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Research Article

Soil Amendment Potential of Liquid and Organo-Mineral Fertilizer on Spent Engine Oil-Polluted Soil

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Abstract

Background and Objective: If plants are to grow to their potential, the soil must provide a satisfactory environment for plant growth. Soil pollution with hydrocarbon affects soil aesthetics, biomass, fertility crop yields and threatens food security. This study, therefore, investigates the effect of organo-mineral and liquid fertilizers in the remediation of spent engine oil-polluted soil. **Materials and Methods:** Soil Sample was polluted at a 10% concentration level with spent engine oil and treated with LF (Liquid fertilizer), OMF (Organo-mineral fertilizer) and a combination LOMF (Liquid organo-mineral Fertilizer). The Total Petroleum Hydrocarbon and five selected heavy metals concentrations of the soil were monitored for 20 weeks. **Results:** It was observed that the soil treated with LOMF had the highest net removal of total petroleum hydrocarbons (TPH) 89.94 %, this was followed by LF 86.17% and then OMF 84.89 %. The control treatment (F0) had the lowest TPH reduction (56.52%). There were significant differences at ($p < 0.01$) between the treatment fertilizers (F0, LF, OMF and LOMF). **Conclusion:** This study has demonstrated Organo-mineral and liquid fertilizers effectively reduced the total petroleum hydrocarbon as well as influence the reduction of heavy metals in the soil.

Key words: Liquid fertilizer, organo-mineral, engine-oil, heavy-metals, hydrocarbon, degradation, soil, amendment, treatment, pollution

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Competing Interest: The authors have declared that no competing interest exists.

Data Availability: All relevant data are within the paper and its supporting information files.

INTRODUCTION

Many techniques have been developed in the past for remediation of contaminated sites. Some of them include physical removal of contaminants, chemical degradation, photo-degradation and excavation of contaminated sites, etc. But these efforts have yielded limited successes, coupled with the fact that often they lead to further degradation of the environment. Hence, the biological approach offers a more reliable and better environmentally friendly method for remediation of hydrocarbons and heavy metals polluted soils. Any substance that enters the soil become part of the biological cycle that affects all life forms either positively or negatively. When the soil is polluted, the ecosystem is altered and agricultural activities are affected.

Contamination of the soil by spent engine oil creates an unsatisfactory condition for life in the soil. This is due to the poor aeration it causes in the soil, immobilization of nutrients, loss of water-holding capacity, lowering of soil pH and reduction in soil catalase enzyme activities as well as inhibitory effects on the nitrate reductase activities of plants^{1,2}. Ogboghodo *et al.*³ reported that, within two weeks, the addition of chicken manure triggered 75% degradation of hydrocarbons in crude oil-polluted soil and is environmentally friendly. Eneje *et al.*⁴ reported that poultry and green manure or in combination tend to break down chemicals of key contaminants and contribute to enhancing the removal of these contaminants. Also, Osuji and Nwoye⁵ reported that the application of NPK fertilizer on oil spill site at Owaza in the Niger-Delta region of Southern Nigeria enhanced restoration of the carbon to nutrient ratios leading to stimulation of microbial activities. The addition of poultry droppings as an organic nutrient was beneficial for maize growth and enhanced biodegradation of oil-polluted soils⁶. Ultra *et al.*⁷ reported that the application of slurry compost and biofiltration (SCB) liquid fertilizer significantly affected the mobility and bioavailability of Cd and Pb metals in the growth media and enhanced uptake of the same by *Populus alba* × *Populus glandulosa* seedlings. Irina *et al.*⁸ reported that when fertilizers are added as amendments to polluted soil, it enhances the transfer of certain elements including heavy metals and the use of a combination of hyperaccumulator and appropriate fertilizer will reduce the time necessary to effect a reduction of metal concentration to permissible levels in polluted soils. Organo-mineral fertilizer (Slurry from battery cage poultry farm plus ash from burnt palm fruit bunch and NPK fertilizer), which was employed in this study, has never been used in any research of this nature. Also, the combination of liquid fertilizer and organo-mineral

fertilizer treatments in the same research work is not common in literature. The findings from this research work will have practical implications on the use of organic fertilizer for hydrocarbon breakdown and bioavailability.

This study has environmental, agricultural, socio-economic and research implications as it tends to influence the use of waste materials to degrade and clean-up pollutants to tolerable levels and restore the environment. Economically, the huge cost associated with pollutant decontamination and restoration of environmental safety is drastically reduced using natural and ecological benign processes such as supported in this study. The study aimed to investigate the effect of spent engine oil, liquid and organo-mineral fertilizer treatments on soil properties.

MATERIALS AND METHODS

Study location: The experimental plot for this study is located in the farm area of the University of Calabar Botanic Gardens, Calabar. Calabar is located between Latitudes 4° 78' and 5° 09' N and Longitudes 8° 15' and 8° 26' E and lies between the valleys of two rivers: The Great Qua River on the Eastern side and the Calabar River on the West. This research project was conducted from March 2014-September, 2015.

Experimental laboratory: Physicochemical analysis of soil samples was carried out in the Soil Science Laboratory, Faculty of Agriculture, University of Calabar while analysis of total petroleum hydrocarbons (TPH) and heavy metals was undertaken at the BGI Resources Laboratory, Port Harcourt, Rivers State.

Source of materials: Spent engine oil was collected from mechanic workshops in Calabar. The oil was thoroughly mixed in a drum before application in experimental plots. The Organo-mineral fertilizer composed of fowl droppings (slug) collected from a battery cage Poultry farm located in Ekureku, Abi LGA, Cross River State and was mixed with sawdust to form organic fertilizer (compost). The organic fertilizer was further mixed with NPK fertilizer (to form organo-mineral fertilizer) before application. The Liquid fertilizer was obtained from John Ker Company Limited, Utu Ikpe, Ikot Ekpene, Akwa Ibom State

Soil sampling: Topsoil (0-20 cm depth) was collected from each of the four plots arranged in a complete randomized design using a spade. The collected soil was weighed in 20kg parts and transferred into nylon bags after proper mixing. It was labeled accordingly.

Soil pollution: At 0% pollution level, 0 liters of spent engine oil was added to the soil, 2 L were added to the soil at 10% pollution level. Calculation of soil pollution percentage:

$$\text{Soil pollution (\%)} = \frac{\text{Quantity of spent engine oil}}{\text{Quantity of soil}} \times \frac{100}{1}$$

$$\text{Soil pollution (10\%)} = \frac{2\text{L}}{20\text{kg}} \times \frac{100}{1}$$

Treatment application: The excavated soil was polluted with spent engine oil at 10% (2 L) levels and returned to their respective pits lined with a nylon bag and allowed to settle for 2 weeks. After 2 weeks, the soil was re-excavated with the polythene bag and treated with organo-mineral fertilizer (200 g) and Liquid fertilizer (450 mL) and a Liquid organo-mineral fertilizer (LOMF) as contained in the treatment combinations and returned to their respective pits. Re-excavation was done to allow for the proper mix with the treatment and enhance the aeration.

Hydrocarbon degradation in n-days (%) = $100 - (\text{TPH}_n - \text{TPH}_{\text{initials}}) \times 100$

where, n is number of days of study = 140 days⁹

Analytical methodologies

Physicochemical properties: Soil samples were taken before the zero-day and at the end of the bioremediation study. The collected samples were taken to the Soil Science laboratory for the physicochemical analysis. The soil samples were air-dried for three days, powdered and sieved through a 2mm mesh sieve. The physicochemical qualities of the soil samples were determined using the AOAC¹⁰ methods as described by Anetor and Omueti¹¹.

The parameters determined were: pH, organic carbon, nitrogen, phosphorus, potassium, calcium, magnesium, hydrogen, aluminum, effective cation exchange capacity (ECEC) and base saturation.

Determination of soil pH: This was determined using a pH meter. Ten grams of the soil sample was placed in a beaker, with 10 mL of distilled water added to it. The mixture was stirred and allowed to stand for 30 min. Then the electrode of the pH meter was inserted into the mixture and the pH reading taken.

Determination of soil organic carbon: This was determined using the dichromate wet oxidation method as modified by

Eno *et al.*¹². One gram of the soil sample was dispersed with 10 mL of 1 N potassium dichromate ($\text{K}_2\text{Cr}_2\text{O}_7$). The organic matter in the solution was oxidized using 20 mL of concentrated sulphuric acid. The resulting solution was allowed to stand for 30 min and diluted with 200 mL of distilled water. Then 1 mL of diphenylamine indicator was added before titrating with 1 N ferrous sulphate (FeSO_4).

Determination of total nitrogen: The determination of total nitrogen was done using the Kjeldahl method modified by Eno *et al.*¹² in which the nitrogen in 10 g of the air-dried soil sample was converted into ammonium product by digestion with concentrated H_2SO_4 . The reaction was facilitated by adding 2 g of CuSO_4 and 5 g of Na_2SO_4 (Oxidative catalyst). The nitrogen content of the sample was obtained by titration with 0.1 N HCl.

Determination of phosphorus: Available phosphorus was determined by Bray P-1 method as modified by Eno *et al.*¹². Six grams of air-dried soil sample was extracted with 30 mL of P-1 solution, centrifuged at 2000 rpm for 15 min and filtered. Then 21 mL of the resulting solution was diluted to 40 mL using distilled water. To the reaction mixture was added 8 mL of ascorbic acid for color development and the absorbance of the resulting solution determined using a spectrophotometer at 882 nm.

Determination of exchangeable cations: Exchangeable cations were extracted by leaching 5 g of each soil sample into a density bottle using 1 N NH_4OAC solution (ammonium acetate)¹³ for 1 h. The filtrate obtained following centrifugation at 2000 rpm for 30 min was used for the determination of calcium and Magnesium content by titration with 0.1 M EDTA solution using NH_4CL buffer and calcium with KOH in the first and second titration, respectively.

Determination of heavy metals in soil: The method of the American Public Health Association (APHA) 3111B was used APHA¹⁴. Procedure: 10g of the dried solid sample was weighed and transferred into an acid-washed 250 mL extraction bottle. About 100 ml of extraction reagent (reagent-grade disodium ethylenediaminetetraacetate- Na_2EDTA 0.1M) was poured into the flask, sealed and was shaken for 30-45 min in an automatic Stuart SSL1 Orbital Shaker (Cole-Parmer, Staffordshire, ST15 OSA, UK). The suspension was filtered through a Whatman Grade 1 Qualitative Filter Paper (Cytiva, Marlborough, MA USA). The extracted solutions and blank were then run using Agilent 240 AA Atomic Absorption Spectrophotometer (Agilent, Santa Clara, Ca, USA):

$$\text{Concentration of element X (mg L}^{-1}\text{)} = A - B$$

Where:

A : Concentration of Element X (mg L⁻¹)

B : Concentration of Blank (mg L⁻¹)

Statistical analysis: The collected data were subjected to a two-way Analysis of Variance (ANOVA) using a 2-factor (2 × 4) factorial in a Completely Randomized Design (CRD). Significant means were separated using Duncan Multiple Range Test (DMRT).

RESULTS

Any material that can improve soil fertility must be rich in soil nutrients Nitrate (N), Phosphate (P) and Potassium (K). Table 1 shows the nutrient properties of the treatment used in this study. Organo-mineral fertilizer (OMF) had a pH of 7.9 which is neutral whereas Liquid Fertilizer had a pH of 9.5 (Basic). The OMF treatment had a higher concentration of N, P, K and Ca compared to the LF treatment. This may be as a result of the organic nutrient-rich constituent of the OMF-Slurry from battery cage poultry farm plus ash from the burnt palm fruit bunch.

Table 2 shows the baseline properties of the soil compared with changes in the properties after pollution. It revealed a decrease in the concentration of some nutrients properties including Total Nitrogen from 0.18-0.08%, Phosphorus from 8.70-6.75 mg kg⁻¹ and organic carbon from 2.3-1.07% after polluting the soil with spent engine oil. There were observed increase in some of the heavy metal including Zinc from 0.06-1.20 mg kg⁻¹, Lead, cadmium and copper from 0.06, 0.05 and 0.17 to 0.19, 0.07 and 0.42 mg kg⁻¹, respectively.

The treatments- Liquid fertilizer, Organomineral fertilizer and a combination of both denoted as LOMF were applied to the polluted soil. Four weeks after treatment application, the soil properties were analyzed to measure the effect the treatments had on the soil relative to the Polluted control sample seen in Table 3. Improvement Changes were observed amongst the three treatments inclusive of the control which may have occurred due to natural attenuation.

In Table 4, shows the percentage net reduction of the Total petroleum Hydrocarbon in the soil after 20 weeks. It revealed that the maximum net reduction of 89.94% was achieved using the combination treatment LOMF in a 10% soil pollution level and 85% reduction where there was no pollution.

Table 1: The chemical properties of liquid fertilizer and organo-mineral fertilizer used as amendments in this study

Treatment	pH	N	P	K	Ca	Mg
Liquid fertilizer (mg L ⁻¹)	9.5±0.03	26.40±0.1	7.30±0.05	15.00±0.00	10.00±0.00	24.00±0.25
Organo-mineral fertilizer (mg kg ⁻¹)	7.9±0.02	47.30±0.03	19.00±0.00	37.50±0.20	9.00±0.00	57.50±0.1
Mean±S.D						

Table 2: Soil Physicochemical properties before and after pollution

Parameters	Soil properties before pollution	Soil properties after pollution
pH	4.30±0.01	4.17±0.15
Org. C (%)	2.30±1.20	1.07±0.08
Tot. N (%)	0.18±0.10	0.08±0.01
P (mg kg ⁻¹)	8.70±1.89	6.75±1.11
Mg (mg kg ⁻¹)	0.67±0.23	0.67±0.12
K (mg kg ⁻¹)	0.08±0.01	0.08±0.02
Na (mg kg ⁻¹)	0.06±0.01	0.06±0.01
H+	2.78±2.06	1.68±2.36
ECEC	7.62±1.85	5.85±1.41
BS	41.53±10.93	53.67±8.33
CLAY	6.33±3.06	12.00±1.73
Silt	8.33±0.58	8.00±1.73
Sand	78.67±3.79	78.67±0.58
Ni (mg kg ⁻¹)	0.02±0.002	0.02±0.002
Zn (mg kg ⁻¹)	0.06±0.001	1.20±0.02
Pb (mg kg ⁻¹)	0.06±0.002	0.19±0.001
Cd (mg kg ⁻¹)	0.05±0.002	0.07±0.002
Cu (mg kg ⁻¹)	0.17±0.002	0.42±0.001
Mean±SD		

Table 3: Soil physicochemical properties of soil 4 weeks post-treatment

Parameters	F0	LF	OMF	LOMF
PH	4.17±0.15	4.30±0.00	4.30±0.06	4.50±0.15
Org. C (%)	1.07±0.08	2.90±0.62	3.10±0.42	2.50±0.36
Tot. N (%)	0.08±0.01	0.26±0.02	0.24±0.06	0.10±0.08
P (mg kg ⁻¹)	6.75±1.11	10.25±0.50	21.83±2.08	15.62±3.80
Mg (mg kg ⁻¹)	0.67±0.12	0.47±0.12	0.80±0.20	0.67±0.12
K (mg kg ⁻¹)	0.08±0.02	0.07±0.01	0.08±0.01	0.07±0.01
Na (mg kg ⁻¹)	0.06±0.01	0.05±0.01	0.06±0.01	0.06±0.01
H+	1.68±2.36	1.82±1.32	2.51±1.63	2.80±1.95
ECEC	5.85±1.41	6.07±1.28	6.36±1.48	6.57±2.15
BS	53.67±8.33	47.00±13.75	41.33±5.51	40.67±8.02
CLAY	12.00±1.73	6.00±3.00	4.67±1.53	5.33±2.52
Silt	8.00±1.73	9.00±1.00	8.33±0.58	9.00±1.00
Sand	78.67±0.58	78.00±4.36	80.00±4.58	85.33±3.79

F0: No fertilizer, LF: Liquid fertilizer, OMF: Organo-mineral fertilizer, LOMF: Liquid organo-mineral fertilizer

Table 4: Percentage reduction of the Total Petroleum Hydrocarbon (TPH) in the Soil

Treatment	Week 10 post treatment (%)	Week 20 post treatment (%)	Net reduction (%)
AT 0% Pollution			
F0 (control)	47.83	16.67	56.52
LF	25.00	33.33	50.00
OMF	35.00	64.08	76.65
LOMF	40.00	75.00	85.00
AT 10% Pollution			
F0	23.50	77.40	82.71
LF	35.97	78.40	86.17
OMF	49.72	69.96	84.89
LOMF	48.87	80.32	89.94

F0: No treatment, LF: Liquid fertilizer, OMF: Organo-mineral Fertilizer, LOMF: Liquid organo-mineral fertilizer

Table 5: Net percentage reduction of heavy metals in the soil at the end of the study (20 weeks)

Soil pollution level (%)	Treatment	Heavy metals (mg kg ⁻¹)				
		Ni	Zn	Pb	Cd	Cu
No pollution control	F0	23.75	78.33	73.02	80.85	72.51
	LF	16.67	80.00	84.13	65.96	58.48
	OMF	16.67	76.67	65.08	59.57	85.96
	LOMF	16.67	60.00	58.73	89.36	84.79
Polluted (10%)	F0	45.83	83.60	75.40	90.54	69.14
	LF	16.67	85.79	86.63	91.89	66.75
	OMF	16.67	77.96	78.61	94.59	58.85
	LOMF	16.67	71.77	85.02	94.59	36.12

F0: No treatment, LF: Liquid fertilizer, OMF: Organo-mineral Fertilizer, LOMF: Liquid organo-mineral fertilizer

The effect of the treatment on the heavy metal concentration in the soil was monitored for the 20 weeks duration of the study. Table 5 revealed that the maximum reduction of 45.83 % observed for Ni was achieved in the polluted soil that had no treatment. The net percentage reduction in the concentration of Zinc in the soil was observed in the soil treated with Liquid fertilizer treatment. Lead (Pb), Cadmium (Cd) and Copper (Cu) had a maximum net reduction of 86.63% with LF, 94.59% with OMF and 69.14% without treatment, respectively.

The results show that spent engine oil pollution causes an alteration in the physical and chemical properties of the soil especially depletion of the soil nutrients and biomass

and that the soil can restore itself by natural attenuation. The treatments act enhancers as well as nutrient replacement.

Even though the Nutrient properties of the OMF was significantly higher than those of the LF, the result revealed a higher TPH reduction and Heavy metal with the LF treatment compared to the OMF. This may be owing to the form of application. Soil may be able to take up nutrient more in the liquid form than the pulverized form.

The combination of both treatments LOMF which gave the maximum TPH reduction of 89.94%. can be said to have an improved effect on the soil properties than the individual treatments.

DISCUSSION

The Study shows spent engine oil in the soil had significant effects on soil properties like nitrogen content, pH, carbon as well as the presence of heavy metals. Agbogidi *et al.*¹⁵ described the deleterious effects oil in soil has on the biological, chemical and physical properties of the soil depending on the dose, type of the soil and other environmental factors. However, Zheljzkov and Warman¹⁶ reported that the application of compost and vermin-compost to oil-contaminated soil improved soil fertility, physical properties and contribute to a successful approach to phytoremediation. Baek *et al.*¹⁷ demonstrated that there were additions in the level of heavy metals (Vanadium and Lead) as the level of spent engine oil pollution increased. The reduction in the level of available soil nutrient (N, P and Mg) and rise in the level of heavy metals concentration is in agreement with the report of Udo and Fayemi¹⁸ that crude oil in the soil makes the soil condition unsatisfactory for plant growth, due to the reduction in the level of available plant nutrients in the soil or a rise in toxic levels of certain elements such as iron and zinc. Table 1 shows the chemical properties of the treatment used in this study which depicts their prerequisite for use as soil amendments. From this study, it was observed that organic fertilizer significantly increased organic C, total N, exchangeable cations and decreased heavy metal concentration in the soil. The addition of organic matter amendments such as compost, fertilizer and waste, is a common practice for immobilization of heavy metals and soil amelioration of contaminated soils¹⁹. Peat, compost and vermicomposting application led to effective immobilization of Pb, Cu, Zn and Cd photo accessible forms in soil. The effect of organic matter amendment on heavy metal bioavailability depends on the nature of the organic matter, their microbial degradability, salt content and effects on soil pH and redox potential, as well as on the particular soil type and metals concerned²⁰.

The actual toxicity of heavy metals will be affected by soil texture, organic matter content and pH. Adding organic matter to the soil can help tie heavy metals chemically, reducing their availability for potential uptake. Similarly, limiting to a neutral pH and maintaining optimal soil phosphorus levels can reduce heavy metal availability to plants. There is little evidence to suggest that some heavy metals such as lead are accumulated within crops, the main health hazards being through indirect ingestion from the soil and inhalation²¹.

Petroleum hydrocarbons are among the most common group of persistent organic contaminants and are known to have destructive effects on the environment and human health. The presence of these compounds which are components of spent engine oil has negative effects on plant germination and growth²². The toxicity of hydrocarbon could be caused by volatile compounds and hydrophobicity. Volatile hydrocarbons, primarily small and lightweight hydrocarbons, can easily move through cell membranes, thus causing toxic effects²³. Whereas, the hydrophobic property of oil-contaminated soils makes it difficult for water and air to get to the roots of plants.

The fertilizer treatment affected soil TPH, with the reduction being observed at all the different treatments. More reduction was seen with the LOMF (liquid organo-mineral fertilizer), which showed an 89.94% reduction at the 10% soil pollution, this was followed by the Liquid fertilizer. A two-way analysis of variance on the total petroleum hydrocarbon showed there was a significant difference between the treatments ($p < 0.01$). The TPH reduction could be attributed to the presence of sufficient organic matter in these fertilizers, which enhances higher microbial population and activities, leading to the breakdown of hydrocarbon in the spent engine oil and subsequent release of nutrients to growing plants. Similar observations on various treatments being effective in hydrocarbon removal to have been made by researchers^{22,24,25}. The reduction of the TPH level in the soil over time even without treatments may also be caused by natural attenuation. Liquid organo-mineral fertilizer (LOMF) and Organo-mineral fertilizer (OMF) have a high potential to improve properties of spent engine oil-polluted soil, enhance biodegradation of heavy metals (Zn, Cu, Pb, Ni and Cd).

This study has environmental, agricultural, socio-economic and research implications as it tends to influence the use of waste materials to degrade and clean-up of pollutants to tolerable levels and restore the environment. Economically, the huge cost associated with pollutant decontamination and restoration of the environmental safety is drastically reduced using natural and ecological benign processes such as supported in this study.

There is, therefore, the need for the government to enact strict laws on disposal of spent engine oil. There is also a need to educate the citizenry on the hazards of indiscriminate disposal of these pollutants into the environment, especially on farmlands. This will ensure crop safety, food security, prevention of ingestion of toxic elements through food chains by humans and animals; damaging the environment as a whole.

CONCLUSION

Results indicate that soil contamination with spent engine oil can be destructive to crop growth as it alters soil components and its micro-organisms, can affect human and animal health and detrimental to the environment in general. Amendment with fertilizers increases the nutrients in the soil which stimulates microbial growth, thereby increasing pollutant biodegradation and bioavailability of products for plant uptake.

SIGNIFICANCE STATEMENT

This study has shown that Organo-mineral fertilizers (made up of chicken droppings, NPK and ash) and liquid fertilizer are very effective in enhancing biodegradation and reduction in the total petroleum hydrocarbon (TPH) in polluted soils. Organo-mineral fertilizer (Slurry from battery cage poultry farm plus ash from burnt palm fruit bunch and NPK fertilizer), which has been employed in this study, has never been used in any research of this nature. Also, the combination of liquid fertilizer and organo-mineral fertilizer treatments in the same research work is not common in literature. Most literature reviews are limited to either organic fertilizer treatment or liquid fertilizer treatment and not a combination of the two.

The findings from this research work will have practical implications on the use of organic fertilizer for hydrocarbon breakdown and bioavailability. Though inorganic constituent can be used in phytoremediation activities, using organic constituent is ecologically friendly, enhances ecosystem nutrient cycles which are of great benefit to the environment. This is also beneficial to agriculture as it facilitates nutrients availability and uptake by plants.

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