectrometry/atomic emission spectrometry techniques, while total petroleum hydrocarbon and physicochemical parameters contents were determined using standard analytical techniques. Ten elements were analyzed and detected and their concentrations, enrichment factors, pollution index, and geoaccumulation index (I_{geo}) values established. The concentrations of most of the analyzed elements were higher in the oil-contaminated soils than those of other Nigerian soils, while the values of the analyzed physicochemical parameters followed the same trend. The results of the I_{geo} indicated that the soils were *very heavily polluted* (6.2–18.0) with all the metal, except V, which was *heavily polluted* (4.2). Very high enrichment factors and pollution index were obtained for Mn, Zn, K, Cu, Ni, Cr, and Pb. This was corroborated by their values, which were excessive when compared with their tolerable limits. The clustering results indicated that the elements were fairly correlated, indicating chemical affinity and/or similar sources. These were supported by the cross plot results (R^2 = 0.5), and the results of Pearson correlation matrices for the elements. Average total petroleum hydrocarbons level (1,233.0 mg/kg) of the soils was high when compared to studies of similar settings. The overall results showed that the soils were highly contaminated and may pose a threat to public health.

Keywords: atomic absorption spectrometry/atomic emission spectrometry, element, physicochemical, soil, total petroleum hydrocarbons

1. INTRODUCTION

Exploration, exploitation, and processing of petroleum hydrocarbons have been in existence for many years in Nigeria and the associated effects on the oil producing communities and areas where petroleum products retailing stations are located have been quite uncomfortable. These activities

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are in phase, but are of great destructive impact, ranging from the just allowable ones to totally terrible effects. For instance, the terrestrial and the aquatic biota, which composes the major source of livelihood to human lives in places where these activities are carried out, is known to be seriously affected. The pollution problems associated with incidents of oil spills around petroleum exploration and refinery areas in Nigeria have been the subject of many reports, but these transcend beyond those occurring around the exploration and processing zones. Contamination remote from these zones may have resulted from operations involved in petroleum products transportation, marketing, and end-users handling (Adebiyi et al., 2005, 2008; Osibanjo et al., 1986; Odeyemi and Ogunseitan, 1986). Invariably, kerosene, gasoline, diesel oil, and lubricating oil have been the most commonly marketed products, contaminating the environment through spillage from accidents involving transporting vehicles, overflow leaks from motor vehicles, poor handling at petrol retailing stations and auto-mechanic workshops, and other domestic activities. It is noteworthy that petroleum hydrocarbons are also known to be included in vehicular emissions, which could cause serious health damage as a result of air pollution (ATSDR, 1995). The origin of contaminants has a significant bearing upon the species present and, hence, the analytical methodology to be used. Unlike many other chemicals (notably pesticides), hydrocarbons were generally not applied to soils for a purpose and, thus, hydrocarbon contaminations result almost entirely from misadventure. There is also a body of evidence to show that certain organisms, notably higher plants, are capable of synthesizing hydrocarbons (De et al., 1997) and these too could find their way into the soil environment. These latter sources are, however, fairly minor and are unlikely to result in significant soil contamination.

The major contaminants found in petroleum are those of the non-hydrocarbons, which are heterocyclics containing sulfur, nitrogen, and oxygen; minerals, such as silica and metals, including transition metals like V, Ni, Pb, Cd, Ag, Zn, Cu, Mn, Cr, Fe, etc.; and large molecular weight asphaltic molecules and the hydrocarbons, mainly the polycyclic aromatic hydrocarbons (PAHs). The concentration of these contaminants may be minor, but they are the source of environmental pollutants and the cause of corrosion of equipment and poisoning of processing catalysts (Yen, 1975). The refinery process, however, serves to eliminate these contaminants by chemical conversions and upgrading. In some petroleum fractions, the amount of metal constituents may be lessened essentially to zero by virtue of the treatment received en route through the refinery; for other fractions, the amount may be considerably increased by metal pickup from pipes or storage facilities.

This study involves analysis of contaminated soils around petroleum products retailing stations for physicochemical parameters, total petroleum hydrocarbons, and metals (Zn, Pb, Na, K, Mn, Cu, Fe, Ni, Cr, and V), which are known to associate with hydrocarbon formation, refining, processing, and transportation; all of these may pose a threat to the residents of the areas when present at elevated concentrations. Most of the petroleum products retailing stations in Nigeria are sited within residential and commercial areas (Figures 1 and 2). Petroleum products that contain potential toxic elements and hydrocarbons can contaminate soils and leach into ground and surface waters, which are major sources of water for drinking and domestic use. These can result in ill health effects of the residents of the area. Hazards, such as fire outbreak, could result into environmental degradation, destruction of life and properties (Figure 2).

2. EXPERIMENTAL

2.1. Sample Collection and Treatment

Soil samples within the contaminated areas were collected randomly from petroleum products retailing stations in Ile-Ife, Southwestern Nigeria, which is located within: Latitude 7° 28′ 0 N;

FIGURE 1 Picture of a petroleum products retailing station located within a commercial center.

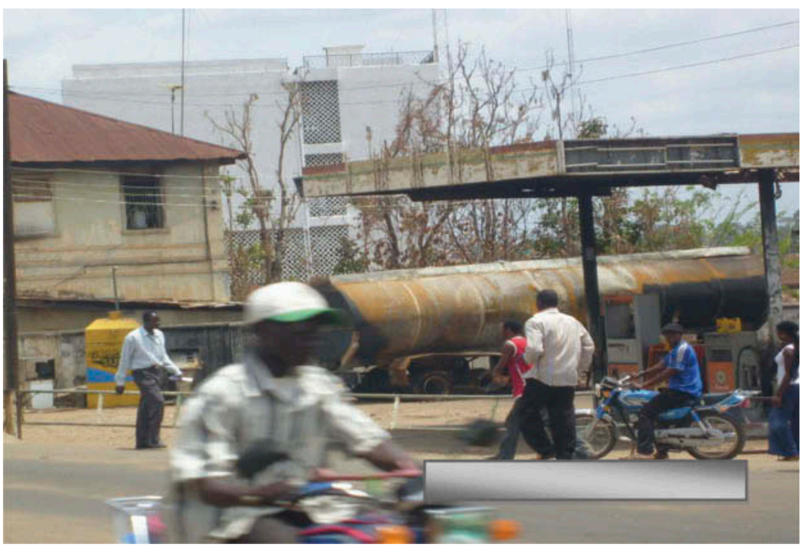

FIGURE 2 Picture of a burnt petroleum products retailing station located in a residential area.

Longitude: 4° 34′ 0 E. The sampling carried out at six different locations with the following specific sampling points of the study area (N 07° 29.772′ E 004° 30.735′; N 07° 29.778′ E 004° 30.196′; N 07° 29.758′ E 004° 30.651′; N 07° 29.377′ E 004° 31.895′; N 07° 29.282′ E 004° 32.583′;and N 07° 29.181′ E 004° 32.86′). At each sampling point, two sub-samples at a depth of 0–5 cm were collected using a hand trowel and were wrapped in aluminum foil. A sub-sample for total petroleum hydrocarbons (TPHs) determination was preserved at 0°C and was immediately taken to the laboratory for analysis. Soil samples for metal analysis were prepared using the IAEA-TECDOC (IAEA, 1993) method.

2.2. Quality Assurance and Quality Control

For quality control, all instruments used were operated as contained in the instrument's handbooks. Calibration of the bulk scientific atomic absorption spectrometry/atomic emission spectrometry (AAS/AES) was done using mixed calibration standard solutions prepared from the pure British Drug House (BDH) Analar grade salt of each element viz. 10 mg/kg of Mn, Zn, K, Na, Cu, Fe, Ni, Cr, V, and Pb and the percentage recovery ranged from 90 to 95. A blank determination was also made following the same procedure described above. All of the reagents used were of BDH Analar grade and the solvents (methanol, *n*-hexane, and water) used were triply distilled. All of the glassware and sample bottles were cleaned using the procedure of Laxen and Harrison (1981). Only glassware was used in the TPHs determination and three replicate measurements were carried out.

2.3. Determination of Physicochemical Parameters

The following physicochemical parameters (pH, chlorinity (Cl^-), electrical conductivity (EC), carbonate (CO_3^{2-}), and organic matter) of the soils were determined using various standard analytical methods (Bailey, 1986), while the total hydrocarbon oil content was determined by the IOC (1982) procedure.

2.4. Elemental Analysis

The soil samples ware digested using the *aqua regia* procedure of the International Standard (IS, 1995) and the elemental concentrations determined using AAS/AES at the International Institute of Tropical Agriculture (IITA), Ibadan, Nigeria.

3. RESULTS AND DISCUSSION

3.1. Total Petroleum Hydrocarbon (TPH) Analysis

The TPH content of the oil-contaminated soils is presented in Table 1 with values ranging between 150.0–2,220.0 mg/kg with a mean value of 1,233.0 mg/kg. Levels observed in this study were close to the values (100.0–3,000.0 mg/kg) obtained in studies of similar settings viz. polluted sediments in the Niger Delta (Adam and Duncan, 1999) and 1,400.0 mg/kg in Lake Washington, USA (Wakeham and Carpenter, 1979). The TPH analysis result showed that the soils of the area were highly contaminated with the petroleum hydrocarbons and may present a threat to public health.

3.2. Physicochemical Parameters Analysis

The results of the physicochemical parameters are also presented in Table 1. The oil-contaminated soils in this study have pH values ranging from 5.64 to 6.63 with a mean value of 6.27. The mean pH value of the oil-contaminated soils was comparatively higher than that (4.85) of soils of bituminous sands area studied by Asubiojo and Adebiyi (2011) but close to the pH value (6.56) of soil around petroleum products depot investigated by Adebiyi and Adeyemi (2010). Oiling must have discouraged the leaching of basic salts, which were responsible for raising pH of the oil-impacted soils, while the

TABLE 1
Comparison of the Analyzed Parameters in the Oil-contaminated Soil Samples with Other Nigerian Soils (±sd)

Analyte	*This Study*	*Adebiyi and Adeyemi (2010)*	*Asubiojo and Adebiyi (2011)*	*SPDC (2004)*	*Amusan et al. (2000)*	*Tolerable Limits (Sonon and Gaskin, 2009)*	*Background Minimum Level (IUGS/IAGC, 2007)*	*Geoaccumulation Index (I_{geo})*	*Pollution Index (PI) (Onianwa and Adoghe, 1997)*	*Enrichment Factor (EF)*
K (mg/kg)	1,826.0–2,105.0 (1,983.0 ± 107.0)	2,886–3,167 (3,025)	33-64–102.1 (54.58)	20.0–50.0 (34.0)	N.A	N.A	0.026	16.6	N.A	1.00
Na (mg/kg)	2,336.0–2,601.0 (2,461.0 ± 105.0)	3,155–3,338 (3,274)	N.A	N.A	N.A	N.A	0.030	16.5	N.A	1.14
Mn (mg/kg)	584.0–1,453.0 (1,016.0 ± 61.0)	2,368–2,549 (2,456)	72.86–275.98 (174.33)	N.A	N.A	N.A	0.004	18.0	N.A	11.4
V (mg/kg)	33.6–49.1 (41.9 ± 6.8)	148.6–185.1 (163.1)	N.D–2.04 (0.78)	0.01 (Range and Mean)	N.A	100	2.71	4.22	0.42	2.78
Cr (mg/kg)	85.8–181.0 (145.0 ± 14.0)	413.8–465.9 (451.3)	N.D–3.97 (0.63)	6.10–10.47 (8.56)	N.A	100	3.00	6.46	**1.45**	15.9
Ni (mg/kg)	67.3–107.0 (90.3 ± 15.0)	268.7–368.7 (329.1)	N.D–4.00 (1.69)	1.38–4.36 (2.70)	N.A	50.0	2.00	6.21	**1.81**	11.3
Pb (mg/kg)	184.0–250.0 (216.0 ± 24.0)	488.6–520.4 (512.1)	N.D–10.22 (2.84)	N.A	18.7	75.0	3.00	6.44	**2.88**	164
Zn (mg/kg)	317.0–489.0 (409.0 ± 57.0)	836.9–1,018 (910.8)	N.D–12.45 (3.88)	3.71–15.79 (6.44)	26.7	100	3.00	7.46	**4.09**	57.5
Cu (mg/kg)	211.0–336.0 (261.0 ± 47.0)	831.4–1,050 (917.7)	7.03–40.45 (19.27)	8.59–21.27 (12.12)	36.6	100	0.860	8.90	**2.61**	55.3
Fe (mg/kg)	1,266.0–1,626.0 (1,501.0 ± 28.0)	2,656–2,940 (2,777)	62.48–386.08 (143.94)	76.88–305 (155)	1659	N.A	0.160	13.6	N.A	0.39
pH	5.64–6.63 (6.27 ± 0.4)	6.18–7.51 (6.56)	4.16–5.50 (4.85)	N.A	N.A	N.A	N.A	N.A	N.A	N.A
EC (μScm^{-1})	668.0–1,230.0 (972.0 ± 24.0)	188–358 (308)	1,670–2,740 (2,400)	N.A	N.A	N.A	N.A	N.A	N.A	N.A
Cl^{-} (mg/kg)	42.5–96.4 (58.3 ± 21.0)	51.0–122 (84.1)	25.51–71.43 (38.59)	N.A	N.A	N.A	N.A	N.A	N.A	N.A
Organic matter (g/kg)	46.0–52.1 (48.4 ± 2.4)	36.7–52.9 (47.3)	59.01–87.90 (73.96)	N.A	N.A	N.A	N.A	N.A	N.A	N.A
CO_3^{2-} (%)	0.136–0.863 (0.670 ± 0.7)	0.818–1.82 (1.209)	N.D–2.64 (0.73)	N.A	N.A	N.A	N.A	N.A	N.A	N.A
TPHs (mg/kg)	150.0–2,220.0 (1,233.0 ± 41.0)	1,670–2,650 (2,050)	N.A	N.A	N.A	N.A	N.A	N.A	N.A	N.A

Note: N.A = Not available; N.D = Not detected; sd = Standard deviation; PI > 1.0 are in bold type; E.F > 10 are in bold type.

mean content (0.67%) of CO_3^{2-} in the oil-impacted soils was close (0.73%) to soils studied by Asubiojo and Adebiyi (2011) but less than the value (1.21%) obtained on oil-impacted soils examined by Adebiyi and Adeyemi (2010). The Cl^- ions (58.1 mg/kg) were generally higher in the oil-contaminated soils in this study than in the soils (38.59 mg/kg) studied by Asubiojo and Adebiyi (2011) and again less than the Cl^- contents (84.1 mg/kg) of soils studied by Adebiyi and Adeyemi (2010). The use of chloride compounds, such as sodium hypochloride (as sweetener) and $CuSO_4/NH_4Cl$ slurry (as catalyst) in the refinery might have resulted in accumulation of the Cl^- ions in refined petroleum oils and, hence, in the oil-contaminated soils (Ofunne, 2008). It was also reported by NALCO (2007) that mineral salts, such as NaCl, $MgCl_2$, and sulphates of Ca and Mg, are present in crude oils as a result of long contact of water and crude oils with salt substrates and soils. If crude oil was not desalted well the inorganic salts could increase the Cl^- ion content of the soils via contamination of the soils by oils; this would eventually elevate the EC of the soils. The electrical conductivity (EC) of the oil-contaminated soils in this study ranged between 668–1,230 μScm^{-1} with a mean concentration of 972.0 μScm^{-1}, which is higher than the values of EC of the soils studied by Asubiojo and Adebiyi (2011) but less than the value obtained on the oil-impacted soils studied by Adebiyi and Adeyemi (2010). The higher value of EC for the oil-contaminated soils was due to the high values of salts mentioned above.

3.3. Elemental Analysis

Table 1 presents the concentrations of elements in the oil-contaminated soil samples. Ten elements viz. Mn, Zn, K, Na, Cu, Fe, Ni, Cr, V, and Pb were analyzed and detected by AAS/AES. Sodium was the most abundant element (range 2,336.0–2,601.0 mg/kg) with a mean concentration of 2,461.0 mg/kg, followed by K (range 1,826.0–2,105.0 mg/kg) with a mean concentration of 1,983.0 mg/kg and Fe (range 1,266–1,626 mg/kg) with a mean concentration of 1,502.0 mg/kg, while V is the least (range 33.6–49.1 mg/kg) with a mean concentration of 41.9 mg/kg in the oil-contaminated soils.

Table 1 also compares the elemental concentrations of the oil-contaminated soils in this study with other Nigerian soils including soils of a similar setting. The results of this study for K, V, Cr, Ni, Pb, Zn, Cu, and Fe were higher than those of other Nigerian soils (Asubiojo and Adebiyi, 2011; SPDC, 2004; Amusan et al., 2000), but less than their values in oil-contaminated soils studied by Adebiyi and Adeyemi (2010). Vanadium, Cr, Ni, Zn, Cu, and Fe were higher in the soils in this study because they are transition elements, which are known to be associated with hydrocarbons formation (Tissot and Welte, 1984), while Pb in the form of its derivative (tetraethyl lead) is deliberately added to gasoline during processing as an anti-knocking agent. Sodium is present in crude oil in the form of its mineral salts as a result of long contact of water and crude oil with salty substrates and soils (NALCO, 2007). From the discussion above, the concentration of these potential toxic metals are expected to be higher in the oil-contaminated soils than other Nigerian soils free of oil contamination or anthropogenic influence.

Table 1 also compares the average concentrations of the analyzed elements in the oil-contaminated soils with their risk reduction standards viz. value limits that pose no significant risk for residential use (Sonon and Gaskin, 2009). It is observed that potential toxic elements—Cr, Pb, Zn, Ni, and Cu—were excessive. This was supported by the calculated pollution index (PI) values of the elements, which were greater 1, indicating that the soils were contaminated with the elements (Onianwa and Adoghe, 1997). The enrichment factor (E.F) (using K as the reference element) for each element was calculated using average concentrations of elements in the crust by Brady (1984) (Table 1). All of the elements except Na, Fe, and V were highly enriched (E.F > 10) in the oil-contaminated soils. The high E.F values obtained for the potential toxic elements should then be a cause of concern due to their possible adverse effect on human's and animal's health.

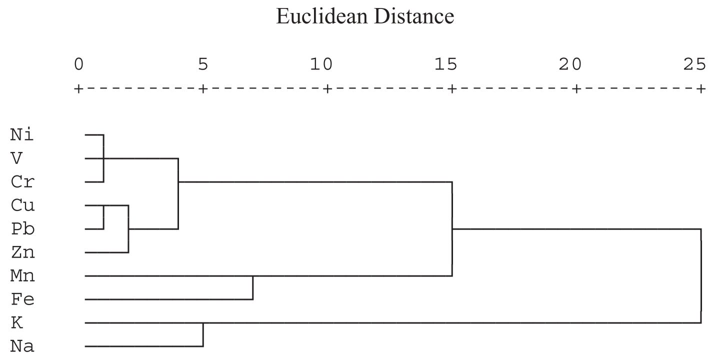

FIGURE 3 Cluster analysis of the analyzed elements in the soils.

Table 1 also compares the results of the analyzed elements with their background levels (Jones et al., 1987). It is observed that all of the concentrations of the elements exceeded their background levels. The geoaccumulation index (I_{geo}) values of the elements in the soils were calculated using the equation according to Diatta et al. (2008) and their background levels as reported by Jones et al. (1987). With reference to Diatta et al. (2008), it can be inferred that the soils were *very heavily polluted* with all the metals analyzed (I_{geo} values > 5), except V ($4 < I_{geo} < 5$), which is *heavily polluted*, showing that the soils have been contaminated/polluted with the elements.

Figure 3 presents clustering analysis results for the analyzed elements in the contaminated soils. On the *X*-axis of the dendrogram is the similarity matrix using Euclidean distance, while on the *Y*-axis are listed the analyzed elements. The results showed four groups viz. Ni/V/Cr, Cu/Zn/Pb, Mn/Fe, and Na/K and also indicated that (Ni, V, Cr, Cu, Zn, Mn, Fe, and Pb) and (Na and K) showed the closest interelement clustering, indicating chemical affinity and/or similar sources. For instance, V, Cr, Cu, Zn, Mn, and Fe are transition metals having variable oxidation states among other similar properties, which they possess, and they are also metals that are associated with hydrocarbon formation and these elements, including Pb, are vehicular wear metals, while Na and K are group I elements with similar properties. These are supported by the strong and positive correlation ($R^2 = 0.5$) of cross plot results of Na/K and Mn/Fe, respectively (Figures 4 and 5), and also the results of Pearson correlation matrices for the analyzed elements in Table 2. It is noted that all of the transition metals have positive correlations, but Fe/Mn ($r = .891$), Cu/Zn ($r = .887$), Fe/Zn ($r = .883$), Fe/Cu ($r = .881$), Ni/

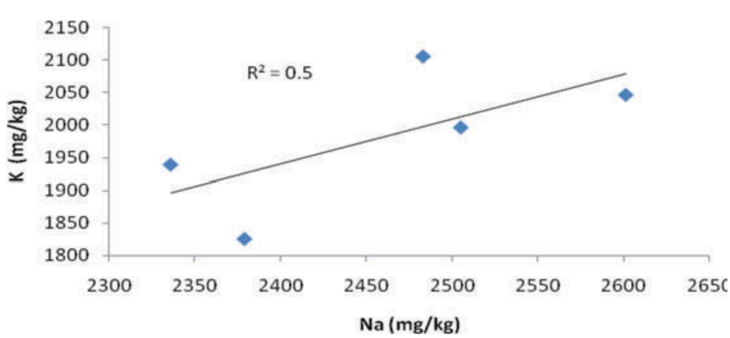

FIGURE 4 Cross plot of K and Na concentrations in the investigated soils.

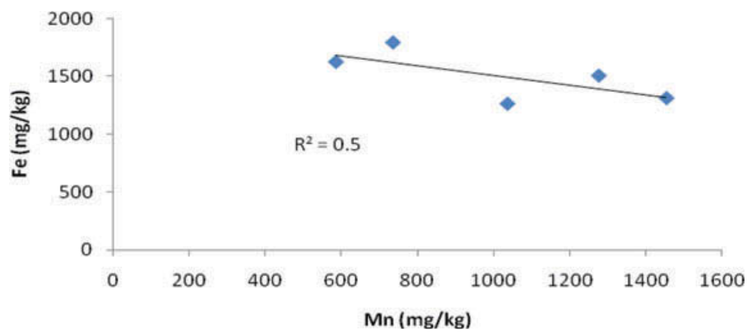

FIGURE 5 Cross plot of Mn and Fe concentrations in the investigated soils.

TABLE 2
Pearson Correlation Matrix of the Analyzed Elements in the Bituminous Sands Using the Elements (mg/kg) as Variables[a]

	Mn	*Zn*	*K*	*Na*	*Cu*	*Fe*	*Ni*	*Cr*	*V*	*Pb*
Mn	1.000									
Zn	.548	1.000								
K	.824	.822	1.000							
Na	.098	.682	**.977**	1.000						
Cu	.336	**.887**	.545	.527	1.000					
Fe	**.891**	**.883**	.618	.794	**.881**	1.000				
Ni	.521	**.953**	.832	.690	.744	**.916**	1.000			
Cr	.562	**.967**	**.900**	.722	.701	**.878**	**.988**	1.000		
V	.394	.772	**.935**	.455	.799	**.884**	**.924**	**.887**	1.000	
Pb	.377	.407	.132	.780	**.980**	**.889**	.368	.315	.477	1.000

[a]Positively, strong and *significant* correlated elements are in bold type.

Zn (r = .953), Ni/Fe (r = .916), Cr/Zn (r = .967), Cr/Fe (r = .878), Cr/Ni (r = .988), V/Fe (r = .884), V/Ni (r = .924), and V/Cr (r = .887), while Na/K (r = .977) has a strong, positive, and *significant* correlation, while Zn/Ca (r = .985) also had a strong, positive, and *significant* correlation.

4. CONCLUSION

In this study, selected physicochemical parameters (pH, Cl^-, CO_3^{2-}, EC, and organic matter contents), metals (Mn, Zn, K, Na, Cu, Fe, Ni, Cr, V, and Pb), and TPHs in soils around petroleum products retailing stations were determined using appropriate different types of analytical techniques. The oil-contaminated soils were very rich in these potential environmental pollutants as a result of their high EFs, which are corroborated by their high values of I_{geo} and PI. The excessive levels when compared with their maximum permissible levels, high EF, PI, and I_{geo} values obtained for these elements and TPHs are worrisome because of their feasible undesirable effects on plants and human and animal health and can also alter the ecosystem components of the area, for instance when the oils are washed into the ground and surface waters of the area or emitted and pollute the ambient air. The clustering results indicated that the elements were fairly correlated, indicating chemical affinity and/or similar sources. These were supported by the cross plot results (R^2 = 0.5), and the results of Pearson correlation matrices for the elements. Average TPHs level of the soils was high when compared with studies of similar settings. The overall results showed that the soils of the area were highly contaminated and may present a threat to public health.

Since it has been established by this study that the oil-impacted soils have excessive levels of potential toxic metals and TPHs, it is recommended that removal of these metals should be carried out; the addition of Pb derivatives as an anti-knocking agent should be discouraged and substituted with other mild anti-knocking agents, such as ethanol and hydrogen fuel, which should be encouraged. Indiscriminate discharge of petroleum oils and petrochemicals should also be discouraged, and improved standards would allow petroleum retail locations to be safely located in many settings in Nigeria. There is, therefore, a call for further inquiry and appropriate action by environmental bodies to prevent environmental degradation and natural disaster, such as fire outbreak, which can result in loss of life and properties as can be seen in Figure 2.

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