Nigerian Research Journal of Chemical Sciences (ISSN: 2682-6054)
Volume 14, Issue 1, 2026

Impact of Spent Lubricating Oil Contamination on the Physicochemical Characteristics of Soil Samples

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Accepted: *January 23, 2026.* **Published Online***: January 29, 2026*

ABSTRACT

The increasing contamination of soil ecosystems due to indiscriminate disposal of spent lubricating oil, underscores the significance of assessing its impact. Soil samples were artificially contaminated in the laboratory by spiking 1 kg with 100 mL of spent engine oil obtained from a mechanic workshop in Akure, Nigeria to simulate in-situ pollution. Using standard analytical procedures, parameters including pH, organic matter content (determined by Walkey-Black wet oxidation method), particle size distribution (analyzed by the hydrometer method), and cation exchange capacity (determined using the ammonium acetate extraction method) were evaluated. Soil samples collected from a football field were used as control. The contaminated soil recorded a total petroleum hydrocarbon (TPH) level of 5500 mg/kg, while no TPH was detected in the control. The polluted soil exhibited lower pH (6.73) compared to the control (7.20); moisture contents (7.97% polluted vs 6.01% control); and organic matter (4.37% polluted vs 2.15% control). Cation exchange capacity increased (1.26 polluted soil vs 1.02 meq/ 100 g control); while oxidation-reduction potential shifted from -200 mV to (200 mV in control samples. The findings indicate that spent oil may impair soil fertility and ecological function. Proper disposal and remediation are recommended to mitigate hydrocarbon pollution in affected environments.

Keywords: Hydrocarbon pollution, physicochemical properties, spent engine oil, soil contamination.

INTRODUCTION

Soil is a very complex ecosystem that sustains plant growth, regulates water flow, and supports microbial communities essential for nutrient cycling. The balance of soil physicochemical properties such as pH, organic carbon, and cation-exchange capacity determines how well these processes occur [1]. Any disturbance that alters these properties can significantly influence soil fertility and ecosystem productivity.

One of the major pollutants that is threatening soil balance is spent lubricating oil (SLO). It is a waste product generated after lubricating oil has been degraded in automobile engines. The compositions of spent oil include hydrocarbons, heavy metals such as lead and cadmium, and polycyclic aromatic hydrocarbons, that are toxic and persistent in the environment [2]. Obnoxious discharge, such as onto open land or drainage channel, is a practice that has been going on for several years in developing regions where waste management infrastructure is weak [3]. When spent lubricating oil is discharged into the soil, it creates a hydrophobic film that makes it difficult for smooth movement of air and water, causing limited infiltration and poor aeration. These physical changes are often accompanied by chemical alterations, including increased total petroleum hydrocarbon content and heavy-metal accumulation [4]. Over time, such contamination can hinder microbial activity, slow nutrient turnover, and reduce soil productivity [5]. The extent of damage of spent oil on the ecosystem varies with the quantity of oil introduced, soil type, and exposure duration. In many documented cases, SLO pollution leads to lower soil pH, reduced moisture retention, and lowered enzymatic activity, which has a direct effect on seed germination and plant growth [6,7]. Microorganisms that are beneficial to the soil also die and this leads to deterioration in the soil structure and organic matter stability.

One of the most reliable approaches to assess whether an area is polluted by spent lubricating oil is through the quantification of total petroleum hydrocarbons in the affected soil. The assessment of hydrocarbon concentrations periodically provides vital information that will be useful in evaluating the degree and persistence of pollution, most especially when compared with data from unpolluted reference sites or established regulatory standards. Such empirical records are very useful to monitor effectively and as well devise a management strategy for the ecosystems exposed to spent engine oil. Assessing the physicochemical properties of soils impacted by spent lubricating oil is critical for understanding the extent of ecological damage. Continuous monitoring and analysis of key physicochemical parameters remains essential for tracking pollutant dynamics and guiding remediation efforts.

The novelty of this study lies in its integrated laboratory-based simulation of spent lubricating oil contamination using soils sourced from a recreational land-use area (football field), an environment that is often overlooked in hydrocarbon pollution studies. Unlike many studies that focus on heavily industrialized or long-term polluted sites, this work provides controlled

experimental evidence of early-stage physicochemical alterations in soil following artificial oil contamination.

Accordingly, this study aimed to investigate the effects of spent lubricating oil contamination on selected physicochemical properties of soil under controlled laboratory conditions in order to assess its implications for soil quality and environmental sustainability. Specifically, the study evaluated changes in pH, organic matter content, moisture content, cation exchange capacity, soil texture, and oxidation-reduction potential in contaminated soils relative to uncontaminated control soil samples.

MATERIALS AND METHODS

Study location and sample collection

Sandy-loamy surface soil samples (0-15 cm depth) were collected from a football field located in Ajipowo Surulere Estate along Ondo Road, Akure, Ondo State, Nigeria. The site was considered relatively undisturbed to ensure reliable background soil conditions, and was used as control for this study. Composite sampling of the soil followed standard field procedures for surface soil collection, as described in soil survey and analytical guidelines [8,9].

Spent lubricating oil was collected from an automobile workshop in Oba-ile, Akure, Ondo State, Nigeria. The oil sample was stored in a clean, airtight container prior to use to avoid been contaminated and as well reduce evaporation [10].

Sample preparation and pre-treatment

Collected soil samples were transported to the laboratory in polythene bags. The organic materials and stones that are visible were removed manually, followed by the thorough mixing of soil samples collected from different collection spots to obtain a composite sample [11]. The composite soil sample was air-dried on thick cardboard for seven days at ambient temperature to reduce moisture content and stabilise the sample before treatment, in accordance with standard soil sample preparation procedures [12, 13]. After air-drying, the soil was gently crushed and passed through a 2-mm sieve to obtain a uniform texture. A mechanical mixer was then used to homogenise, and increase surface area exposure for physicochemical interaction.

Experimental design

A microcosm of the prepared soil was weighed using an analytical balance. The soil was contaminated by spiking the sample with spent lubricating oil in accordance with the procedure

for hydrocarbon-soil interaction studies [14]. Contamination was performed at a ratio of 1 kg of soil: 100 mL of spent lubrication oil. The oil was gradually added to the soil sample and thoroughly mixed with the aid of a spatula and glove to ensure uniform distribution. Control (uncontaminated) soil and oil-contaminated soil samples were stored in labeled containers at the Chemistry Laboratory prior to analysis.

Physicochemical Analysis

Soil pH

Soil pH was measured using standard procedures. Exactly 10 g of air-dried and sieved soil was transferred into a 100-mL beaker in duplicate, and 20 mL of distilled water was added to achieve a soil to water ratio of 1:2 (w/v). The mixture was stirred intermittently for 30 min to allow equilibration. A calibrated glass-electrode pH meter (pre-standardized with pH 7.0 and pH 4.0 buffer solutions) was then inserted into the slurry to obtain pH values, once readings stabilized [15, 16].

Organic matter

Organic matter content was determined using the Walkey-Black wet oxidation method. Approximately 1g of soil was placed in a conical flask, followed by the addition of 10 mL of 0.167 M potassium dichromate solution. The flask was gently swirled to ensure adequate contact between the oxidizing agent and soil particles. Subsequently, concentrated sulphuric acid was added, and the mixture was vigorously agitated and allowed to stand for 30 minutes to facilitate oxidation. Afterward, 100 mL of distilled water and 3-4 drops of ferroin indicator were added, and the solution was titrated against 0.5 M acidified ferrous ammonium sulfate to determine the excess dichromate. A blank titration was also performed, and organic carbon content was computed and converted to soil organic matter percentage using Equation 1 [17].

$$Organic\ Carbon = \frac{(B-T)x\ M\ x\ 0.003\ x\ 1.33}{Weight\ of\ Soil}\ x\ 100 \quad (1)$$

where B = blank titre value, T = sample titre value, M = molarity of ferrous sulphate,

% Organic Matter = %Organic Carbon x 1.724 [17].

Particle size

The particle size analysis was carried out using the hydrometer method. A 51 g sample of air-dried 2-mm sieved soil was weighed into a beaker, and 100 mL of calgon (sodium hexametaphosphate) dispersing solution was added. The mixture was allowed to soak for 30 min and then stirred

mechanically for approximately 3 min to ensure proper dispersion. The suspension was transferred into a 1-L sedimentation cylinder and filled to the mark with distilled water. After thorough mixing using a plunger, one drop of amyl alcohol was added to reduce foaming. The hydrometer was carefully lowered into the suspension, and the first reading was taken at 40 seconds (R_{40sec}), followed by measurement of the suspension temperature. The reading was repeated until consistent values were obtained. Two hours after mixing, the second hydrometer reading (R_{2h}) and temperature were recorded. These values were used to determine the percentage sand, silt, and clay fractions using Equations 2-6 according to Stoke's Law principles. [17].

$$\%\ silt + clay = \frac{(R40sec - Ra) + Rc}{Weight\ of\ Soil}\ x\ 100 \quad (2)$$

$$\%\ clay = \frac{(R2h - Rb) + Rd}{Weight\ of\ Soil}\ x\ 100 \quad (3)$$

Ra = 40 s blank hydrometer reading, Rb = 2h, blank hydrometer reading, Rc = 40 s. Correction factor (temperature x 0.36), Rd = 2h. Correction factor (temperature x 0.36).

$$\%\ sand + \%silt + \%clay = 100\% \quad (4)$$

$$\%silt = (silt + clay) - clay \quad (5)$$

$$\%Sand = 100 - (silt + clay) \quad (6)$$

Cation Exchange Capacity (CEC)

Cation exchange capacity was analyzed using the ammonium acetate extraction method. A 10 g of weighed soil sample was treated with 100 mL of 1 M NH_4OAc, shaken for 2 h, and left overnight. The supernatant was decanted into a 100 mL flask. The residue was re-extracted with 30 mL of 1 M $NH_4OA_{C,}$ shaken for 30 min, and the extract combined and made up to 100 mL. Potassium and sodium were quantified using a flame photometer, while concentrations of calcium and magnesium were determined using an atomic absorption spectrophotometer [17].

Soil moisture content

Soil moisture content was determined using the standard oven-drying method. A clean, pre-weighed moisture can was dried in an oven and allowed to cool in a desiccator before recording its weight (W_1). Approximately 10-20 g of the soil sample was placed into the can, and the weight of the can plus moist soil was recorded (W_2). The sample was then transferred to a thermo-controlled oven and dried at 105-110 ^{0}C for 24 h to ensure complete removal of moisture. After drying, the can was removed from the oven and allowed to cool in a desiccator for approximately 1 h to prevent moisture absorption from the atmosphere. The final weight of the can plus oven-

dried soil was recorded (W_3). The percentage of moisture content was computed using the relationship as shown in Equation 7:

$$\% \, Moisture = \frac{W_2 - W_3}{W_3 - W_1} \, x \, 100 \quad (7)$$

This method is very precise in the determination of soil moisture content, which is crucial for the interpretation of soil physical and biochemical properties [10,17].

Soil oxidation-reduction potential

Soil oxidation potential was measured using a standard redox electrode. Fresh soil samples were homogenized, followed by the preparation of soil-water suspension at a ratio of 1:2.5 using distilled water. Equilibration of the suspension was sustained for 30 min with occasional stirring. Using standard redox buffer solutions (+220 mV and +470 mV), the ORP meter was calibrated prior to use. The electrode was rinsed and inserted into the soil suspension, and the reading was taken between 2-5 min after stabilisation. The values of the ORP were recorded in millivolts (mV) [18].

Heavy metal determination in soil

The total concentrations of selected heavy metals in soil samples were determined following an aqua regia wet-digestion protocol. Exactly 1.0 g of each homogenized soil samples was weighed into a clean 100 mL kjeldahl digestion flask. Subsequently, 20 mL of freshly prepared aqua regia (mixture of concentrated nitric acid HNO_3 and concentrated hydrochloric acid HCl in a ratio of 1:3) was added to the flask. The mixture was heated in a fume cupboard and gently refluxed until near dryness, ensuring complete dissolution of metal bearing mineral phases. Heating continued until a slightly clear solution was obtained, indicating effective oxidation of organic matter. The digest was allowed to cool and was then filtered through Whatman No. 42 filter paper into a 50 mL volumetric flask. The filtrate was quantitatively made up to volume with distilled water. The resulting solution was analyzed for Ca, Mg, Cu, Zn, Mn, Cd, Cr, Pb and Fe using Atomic Absorption Spectrophotometry (AAS), while Na and K were analyzed using flame photometer in accordance with established instrumental procedures [19-21].

RESULTS AND DISCUSSION

Table 1 presents the physicochemical parameters of the soil samples. The physicochemical properties of the oil-contaminated soil differed markedly from the control soil, indicating the influence of spent oil pollution on soil quality. The oil-polluted soil exhibited a slightly acidic pH (6.73) compared to the control soil (7.20). Petroleum hydrocarbons are known to release organic acids during degradation, which lowers soil pH [22]. Reduced pH in oil-impaired soils has similarly been reported by Ayoola and Kutshik [23], who attributed acidity shifts to microbial decomposition of hydrocarbons and associated by-products. The pH decline is particularly significant because soil acidity strongly influences nutrient availability, microbial activity, and overall soil health.

Table 1: Physicochemical parameters of the soil samples (uncontaminated and contaminated)

Parameter	Oil contaminated soil	Control soil
pH	6.73	7.20
CEC (meq per 100 g)	1.26	1.02
Moisture content (%)	7.97	6.01
Soil organic matter (%)	4.37	2.15
ORP (mV)	-200	200
Texture classification	Sandy	Sandy
Sand (%)	91.2	89.3
Silt (%)	3.62	4.55
Clay (%)	5.16	6.06

A lower pH environment can inhibit the growth of beneficial microorganisms while favouring acid-tolerant species, potentially altering the ecological balance of the soil.

The cation exchange capacity was slightly higher in contaminated soil (1.26 meq/100g) than in the control (1.02 meq/100 g). Increased CEC may be linked to elevated soil organic matter, as organic colloids contribute to exchange sites [1]. The oil-polluted soil recorded higher organic matter (4.37%) compared to the control (2.15%). This increase aligns with findings by researchers [24, 25], who reported enhanced organic carbon in oil-polluted soils due to hydrocarbon accumulation and reduced microbial mineralization rates.

Moisture content was greater in the contaminated soil (7.97%) than in the control (6.01%), this observation is likely due to hydrophobic hydrocarbons reducing soil aeration and evaporation [26]. The reduced aeration is further supported by the negative ORP value (-200 mV) observed in the contaminated soil, contrasting with a positive ORP (+200 mV) in the soil. Negative ORP indicates an anaerobic and reduced environment, which is typical in hydrocarbon-impacted soils due to oxygen depletion by hydrocarbon-utilising microbes [18].

Both soils were classified as sandy, though slight variations existed in particle distribution. The contaminated soil showed slightly higher sand (91.2%) and lower slit (3.62%) and clay (5.16%) percentages compared to the control (sand 89.3%, silt 4.55%, clay 6.06%) Hydrocarbon contamination can alter soil structure by forming hydrophobic films around particles, affecting sedimentation and aggregation processes [27].

In Table 2, the concentrations of major and trace metals exhibited noticeable differences between the oil contaminated soil and the uncontaminated control, reflecting the influence of spent lubricating oil on soil geochemistry. Generally, petroleum-impacted soils tend to accumulate metals due to the metallic additives in lubricants and the adsorption of metals onto hydrocarbon-rich matrices [28]. The findings in Table 2 are consistent with these established patterns.

Macronutrients such as Na, Ca, and Mg showed mixed responses to contamination. Sodium increased from 30.70 mg/kg in the control to 35.40 mg/kg in the contaminated soil, a trend commonly associated with the saline nature of some petroleum products and the displacement of exchangeable cations during oil infiltration [29]. Similarly, calcium was slightly elevated in the contaminated sample (32.05 mg/kg) compared with the control (27.90 mg/kg). Previous studies have shown that Ca may remain bound to organic fractions in hydrocarbon polluted soils, leading to localized increases [30]. In contrast, magnesium decreased markedly from 33.90 mg/kg in the control to 27.45 mg/kg in the contaminated soil. This reduction has been linked to reduced microbial mineralization and cation exchange imbalance caused by hydrocarbon coatings on soil particles [31].

Table 2: Total metal concentration in soil (mg/Kg)

Metals	Sample ± SEM (PPM)Oil contaminated soil	Control Soil ± SEM (PPM)
Na	35.400± 0.140	30.700 ± 0.070
K	51.000 ± 0.012	53.300 ± 0.283
Ca	32.050 ± 0.601	27.900 ± 0.283
Mg	27.450 ± 0.035	33.900 ±0.141
Cu	0.438 ± 0.001	0.553 ± 0.001
Cd	0.100 ± 0.0004	0.110 ± 0.000
Fe	3665 ± 0.006	5945 ± 0.001
Mn	0.187 ± 0.001	0.210 ± 0.000
Pb	0.277 ± 0.001	0.421 ± 0.0004
Zn	0.795 ± 0.001	1.048 ± 0.001

Potassium showed a slight decline in contaminated soil (51.00 mg/kg) relative to the control (53.30 mg/kg). Hydrocarbon contamination often disrupts K cycling due to reduced microbial activity and impaired weathering of k-bearing minerals [32]. These changes in macronutrient levels indicate that oil contamination alters the soil's nutrient holding capacity, potentially impairing plant growth and soil fertility.

Trace metals (Cu, Cd, Fe, Mn, Pb, and Zn) generally decreased in concentration in the contaminated soil. Copper declined from 0.553 mg/kg (control) to 0.438 mg/kg (contaminated soil). A similar pattern was observed for Fe (5945 to 3665 mg/kg), Pb (0.421 to 0.277 mg/kg), Mn (0.210 to 0.187 mg/kg), and Zn (1.048 to 0.795 mg/kg). Reduced availability of these metals is a common phenomenon in oil polluted soils where hydrocarbons form hydrophobic barriers, limiting metal solubility and mobility [33]. Additionally, heavy metals may become immobilised by strong adsorption to organic matter present in petroleum residues, thereby reducing their extractable forms [34]. Cadmium showed a slight decline (0.110 to 0.100 mg/kg), though the difference was marginal. Cadmium often shows limited reactivity with hydrocarbons due to its

relatively high mobility and weak interactions with organic coatings [35]. The observed reduction nonetheless aligns with the general pattern of metal immobilisation in hydrocarbon-impacted soils.

CONCLUSION

The physicochemical properties of both the contaminated and control soils were analysed using standard laboratory procedures to assess the impact of the oil contamination. Composite soil samples were collected and artificially contaminated by spiking them with spent engine oil to simulate nearly static subsurface pollution conditions.

The physicochemical analysis in this study shows that oil contamination induces substantial alterations in soil chemistry and redox conditions The contaminated soil exhibited markedly elevated TPH, reaching 5500 mg/kg compared to 0 mg/kg in the control, confirming the presence severe hydrocarbon pollution. This contamination was associated with a shift towards more acidic, organic rich, and reduced soil conditions, all of which are known to negatively impact soil fertility, microbial functioning, and overall plant growth. Additionally, the CEC increased from 1.02 meq/100 g in the control soil to 1.26 meq/100 g in the oil contaminated soil, likely due to the accumulation of organic residues and hydrocarbon-derived colloids. These observations align with previous reports indicating that petroleum hydrocarbons disrupt soil quality by modifying nutrient dynamics, impairing biological processes, and altering soil structural and chemical stability.

Furthermore, the results revealed that oil contamination significantly modifies soil metal profiles elevating some macronutrients while reducing many extractable trace metals. These alterations have implications for soil quality, plant uptake, and ecological health. Hydrocarbon-induced immobilization of metals may temporarily reduce bioavailability, but long-term mineralization could later release these metals into the soil ecosystem, increasing ecological risks. The findings reinforce the need for remediation strategies that address both organic pollutants and metal dynamics in oil-polluted soils.

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