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Investigation of impact of slow-release fertilizer and struvite on biodegradation rate of diesel-contaminated soils



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ABSTRACT

Fossil fuel remains the most used form of energy in the world and while it is only available in some countries, there is always the need to transport it to disadvantaged countries, which often results in oil spillages. Distribution of petroleum and petroleum products upstream and downstream also results in oil spillages. In cases where there is ineffective or no containment during and after oil spillages, the problem of oil pollution could be worsened by migration and seepage into receiving systems. In such a case, there is a need to identify and apply appropriate remediation techniques to reduce the impact and extent of the spillage. Some microbes have been identified as having the ability to degrade hydrocarbons and could potentially be harnessed for oil spillage cleanup but, would require sustainable nutrients supply to enhance biodegradation rate and efficiency. This study investigated the impact of slow-release fertilizer and struvite on the biodegradation rate and efficiency of dieselcontaminated soils with a view to determining whether struvite could be applied in the biodegradation of oilcontaminated soils as an alternative, low-cost, and sustainable approach. The test models were dosed with a cumulative oil loading of 178 mg m⁻² week⁻¹ based on the derived oil loading of 9.27 g m⁻² year⁻¹ with a view to comparing the efficiency of the nutrient sources. The nutrient sources, Osmocote Plus controlled-release fertilizer granules and struvite were applied at a one-off rate of 17 g m^{-2} to provide nutrient requirements and enhance biostimulation. The effect of the nutrients was studied by monitoring microbial growth in different growth media, the evolution of Carbon dioxide, elemental content by Inductively Coupled Plasma Atomic Emission Spectrometry, Electrical conductivity of effluent, Total Petroleum Hydrocarbons by infra-red spectroscopy, and pH of effluent. The results showed that Osmocote® plus controlled-release fertilizer is more effective in enhancing microbial growth than Struvite slow-release fertilizer hence, a relatively faster bioremediation rate. It should be noted that struvite accumulation in public water pipe works is an issue of environmental concern hence, any meaningful utilization and application of this "waste" in the bioremediation of contaminated soils would offer a sustainable and low-cost means of disposal of what is hitherto an environmental problem. However, it may require augmenting of the deficient nutrients to enhance effectiveness. This could be accomplished by incorporating naturally occurring nutrient sources such as rock phosphate, kelp, and bedrock nitrogen for sustainability.

1. Introduction

Fossil fuel remains the most used form of energy in the world and petroleum products are utilized globally for a wide range of purposes. However, crude oil is only available in some countries known as oilproducing countries, and there is a need to transport petroleum and associated products from these regions of production to the consumer countries, depots, refineries, industries, service stations, and homes. Most of these transportation activities result in oil spillages (Gangadhari et al., 2021; Clancy et al., 2018). It is estimated that over 1.3 t of oil is released into the marine environment alone, and about 5.6 million tonnes of oil have been released into the environment since 1970 (Hazra et al., 2012). Whilst there is a global recognition of the impact of major oil spillages on the environment (as these are major news events when

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they occur), there is little or no information on oil spillages of <700 t that occur over the ground. According to ITOPF (2021), an organization that monitors global oil spillages, "information from published sources generally relates to large spills, often resulting from collisions, groundings, structural damage, fires or explosions, whereas the majority of individual reports relate to small, operational spillages; reliable reporting of small spills (<7 tonnes) is often difficult to achieve". Furthermore, the huge majority of oil spillages (81%) fall into the smallest category i.e. <7 t, and occur more frequently than large spillages >700 t (ITOPF, 2021). Poor reporting of oil spillages makes it difficult to monitor remediation efforts and in some developing countries, even more difficult to determine whether there are remediation efforts at all. In cases where there is ineffective or no containment during and after oil spillages, the problem of oil pollution could be worsened by evaporation of volatile organic compounds (VOCs), migration and seepage of oil into other environments, and in some cases, oil can be carried by stormwater into receiving natural water bodies. No wonder, hydrocarbons are one of the main constituents of stormwater particularly, in oil-polluted areas.

Diesel is a petroleum hydrocarbon and a major product of the petroleum refining process. Petroleum hydrocarbons contain chemicals such as benzene, toluene, ethylbenzene, xylene, and naphthalene, which can pollute soils, surface and groundwater systems as well as being hazardous to plants, animals and humans (USDHHS, 1999; Singh and Chandra, 2014; Dhaka and Chattopadhyay, 2021). Studies have shown that extensive exposure to a high concentration of diesel may result in serious health problems such as the development of liver and kidney disease, and potential damage to the bone marrow (EU, 2013; Ezejiofor et al., 2014). Similarly, hydrocarbons in diesel have been reported to be responsible for critical diesel toxicity to plants grown in dieselcontaminated soils (Fatokun and Zharare, 2015). Due to the high toxicity profile of petroleum hydrocarbons, it is expedient to identify oil spillages and apply appropriate remediation techniques quickly in order to reduce the impact and extent of the spillage.

Some microbes have been identified as having the ability to degrade hydrocarbons and hence, provide an important means of dealing with oil pollution and ultimately, a process that could be harnessed for oil spillage clean-up (Das and Chandran, 2011; Kostka et al., 2011; Kumar and Sai Gopal, 2015). Also, numerous studies have investigated the process of biodegradation and how it could be enhanced/optimised as well as the identity and characteristics of oil-degrading microbes [Ferguson et al., 2017; Macaulay, 2014; Sihag et al., 2014). One of the key factors identified is the impact of nutrient availability on biodegradation rate and efficiency (Newman et al., 2011; Sihag et al., 2014; Adams et al., 2015; Karthick et al., 2019). It is important that not only are nutrients available to kick start, sustain and enhance biodegradation to optimal rate, but that the required nutrients are also available in the right form and concentrations until the contaminant is completely degraded. The addition of supplemental nutrients has been observed to provide the much needed nourishment required for growth and energy in conditions where nutrient levels are low or depleted. Padayachee and Lin (2011) observed significantly enhanced biodegradation of diesel with the addition of supplemental fertilizer. However, they could easily be used up if supplied in lesser amounts or pose a risk of pollution to groundwater and receiving waters if provided in larger amounts. Slowrelease fertilizers provide an alternative way of supplying nutrients in such a way that required levels of nutrients are released to leach into active biodegradation sites to enhance and prolong biodegradation process for effective contaminant removal efficiency. Slow-release fertilizers are credible means of delivering essential macro and micronutrients (Giroto et al., 2017). The application of a slow-release fertilizer is considered to be a promising means to improve the utilization of macronutrients as they are designed to release nutrients gradually in order to coincide with the nutrient requirement of microorganisms for optimal biodegradation. This technique has been successfully tested in studies involving sustainable urban drainage system (SuDS) devices

such as permeable pavements (Newman et al., 2011; Nnadi et al., 2015; Mbanaso et al., 2020) and filter drains (Theophilus et al., 2018). Furthermore, attempts have been made to incorporate slow-release fertilizer granules into woven geotextile materials for application in SuDS devices (Newman et al., 2015). In either of these cases, there is an issue of lack of control of nutrient availability once they are introduced in the field, hence the need to optimise nutrient delivery efficiency. Another challenge is to make the nutrient application more 'sustainable' by using sustainable, environmentally-beneficial approaches that deliver the same benefits but through organic sources.

Hence, an alternative approach to consider is the application of Struvite (Magnesium Ammonium Phosphate, (MgNH₄PO₄ .6H₂O)) which has been identified as a sustainable source of nutrient with the benefit of availability as it is a by-product of public water treatment works where it poses a high risk to integrity and efficiency of public piping systems. Struvite has low impurities, odour and pathogens, and can easily be applied at a low cost. It is efficient in a wide range of soils and different pH levels (Tao et al., 2015), making it easily applicable in the bioremediation of oil-polluted soils. Although struvite is moderately soluble in water, Szymańska et al. (2020) demonstrated that it is more effective as fertilizer than commercial ammonium phosphate as it releases nutrients both in the first and second year after fertilisation thus, its yield potential is maintained in the second growing period following the application. In a recent study, Theophilus et al. (2018) concluded that struvite granules have the potential application in biodegradationdependent systems as a source of nutrients necessary for the optimization of the process and showed that the application of struvite can significantly enhance biodegradation rates. While there are reports of application of struvite in areas such as agriculture, and proposed application in systems such as filter drains as reported by Theophilus et al. (2018), there is no known study of potential application of struvite in sustainable drainage devices. The aim of this study was to investigate the impact of slow-release fertilizer and struvite on the biodegradation rate and efficiency of diesel-contaminated soils with a view to determining whether struvite can be applied in the biodegradation of oilcontaminated soils as an alternative, low cost and sustainable approach to use of slow-release fertilizers for such applications.

2. Materials and methods

2.1. Experimental set-up

A set of twelve test models were used for this experimental study based on previous research (Newman et al., 2015). Out of these models, three were used as control, three for osmocote, three for struvite, and three for aged diesel oil-contaminated soil which was used to investigate the impact of nutrients on the ongoing biodegradation process as shown in Table 1. Nine models were configured by embedding 22 kg of topsoil within a strong polypropylene plastic box of packaged dimension 39 cm \times 28 cm \times 28 cm. A plastic tap was installed adjacent to the base while the boxes were covered with polypropylene lids of dimensions 39 cm \times 28 cm. The topsoil used in this experiment was homogenised and characterized before the start of the experiment by determining elemental composition, pH, and electrical conductivity (Alrumman et al., 2015).

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Test models showing treatments and control
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Test Models and Treatments	Acronym	Replicate
Osmocote + Diesel	OD	3
Struvite + Diesel	SD	3
Control (Diesel only)	D	3
Aged Diesel-Contaminated Soil + Osmocote	AOD	3
Aged Diesel-Contaminated Soil + Struvite	ASD	3
Control (Aged Diesel-Contaminated Soil Only)	AD	3

2.2. Rainfall simulation

Rainfall was simulated to replicate a 'rain event', which is typically considered by most researchers in this field to require a minimum of 13 mm of rainfall (Brownstein, 1998; Newman et al., 2006; Mbanaso et al., 2016). The simulation of rain event was accomplished using a perforated tray simulator suspended over each of the test models. Approximately 500 mL of deionized water was discharged onto the rain simulator and allowed to trickle under gravity at 100 mL per minute over 5 min. Rainfall was simulated every week and sample collection was carried out at an interval of 1 h from when the rainfall event had ended.

2.3. Oil application

Diesel oil was the hydrocarbon used as the contaminant for this study. Hydrocarbon pollution was simulated by dosing diesel oil onto the surface of each test model at the rate of $178 \text{ mg m}^{-2} \text{ week}^{-1}$ based on the derived oil loading of 9.27 g m⁻² year⁻¹ (Bond, 1999). This was achieved by randomly adding 1.84 mL of diesel oil onto each of the models using a 6-mL calibrated syringe (Nnadi, 2009) before the start of each rainfall simulation.

2.4. Nutrient application

Osmocote (supplied by Wilkinsons, UK) and struvite (supplied by Ostara Nutrient Recovery Technologies Inc., Canada) were the two nutrients applied in this study and these were applied as a one-off treatment. The elemental constituent of Osmocote fertilizer has been determined and reported in previous studies (Bond et al., 1999). The struvite used in this study was characterized (weight, electrical conductivity, and elemental composition) and reported in earlier studies (Theophilus et al., 2018). A typical physical and chemical composition of struvite and comparison of nutrient release from struvite to that of NPK was reported in Latifian et al. (2012). These nutrients were applied by spreading the granules onto the surface of each test rig at a one-off rate of 17 g m⁻² (Nnadi, 2009).

2.5. Microbial analysis and enumeration

Microbial analysis was carried out to determine the rate of biodegradation and the effect of the applied nutrients (Osmcote and struvite) on the establishment of biofilms. The microbial analysis was carried out by direct plate count method while, an automated colony counting and zone measurement instrument, the Protocol-2 scientific counter (Manufactured by Synoptics Group) was used for the enumeration of the bacterial and fungal populations (Mbanaso et al., 2013; Mbanaso et al., 2020).

2.6. Microbial activity (gas monitoring)

Previous researchers have used carbon dioxide (CO₂) release as an indicator of the extent of organic carbon mineralization in soils and pavements (Bond, 1999; Mbanaso et al., 2013; Atlas, 1991; Coupe et al., 2003). In this experiment, the extent of microbial activity was monitored using an infrared gas analyzer (IRGA) for measuring CO₂ concentration supplied by Shawcity Limited, UK (Mbanaso et al., 2016).

2.7. Elemental analysis

The elemental analysis was conducted to determine the concentrations of the following elements (Al, Ca, Cd, Cu, Fe, K, Mg, Na, P, Pb, S, Zn) using Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES), Optima 5300DV© supplied by Perkin Elmer, USA (Newman et al., 2011). Equipment calibration was achieved using analytical grade standards from a 1000 mg/L stock concentration supplied by Fisher Scientific, UK.

2.8. Total petroleum hydrocarbon (TPH) analysis

Effluents from test models were analysed for TPH by infrared spectroscopy using Horiba OCMA 310 oil analyzer as reported in previous studies [Nnadi et al., 2015; Theophilus et al., 2018; Mbanaso et al., 2013).

2.9. pH and electrical conductivity

An expression of the acidity or alkalinity of the effluent solution was determined using a Fisher brand Hydrus 300 pH meter manufactured by Orion Research Inc., USA (Mbanaso et al., 2016), while the electrical conductivity was determined using a PTI-8 digital meter (Nnadi, 2009).

2.10. Statistical analysis

Descriptive statistics was employed and mean values of measured parameters were used for the results presented in this paper. In addition, the variability of data was determined by using Error bars.

3. Results

3.1. Characterization of soil and struvite

The results of elemental analysis, pH, moisture content, and electrical conductivity of soil and struvite used in this experiment are shown in Table 2.

Table 2 shows that the soil is slightly acidic at 6.79 pH value while that of struvite is 7.86. However, both have the same cadmium content (0.0006 mg/ L).

3.2. Microbiology analysis

The results of the microbial analysis (bacteria and fungi) are as shown in Figs. 1 and 2 respectively. The background analysis showed that at the beginning of the study, the total indigenous aerobic heterotrophic bacterial growth in effluent from the test models was relatively at the same level. Before the contaminant addition, a significant difference was observed as bacterial growth increased in effluents from control test models while growth decreased in aged oil-contaminated test models as shown.

A significant increase in bacteria growth was observed after the application of Struvite and Osmocote to rigs particularly those AOD models as they maintained a significant increase in growth progressively until the end of the study.

Results of fungi growth showed a similar response as that of bacteria as effluents from test models with Osmocote maintained a relatively

Table 2

Physiochemical characteristics of soil and struvite used in this study.

Property	Values		
	Soil	Struvite	
Moisture content (%)	18.28	-	
рН	6.79	7.86	
EC _w (μS/cm)	731	989.333	
Al mg/L	0.275	0.019	
Ca mg/L	120.35	0.042	
Cd mg/L	0.0006	0.0006	
Cu mg/L	0.140	0.0223	
Fe mg/L	0.226	0.045	
K mg/ L	6.920	9.07	
Mg mg/L	4.844	37.99	
Na mg/L	4.087	0.521	
P mg/L	1.010	131.14	
Pb mg/L	0.0213	0.001	
S mg/L	4.483	0.171	
Zn mg/L	0.108	0.004	



Fig. 1. Mean bacterial count in effluent from rigs. Sampling events 1–4 represent background study while sampling event 5 represents the start of contaminants addition followed by weekly contaminants addition. The error bars represent the mean \pm S.E. of three replicates.



Fungi Sampling Events

Fig. 2. Mean fungi count in effluent from rigs. Sampling events 1–4 represent background study while sampling event 5 represents the start of contaminants addition followed by weekly contaminants addition. The error bars represent the mean \pm S.E. of three replicates.

high growth throughout. However, there was a comparatively higher population density before the addition of the contaminant as the trend afterward showed a relative decline.

For AOD test models, results showed that bacterial growth increased progressively while those of ASD models did not show a consistent growth pattern. Fungi growth, on the other hand, displayed a similar pattern as all test rigs recorded decreasing and inconsistent growth trends whereas, those of AOD test models showed a relatively higher fungi growth.

3.3. Gas analysis (CO₂ evolution)

The result of CO_2 evolution from all test models is as shown in Fig. 3. The rate of hydrocarbon biodegradation in the test models showed a significant increase in CO_2 production after the application of contaminant (diesel oil) and nutrients (Osmocote and Struvite). A higher microbial respiratory activity was observed in test models with applied Osmocote as a nutrient than those to which struvite was applied as a nutrient source.

A similar response in the microbial respiratory activities was observed in the aged oil-contaminated soil test models. Microbial activity was very low during the background study, however, this changed after the application of nutrients to the test models as microbial activities increased in response to nutrients application especially, in rigs to which osmocote was applied.

3.4. pH

The result of the pH of effluent from test models showed that they were within the pH range of 7.1-8.20 throughout the study (Fig. 4).



Fig. 3. Mean microbial respiration from test models. Sampling events 1–4 represent background study while sampling event 5 represents the start of contaminants addition followed by weekly contaminants addition. The error bars represent the mean \pm S.E. of three replicates.



Fig. 4. pH reading of effluents from test models. Sampling events 1–4 represent background study while sampling event 5 represent start of contaminants addition followed by weekly contaminants addition. The error bars represent the mean \pm S.E. of three replicates.

However, results from the post-contaminant and nutrients addition phase of the study were generally higher than those of the precontaminant/nutrients addition era.

3.5. Electrical conductivity (EC_w)

Electrical Conductivity (ECw) of effluents from test models showed relatively higher levels of EC_w in all test rigs before treatments but, declined significantly after the application of contaminant and nutrients to the test models (Fig. 5).

3.6. Elements in effluent analysis

3.6.1. Aluminium (Al)

The aluminium concentration in the effluent from the test models showed high concentrations in effluents from aged oil-contaminated soil samples than those with freshly contaminated soils as presented in Table 3.

3.6.2. Calcium (Ca)

Calcium concentration was relatively high in all test models. However, a significant decrease was observed after the background study as calcium concentration in all test models decreased significantly except in aged oil-contaminated samples (excluding struvite models) as shown in Table 3.

3.6.3. Cadmium (Cd)

The result of cadmium concentration in effluent from the test rigs as shown in Table 3 shows that effluent from all the test rigs had no cadmium content or, their concentrations were below the limit of detection.

3.6.4. Copper (Cu)

Table three (3) showed that effluent from aged oil-contaminated soil with added nutrients had a relatively higher concentration of copper while effluents from other models were relatively low throughout the study.



Fig. 5. Electrical Conductivity (ECw) of effluents from test models. Sampling events 1–4 represent background study while sampling event 5 represent start of contaminants addition followed by weekly contaminants addition. The error bars represent the mean \pm S.E. of three replicates.

3.6.5. Iron (Fe)

Results of iron analysis as shown in Table 3 indicated low iron concentrations in effluents from all test models except the aged oil-contaminated soils (with and without nutrients).

3.6.6. Potassium (K)

Potassium concentration in effluent from test models as shown in Table 3 was relatively high in aged oil-contaminated test rigs with Osmocote but low in oil-contaminated soil with osmocote. The result showed sufficient concentration of potassium in all tests models.

3.6.7. Magnesium (Mg)

Results of elemental analysis showed that effluent from OD, SD and D test models had the highest concentrations of magnesium as shown in Table 3 while, effluents from AOD, ASD and AD had relative low magnesium concentrations.

3.6.8. Sodium (Na)

Relatively high concentration of sodium was observed in effluent from test models in the same pattern as recorded for potassium and magnesium.

3.6.9. Phosphorus (P)

Result of elemental analysis of effluent from test models as shown in Table 3 indicated that after the background study, phosphorus concentration increased in OD and AOD test models while other test models had relatively low phosphorus concentration.

3.6.10. Lead (Pb)

Lead concentration in effluent from OD, SD and D test models was low as shown in Table 3 however, relatively high concentration was observed in effluent from AOD, ASD and AD test models.

3.6.11. Sulphur (S)

Sulphur concentration in effluents from OD, SD and D test models was relatively high during the background study, on the other hand, a decline in the concentration was recorded after the application of contaminant and nutrients as shown in Table 3. However, the reverse was the case in AOD, ASD and AD test models.

3.6.12. Zinc (Zn)

Result of the elemental analysis of zinc in the effluent from all test models showed similar pattern as recorded for sulphur as shown in

Table 3.

3.7. Total Petroleum Hydrocarbon (TPH)

The result of Total Petroleum Hydrocarbon analysis in effluent from test models, as presented in Fig. 4, showed low concentration of TPH in effluents from non-aged models, but relatively high concentration levels in effluents from aged oil-contaminated test models.

4. Discussion

4.1. Effect of osmocote and struvite on bacterial growth

Microorganisms are necessary for maintaining natural habitats. The ability of microbes to use petroleum hydrocarbon as a source of carbon and energy in order to survive in polluted environments is an important role that could be harnessed and optimised for biodegradation of oilcontaminated soils. However, the biodegradation rate needs to be optimised in order to ensure that efficient removal of contaminants is achieved. Nutrient (NPK) deficiency is one of the factors that can limit biodegradation by microbes. Application of fertilizers to augment and boost nutrient supply is one of the means of ensuring continuous and essential nutrient supply to kick start, maintain and optimise microbial removal of contaminants. Hence, fertilizers (Osmocote and Struvite) containing essential nutrients were applied in this study to investigate and compare their impact on the enhancement of biodegradation of diesel.

Results in Fig. 1 showed higher bacterial growth in AOD test rigs in background samples in Weeks 1 & 4 relative to other treatments. However, the control samples (D) recorded higher bacterial growths in weeks 2 & 3, followed by SD and OD test rigs. Similarly, Oscomote containing test rigs showed higher fungal and bacterial growth respectively, relative to other treatments in Figs. 2 and aged oil-contaminated models. Theophilus et al. (2018) showed that nutrient application encouraged microbial activities and enhanced biodegradation rates, but observed differences in increase based on the type of nutrient applied. Also, Robles-Aguilar et al. (2020) observed significant dissimilarities in the growth of microbes between treatments with the addition of struvite and slow-release fertilizer. However, the figures indicate a decrease in bacteria growth after the application of nutrients and contaminants, which, may be linked to various reasons. Contaminant application might have led to a sudden reduction in bacterial population and consequent reduction in bacterial activities. The release of the contaminant in the

Table 3

Result of the elemental analysis of rig effluent showing mean concentrations from different treatments.

Treatments Element		Pre- contaminants addition phase (mg ^{- L})	Post- contaminants addition phase (mg ^{- L})	FAO* limits (mg⁻ ^L)	USEPA** limits (mg ^{- L})
		Maximum	Maximum		
OD SD	Al	0.26	0.11	5.0	5.0
D		0.12	0.72		
AOD		2.61	0.75		
ASD		1.18	2.30		
AD		0.23	3.06		
OD	Ca	201.20	145.70	-	-
SD		198.8	161.33		
AOD		178.03	111.68		
ASD		94.80	75.53		
AD		53.41	88.88		
OD	Cd	0.00	0.00	0.01	0.01
SD		0.00	0.00		
D		0.00	0.00		
AOD		0.00	0.00		
AD		0.00	0.00		
OD	Cu	0.06	0.06	0.2	0.2
SD		0.06	0.15		
D		0.05	0.13		
AOD		0.25	0.14		
ASD		0.23	0.25		
AD OD	Fe	0.08	0.22	5.0	5.0
SD	10	0.11	0.15	010	010
D		0.14	0.48		
AOD		1.67	0.47		
ASD		0.84	0.76		
AD	17	0.14	1.86		
SD SD	ĸ	37.80	30.79	-	-
D		30.42	23.81		
AOD		32.76	46.66		
ASD		20.20	20.74		
AD		21.06	37.52		
OD	Mg	27.51	23.56	-	-
3D D		26 71	20.09		
AOD		14.27	15.09		
ASD		13.56	7.15		
AD		6.41	10.74		
OD	Na	108.18	75.51	-	-
SD		108.80	94.38		
AOD		68.64	88 15		
ASD		110.30	88.08		
AD		78.86	57.20		
OD	Р	0.89	2.90	-	-
SD		0.50	1.69		
		1.14	0.35		
ASD		0.70	0.56		
AD		0.54	0.95		
OD	Pb	0.02	0.04	5.0	5.0
SD		0.02	0.01		
D		0.05	0.01		
AUD		0.04	0.10		
AD		0.03	0.04		
OD	S	102.60	47.80	_	_
SD		128.63	87.60		
D		89.83	37.11		
AOD		14.18	17.22		
ASD		20.14	25.61 15.29		
AD OD	Zn	0.01	0.03	2.0	2.0
SD		0.01	0.07		

Table 3 (continued)

Treatments Element		Pre- contaminants addition phase (mg ^{- L})	Post- contaminants addition phase (mg ^{- L})	FAO* limits (mg ⁻ ^L)	USEPA** limits (mg ^{- L})
		Maximum	Maximum		
D		0.05	0.06		
AOD		0.03	0.06		
ASD		0.03	0.05		
AD		0.01	0.05		

* (FAO (2008).

** (US EPA (2012).

environment, as well as rainfall simulation, might have led to an initial blockade of the surface area with oil-contaminated water which then prevents the flow of oxygen thus, causing an anaerobic condition and leading to the death of bacteria unable to strive in such conditions (Atlas, 1991). Also, some petroleum hydrocarbon degraders might have died as a result of limited nutrients necessary for growth due to the slow and controlled release of nutrients by applied fertilizers (Sarkar et al., 2005). Furthermore higher turbidity of used oils has been observed to inhibit biofilm formation, microbial growth, and functions (Raju et al., 2017). Abioye et al. (2012) also reported microbial growth inhibition properties of oil.

Substantial growth was observed in AOD test rigs post-contaminant addition compared to rigs to which struvite was added. This may be attributed to the slower mechanism of nutrient release by struvite, considering that nutrients are not readily available when fertilizer is introduced in an acidic environment (Emilsson et al., 2007). Another possible explanation of the increase observed in Osmocote treated test rigs could be attributed to anaerobic conditions created as a result of oil application which probably led to denitrification of Nitrogen in Osmocote slow-release fertilizer. However, this condition could be dependent on the volume of oil present and the rate of release of Nitrogen from Osmocote relative to other available nutrients as well as the rate of denitrification, soil characteristics, and environmental factors.

4.2. The effect of osmocote and struvite on fungi growth

The fungi growth from the results in Fig. 2 showed that test models contained a relatively large population of fungi though a significant decrease was observed after the application of contaminant and fertilizers but just like the bacteria growth, the highest fungi growth was seen in effluents from test rigs containing osmocote. Similarly, previous studies observed that the application of fertilizers brought about an increase in the number of fungi spores and biomass (Qin et al., 2015; Juntahum et al., 2020).

The decrease in fungi growth following the application of contaminant and fertilizers indicates that the concentration of contaminant applied to the system was too high for fungi to survive in such an environment and the simulated rainfall also had an impact on the growth (Ros et al., 2014). Subsequent fungi growth was observed in effluent from all test rigs after the initial shock from the addition of contaminant, which suggests that fungi present in the system adapted to the environmental conditions, and with the aid of nutrients released to the environment by applied fertilizers, they were able to survive and grow (Liu et al., 2022; Boopathy, 2000).

Similar to the trend observed with bacteria, fungi growth was also relatively higher in aged oil-contaminated test models containing osmocote. Similarly, there was a relative reduction in fungi growth postcontaminant and nutrients addition. Many factors besides the concentration of the contaminant might have played a role in limiting the growth of fungi, such as the temperature of the environment, availability of oxygen, and nutrient levels (Stanaszek-Tomal, 2020).

4.3. Comparative analysis of microbial respiration

Microbial respiration analysis was carried out to determine the activities of microbes in the system as well as the rate of biodegradation. The results of CO₂ production (Figs. 5) indicate that microbial activities increased in all test models after the application of contaminant. In relation to microbial growth, it was observed that high microbial respiratory activities were observed in test models containing osmocote due to the highest CO₂ evolution. Microbial respiratory activities can be linked to microbial growth or population therefore, the higher the microbial growth, the higher the rate of biodegradation resulting in CO₂ production (Singh and Chandra, 2014). Clearly, the oil provided a source of carbon in the system and the microbes responded well to it in the presence of nutrients. In a recent study, Ward et al. (2017), demonstrated that there was a negative correlation between soil respiration and increasing levels of nitrogen fertilizer application but, was positively correlated with soil pH, although, the possible mechanisms behind this are still unclear. The rate of petroleum hydrocarbons biodegradation can be influenced by the availability of hydrocarbondegrading microbes (Singh and Chandra, 2014). However, an increase or decrease in the rate of biodegradation can also be as a result of other factors like the physical and chemical composition of the petroleum hydrocarbon, temperature, soil characteristics, and oxygen (Das and Chandran, 2011).

4.4. The effect of osmocote and struvite on nutrient availability

The concentration of elements in effluent does not only influence the rate of biodegradation but can be harmful to environmental and human health thus, an analysis of elemental concentration in effluents was carried out. The result of this analysis was discussed in comparison with some international standards such as USEPA Guidelines for Water Reuse, FAO's Threshold Levels of Trace Elements for Crop Production, USEPA Maximum Contaminant Level for drinking and irrigation water, etc. However, it should be noted that these effluents were not meant for drinking water, but should be seen as highlighting the potential risk that hydrocarbon-polluted soil poses to groundwater.

The results of aluminium concentration in the effluent as shown in Table 3, indicates that effluent from test models had a high concentration of aluminium compared to the maximum contaminant level of 0.015 mg/L in drinking water (US EPA, 2012; US EPA, 2014). However, the concentration levels are far lower than USEPA and FAO's limits of 5 mg/L.

Results (Table 3) showed a low concentration of calcium in effluent from aged oil-contaminated test rigs while a relatively sharp decline of calcium levels was observed in effluents from other test models after the application of contaminant and nutrients to the test models. Maximum contaminant level for calcium was not available in the literature, which may be traced to its importance in the environment, and human body functions. Nevertheless, the reaction of calcium with other metals forming calcium hydroxide Ca(OH), calcium sulfate CaSO₄.2H₂O and many others can be toxic to the environment and human health (Csuros, 1994) and for this reason, an upper limit of 75 mg/L can be used as a guide (Csuros, 1994). High calcium availability in the system can also limit the availability of nutrients depending on other factors such as pH. Conversely, Calcium compounds can be applied as chelating agents for remediation of polluted soils (Wuana and Okieimen, 2011).

Cadmium concentration in effluent from test models indicates that there was little or no cadmium in the effluent from all test models. A trace amount of cadmium below the maximum contaminant level of 0.005 mg/L may have been possible but, was all below the limit of detection.

High potassium concentration was observed throughout the study as shown in the result (Table 3). It was observed that effluents from test rigs containing osmocote had a higher concentration of potassium compared to effluents from test rigs containing struvite. Similar to calcium, no maximum level was found for potassium since it is an essential element that maintains environmental and human functions.

From Table 3, the result of phosphorus concentration in effluent from test models suggests that effluents had a relatively high concentration of phosphorus. However, a significant increase was observed in effluent from test rigs to which contaminant and osmocote were applied. Phosphorus availability is important for optimal biodegradation hence, the supply of phosphorus in the system by the added nutrient sources is vital for the biodegradation of hydrocarbon by microbes.

A low concentration of zinc was observed in effluent from test models as presented in Table 3 compared to the maximum contaminant level of 5 mg^{-L} (Salavato et al., 2003). An increase in concentration was higher post-contaminant addition phase in all test models. It might have been due to the presence of Zn in oil which is usually added as a component of antiwear formulations or additives.

4.5. pH

The result of pH analysis indicates that effluent from test models was relatively neutral or a little alkaline considering the values were between 7 and 8.20. This denotes that the pH level in the systems was suitable for microbial growth since most bacteria are neutrophiles, meaning they grow optimally at a pH within one or two pH units of the neutral pH of 7 (OpenStax (2016). pH is an important parameter that does not only determine the kind of plants, animals, and microbes that can be found in a given environment, it also determines the availability of nutrients as well (Peterson, 1999). In determining the appropriate use of water, pH levels of effluent from test models fall within the range of 6.5–8.5 which is good for irrigation, surface, and drinking water (Salavato et al., 2003).

4.6. Electrical conductivity (ECw)

The Electrical Conductivity (ECw) of effluent from test models indicates that it decreased over time as high electrical conductivity was observed at the beginning of the study in effluent from all test models except effluents from aged oil-contaminated models. Electrical Conductivity (ECw) is an important parameter used in determining the appropriate use of water. The acceptable electrical conductivity for irrigation falls within the range of 750 to 2000 μ S/cm, indicating that effluents from test rigs during the background study are suitable for irrigation but the decrease in ECw after the application of contaminant and slow-release fertilizers overtime highlights that effluent below 750 μ S/cm cannot be used for irrigation (Tutmez et al., 2006). However, effluents are suitable for release into the environment as the ECw meets the acceptable value for streams and freshwater which ranges from 50 to 1500 μ S/cm (Mosneag et al., 2014).

4.7. Total Petroleum Hydrocarbon (TPH)

Results of TPH presented in Fig. 6 showed that effluents from test models had low TPH concentration levels despite the simulation of a heavy oil spill. A significant drop in TPH concentration was observed during the background study but an increase was observed in effluent from aged oil-contaminated test models containing nutrients, which could have resulted from the test rigs' inability to retain contaminant, or reduced microbial activities in the test models. However, TPH levels reduced throughout the study after the addition of contaminant and nutrients, which could be an indication of soil retention and biodegradation. Throughout the study, the TPH concentration in effluents from all the test models was below 10 mg $^{-\,\rm L}$ except on a particular sampling event where a test model (AOD treatment) recorded a concentration of 16.9 mg/L (outlier). Apart from this singular incident which might have been due to sampling or analytical error, all others were lower than the recommended maximum effluent contaminant level of 10 mg/L suggested by the US National Pollutant Discharge Elimination System



Fig. 6. Effluent Total Petroleum Hydrocarbon (TPH) content from test models. Sampling events 1–4 represent background study while sampling event 5 represents the start of contaminants addition followed by weekly contaminants addition. The error bars represent the mean \pm S.E. of three replicates.

(NPDES) permit (McLaughlin et al., 2020). This suggests no potential impact arising from the reuse of effluent by downstream users and ecosystem services.

5. Conclusions

This study investigated the impact of slow-release fertilizer and struvite on the biodegradation rate and efficiency of dieselcontaminated soils with a view to determining whether struvite can be applicable and effective in biodegradation of oil-contaminated soils as an alternative, low cost and sustainable approach to use of slow-release fertilizers for such applications.

Water quality analysis was also carried out to determine and highlight the potential risk of hydrocarbon pollution by assessing the suitability of effluents for reuse applications such as irrigation, recreational activities, surface water, and percolation into groundwater. It must be noted, that from the perspective of achieving optimal biodegradation of hydrocarbon in soils, availability of nutrients in effluents is desirable as this is much needed for optimal biodegradation and thus, the degrees of nutrient availability demonstrates the efficiency of nutrient sources investigated in this study. Also, relatively higher levels of aluminium were detected in effluent from struvite treated test rigs, though the levels were far lower than USEPA and FAO's limits of 5 mg/L, but care should be taken in the application of effluent in fertigation by considering the aluminium requirement of plants before application and it is not recommended as drinking water for animals without treatment. Furthermore, treatment or dilution is required before effluent discharge into natural waters according to guidelines.

Microbial analysis was conducted to determine and compare the rate of biodegradation and the effect of the applied nutrients (Osmcote and struvite) on the establishment of biofilms. The results of this analysis revealed that Osmocote plus controlled-release fertilizer is more effective in enhancing microbial growth in diesel polluted soils compared to Struvite. However, this may have been due to the relatively slow release of nutrients contained in struvite fertilizer due to its structure and solubility, especially in the presence of diesel oil and, highlights that struvite (which is mainly composed of magnesium ammonium phosphate and calcium carbon-apatite) is not as nutrient-rich as osmocote, which is formulated with essential elements (NPK) necessary for plant growth and in this case, biodegradation. A similar observation was made by Theophilus et al. (2018). It should be noted that struvite accumulation in public water pipe works is an issue of environmental concern hence, any meaningful utilization and application of this "waste" in bioremediation of contaminated soils would offer a sustainable and lowcost means of disposal of what is hitherto an environmental problem. However, it may require augmenting the deficient nutrients to enhance the effectiveness of struvite as an oil biodegradation enhancer. This may be accomplished by incorporating naturally occurring nutrient sources such as rock phosphate, kelp, and bedrock nitrogen for sustainability.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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