Abstract

The adsorption of Total Hydrocarbon Content (THC) from petroleum products contaminated soil using wood charcoal blocks and sawdust was investigated in this study. 130kg of the sieved topsoil was contaminated with 5L of spent motor engine oil, 5L of kerosene, 5L of petrol and 5L of diesel, and left to stabilize for two weeks; after which it was thoroughly mixed together. The thoroughly mixed soil was filled into plastic buckets (7kg per bucket) and arranged in five rows. Row A contained the un-amended contaminated soil sample (control), rows B and C contained non-replacement amendments(s) (wood charcoal blocks and sawdust), while rows D and E contained replacement amendment(s) which was/were replaced with fresh ones after every ten experimental days. Laboratory test results conducted on the soil samples at the end of the 40-day experimental period showed a general declined in the THC concentration. It was observed that the THC concentration, in general, declined by about 90% in the remediated contaminated soil samples, as against about 18.71% decline recorded in the control. Furthermore, the contaminated soil samples, with replacement amendments had better results after the experimental period (40 days) with a mean value of 1196 mg kg⁻¹ in THC residuum; when compared with the results from the non-replacement amendments with a mean value of 3269 mg kg⁻¹ THC concentration residual. These results show that charcoal blocks and sawdust significantly degraded the THC concentration in the contaminated soil samples. Data obtained from this study would provide useful information in the utilization of charcoal block and sawdust in bioremediation techniques, for the cleaning up of petroleum products/crude oil contaminated sites.

Keywords: Adsorption; Charcoal block; Remediation; Sawdust; THC

Introduction

Soil is a loose and heterogeneous thin layer of organic and inorganic materials that covers a large expanse of the earth’s surface. Soil is mainly made of two constituents: namely the organic and inorganic parts. The organic part is derived from the decayed plants and animal materials, which is concentrated mostly in the topsoil. The inorganic part, which consists of the subsoil and rock fragments, was formed over thousands of years by physical, biological and/or chemical weathering of the bedrock [1-3]. Soil helps in the breakdown and immobilization of materials (fertilizers, chemicals, waste products, etc.) added to its surface. The soil is a complex environmental system, which employs (interactions) biological, chemical, and physical processes, to degrade harmful materials into less dangerous materials [4]. These interactions and the degradation process may take a short duration (few weeks), or a longer duration (years) to achieve its aim. Contaminants intentionally or unintentionally discharged to the soil, mostly the surface soil or of the topsoil is one of the greatest environmental problems globally. This is because soils play a vital role in food production within the ecosystem [4,5]. Soil pollution is usually caused by the presence of synthesized chemicals or other alteration in the natural soil environment. Soil contamination typically occurs through the rupture of underground storage tanks, application of pesticides, and the percolation of contaminated surface water into subsurface strata, oil and fuel dumping, etc. [2].

Oil exploration and exploitation in Nigeria has taken an aggressively new dimension in the last decade, with a more focus on Northern and Central Nigeria (Lake Chad basin and Benue trough basin). According to the Organization of Petroleum Exporting Countries (OPEC), Nigeria’s oil and gas sector accounts for about 10 per cent of its gross domestic product (GDP), while petroleum exports revenue, accounts for about 86 per cent of...
Nigeria total exports revenue [6]. Currently Nigeria has the largest proven natural gas reserves (5,675 billion cu. m.) and the second largest proven crude oil reserves (36,972 million barrels) in Africa [6,7]. Crude oil spills are common occurrence in the oil rich Niger Delta region of Nigeria. The Shell Petroleum Development Company of Nigeria Limited (SPDC), the major oil exploring company operating in Nigeria had attributed about 80% of oil spill incidence to crude oil theft and sabotage of facilities, as well as illegal refining activities [8]. The largest crude oil spill that had occurred in Nigerian history was the SPDC Forcados Terminal tank failure in 1978, which spilled out about 580,000 barrels of crude oil into the environment [9]. According to statistics from the International Tanker Owners Pollution Federation (ITOPF), [10], a total volume of 1,000 tonnes of crude oil was spilled into the environment worldwide, which was the lowest annual spillage volume recorded globally in the last five decades. Contamination of the soil and water bodies with either crude oil or refined petroleum products has had adverse effects on plants, animals, fishes and soil/water microorganisms [11]. According to Scott [12], contamination of soil by petroleum/petroleum products can occurs through extraction, exploration and processing accidents, spillages, illegal refining methods, and indiscriminate disposal of the waste products [11].

According to previous studies, there are significant changes in the soil's physical characteristics, as well as chemical and microbiological properties after being contaminated with crude oil or petroleum products [3,12]. Akpomere and Uguru [3] reported that the Lead (Pb), Nickel (Ni), Copper (Cu), cadmium (Cd) and Iron (Fe) concentrations in soil samples increased by over 100% after being contaminated with petroleum products. Udonne and Onwuma [13] observed that lubricating oil altered the soil bulk density from 1.10 to 1.15 gcm⁻³ and the soils capillarity from 8.10 to 0.04 cm h⁻¹. Likewise, Akpokodje and Uguru [14], reported that soil porosity decreased from 35% to 14%, and specific gravity from 2.34 to 1.35; while the soil Cu concentration increased from 4.892 mg kg⁻¹ to 7.729 mg kg⁻¹, and Pb concentration from <0.0001 mg kg⁻¹ to 1.128 mg kg⁻¹ after soil samples were contaminated with petroleum products. Hydrocarbon pollutants cause disruptions of the natural equilibrium existing between the living things and their natural environment; and belong to the family of carcinogens and neurotoxic organic pollutants [15].

Remediation of crude oil or petroleum products contaminated polluted soils is an important task to all oil exploring companies. Remediation of polluted site can be accomplished through physical, chemical, thermal, and biological/natural methods. Remediation of petroleum polluted soils and water bodies could be achieved by either physical/mechanical (excavation, burning), chemical (detergent, surfactant, degreaser), plants (phytoremediation) and biological (bioremediation) methods [14,16]. Bioremediation is a technique that uses the microorganisms (plants inclusive) to detoxify or degrade contaminants from the environment; it is a remediation method that offers a green technology solution to the problem of environmental degradation [17]. Bioremediation relies on microbial enzymatic activities to degrade contaminants from the ecosystem [18]. Within the past two decades, many natural remediation techniques have been developed by researchers to provide more economical, effective and environmentally friendly methods of cleaning up polluted sites [11,19].

The use of plant-based adsorbent in the cleaning-up of oil spill areas has many advantages due to its simplicity of approach, cost-effectiveness, and biodegradable nature, which leaves no permanent residue [20]. Abiye et al., [21] reported that melon shells have the potential of degrading the crude oil content in crude oil contaminated soils by about 75% within a 28-day experimental period. According to Aslam and Ayush [22], charcoal powder is an efficient adsorbent, absorbing about 99 % crude oil from crude oil contaminated seawater. Vasilyeva et al. [23] reported that activated carbon is a good adsorbent for organic contaminants; probably due to its hydrophobicity and microporous structural makeup. According to Rhodes et al. [24], the biodegradation of phenanthrene in soil by activated charcoal was highly influenced by the quantity of the charcoal used during the remediation process, and the experimental period. Likewise, Bushnaf et al. [25] observed that biochar (2% concentration by weight) was able to increase the biodegradation rate of polycyclic aromatic hydrocarbon (PAHs) contaminants in the soils to a significant level.

However, from the literature reviewed, there was no evidence of the use of wood charcoal block and sawdust amendment in remediating petroleum products contaminated indigenous soils. Therefore, in this study, the bioremediation potency of wood charcoal and sawdust on petroleum products contaminated soils was investigated. Likewise, the sorption potential of the charcoal blocks was measured. To the best of our knowledge, this is the first extensive research on bioremediation of petroleum products using replaceable combination of wood charcoal blocks and sawdust.

Materials and Methods

Petroleum products

Spent motor engine oil was purchased from a motor mechanic workshop located at Ozoro, Delta State, Nigeria; while the petrol, diesel and kerosene were obtained from a filling station located at Ozoro, Delta State, Nigeria.

Wood charcoal blocks

The wood charcoal blocks were purchased from a located market located at Ozoro, Delta State, Nigeria. Citing the International Union of Pure and Applied Chemistry (IUPAC), wood charcoal is the char obtained by the carbonization of wood (timber) [26,27].

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Sawdust

Sawdust was obtained from a timber market located along Ozoro-Oleh road, Delta State, Nigeria. Dry sawdust contains about 60% carbon, 33% oxygen, and 1% nitrogen [20].

Topsoil

Topsoil used for this study was dug (0 - 15cm deep) from a corner of the research field station of Delta State Polytechnic, Ozoro, Nigeria.

Plant of interest

The bean (Phaseolus vulgaris L) seeds were obtained from the Department of Agricultural and Bio-environmental Engineering Technology, Delta State Polytechnic, Ozoro, Nigeria.

Methods and samples preparation

Wood charcoal blocks and sawdust

The wood charcoal blocks, and sawdust were dried with an electric laboratory oven at a temperature of 1300°C for 6h to reduce their moisture content. Inactivated charcoal block was chosen over activated charcoal block because of its relative availability, even though the later may have a better efficiency; and also because the technology involved in the production of activated charcoal, may not be easily available to Nigerian local farmers.

Topsoil

Topsoil used for this study was air-dried under ambient atmospheric temperature (34±4°C) for two weeks; after which it was sieved with a 2mm gauge stainless steel sieve, to remove all stones and roots. 130kg of the sieved soil was filled into a drum and impacted with 5L of spent motor engine oil, 5L of kerosene, 5L of petrol and 5L of diesel and left to stabilize for two weeks.

Phytotoxicity test

The seed germination rate test of the contaminated soils was carried out in accordance with United States Environmental Protection Agency (US EPA 712-C-008) recommendations [29]. This was done in order evaluate the effects of the mixture of petroleum products on the bean plants. Seven healthy bean seeds were planted directly into the soil after contamination (i.e. on the first day of remediation) failed to germinate ten days after planting (DAP).

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Laboratory tests

Soil heavy metals analysis

Some heavy metals (Nickel (Ni), Copper (Cu), Cadmium (Cd), Lead (Pb), and Iron (Fe)) concentrations in the uncontaminated and contaminated soil samples were analyzed. This was carried out to determine the concentrations of some heavy metals present in the soil samples in order to further characterize impacts on the soil and on the morphology of the bean plants. All the parameters (heavy metals) were determined by employing the standard recommended methods approved by American Public Health Association [11,31]. Results of the soil analysis are presented in Table 1.

Table 1: Heavy metals and THC concentrations of soil samples.

| Parameter       | Level                                                   | Before contamination | After contamination |
|-----------------|---------------------------------------------------------|----------------------|---------------------|
| Heavy metals    |                                                         |                      |                     |
| Nickel          | mg kg⁻¹                                                 | 4.63ᵇ                 | 13.18ᵇ              |
| Copper          | mg kg⁻¹                                                 | 2.45ᵇ                 | 10.67ᵇ              |
| Cadmium         | mg kg⁻¹                                                 | 1.47ᵇ                 | 2.74ᵇ               |
| Lead            | mg kg⁻¹                                                 | 1.28ᵇ                 | 3.82ᵇ               |
| Iron            | mg kg⁻¹                                                 | 1943ᵇ                 | 2954ᵇ               |
| THC             |                                                        | 40.56ᵇ                | 14,745ᵇ             |

Determination of Total Hydrocarbon Content (THC)

The THC of the soil samples was determined by employing approved standards recommended by ASTM D 9071B-7, as described by Akpan & Usuah [32]. Results of the soil THC analysis are presented in Table 1.

All the laboratory tests were carried out under ambient laboratory temperature. In addition, each test was done in triplicate, and the average value recorded.

Percent removal of THC

The percentage of THC adsorbed by the charcoal block during the experimental process was calculated by using equation 1.

\[
\text{Percent removal} = \frac{C_1 - C_2}{C_1} \times 100
\]

Where:

- \( C_1 \) = initial THC concentration
- \( C_2 \) = Final THC concentration

Statistical Analysis

All data gotten from this study were statistically analyzed by employing the Statistical Package for Social Statistics (SPSS version 20.0); while the means were separated using The Duncan’s New Multiple Range (DNMR) Test (P ≤0.05). In addition, the summary of the readings was plotted with Microsoft Excel 2015.

Results and Discussion

Laboratory results of the heavy metals concentrations and THC of the soil samples are presented in Table 1. The petroleum products have significant (p ≤0.05) effect on the heavy metal concentration and THC of all the soil samples, which is not contradictory to previous research results of Ameh et al. [33], Akpan and Usuah [32], and Akpokodje et al. [11]. The fairly appreciable concentration of THC (40.56 mg/kg) observed in the uncontaminated soil samples can be attributed to petroleum hydrocarbon contaminated atmospheric air; impacted through oil exploration, illegal crude oil refining, and traffic pollution [34]. Significant dose of petroleum hydrocarbons pollutant in the atmosphere will dissolve in the rainwater (since Delta State is a crude oil production hub in Nigeria), which will precipitate down during the rainy season (April to October) and subsequently infiltrate into the soil. Likewise, the area where the topsoil was collected is prone to flooding; therefore, there is a high probability that significant amounts of contaminants (hydrocarbons) will flow along with the floodwater and infiltrate into the soil.

Values are mean ± standard deviation; Rows with the same common letter (superscript) are not significantly different at (p ≤0.05).

Morphological performance of the Honey bean plant

The experimental set up for morphological analysis of the honey bean plants cultivated is shown in Figures 1 & 2. The bean plants growing in setups with replacement amendments (wood charcoal blocks and sawdust) performed better, when compared to those growing in setups with non-replacement amendments. In contrast, the few bean seeds that germinated in the un-amended soils samples died within two weeks after germination (Figure 3). This could be attributed to the heavy metals and petroleum hydrocarbons toxicity. According to Ahsan et al. [35] and [8], heavy metals and petroleum hydrocarbons are highly poisonous to plants and can lead to their dead if present in the soil in high quantity.
Figure 1: Honey bean growing in replacement amendment.

Figure 2: Honey bean growing in non-replacement amendment.

Figure 3: Bioremediation of contaminated soil using wood charcoal and sawdust amendment.
The bean plants growing in the non-replacement amendments showed signs of chlorosis in their leaves. Chlorosis in plants is usually attributed to heavy metals and petroleum hydrocarbons poisoning. According to Mohanpuria et al. [36], crops growing in soils with high concentrations of heavy metals (e.g. Cd) usually show symptoms of chlorosis and growth inhibition, resulting in death if such soils are not remediated on time.

**Bioremediation (Biodegrading) of THC of the soil samples**

With reference to the laboratory results, the THC concentration of all the soil samples (both amended and un-amended) generally declined during the experimental period. For all cases, it was observed that the decline in THC concentration was higher (about 90%) in the contaminated soil samples inserted with charcoal blocks and sawdust, when compared to results (18.71%) obtained from the un-amended contaminated soil samples (control). This shows that charcoal block and sawdust (individually) significantly degraded the THC concentration in the contaminated soil samples; however, the combination of the wood charcoal block and sawdust gave a better result. Previous research [7,37] had shown that dry sawdust has a good sorption ability, having the ability to absorb potential contaminants (mostly petroleum products) from contaminated soils samples. Apart from the sorption quality of the sawdust, during the biodegradation period, the decomposition of the sawdust also provides nutrients (mostly nitrogen) thus creating an enabling environment for the soil macro/micro-organisms to thrive. Ijah & Safiyanu [38] noted that nitrogen helps to facilitate the biodegradation of petroleum hydrocarbon in soils impacted by crude oil spills.

Results obtained from this study showed that wood charcoal block and sawdust create enabling environments for microbial growth in petroleum hydrocarbons contaminated soils. Vidali [39] indicated that bioremediation could only be effective if environmental parameters permit microbial growth and activity; therefore, bioremediation techniques often involve the manipulation of environmental parameters to allow microbial growth and degradation proceed at a faster rate. According to Lee et al. [40], addition of any carbon rich material (charcoal) as a bio-stimulant to a contaminated soil, helps to enhance the degradation of the pollutants (e.g. PAHs), by stimulating the growth of microorganisms responsible for biodegradation of the pollutants in the soil.

![Figure 4: Effect of experimental duration on the THC removal efficiency of charcoal block.](image)

The remediated soil samples, with replacement amendment(s) had better results after the experimental period (40 days), with a mean THC residual value of 1196 mg/kg; when compared with the mean results (3269 mg/kg) obtained from the setups with non-replacement amendment(s). Figure 4 shows the effect of contact duration on the removal efficiency by the charcoal blocks during the experimental process. With reference to the non-replacement amendments, the THC removal efficiency by the charcoal blocks was higher (48.1%) at the initial stage of the experiment (between Day 0 and Day 20), when compared to the removal efficiency value (6.36%) recorded during the later stage of the experiment (between Day 20 and Day 40). Additionally, Figure 5 further conform the effect of contact duration on the adsorption of THC from the contaminated soil samples. While a decline in the accumulated THC concentration adsorbed was observed in the non-replacement charcoal blocks after 20 days; a further increment was observed.
(after the same period) in the accumulative THC concentration for the replacement setup employing charcoal blocks. This portrayed that after a charcoal block had attained its adsorption climax, it will lose its adsorption efficiency; and if possible, some of the already absorbed contaminants (THC) could leak back into the soil. Similar trend was observed by Edokpayi et al. [41]. Edokpayi et al. [42] observed that the adsorption efficiency of coconut shell-based adsorbent increased drastically (6% to 35%) as the contact time increased from 20 min to 60 min, after which it slowed down (35% to 40%) between 60 min to 100 min contact time, before it started to decline after 100 min. According to Abu Bakar et al. [42], the higher adsorption efficiency (fast kinetics) detected during the initial contact time could be credited to the abundant availability of active binding sites within the adsorbent material, which are later filled up as the adsorption process progresses.

![Figure 5: Amount of THC adsorbed by charcoal block during the experimental period.](image)

Since charcoal is not a selective adsorbent material; it will therefore absorb not only the harmful contaminants, but also the useful nutrients (and trace elements) required by the plant’s growth and microbial activity within the soil. Good soil microbial activity is an indicator of good soil quality as it encourages soil microorganisms’ performance. Plants rely heavily on soil microorganisms to mineralize organic nutrients for their growth and development [43]. Therefore, the charcoal quantity (concentration) used for bioremediation needs to be monitored (controlled) during the bioremediation process. Some researchers had provided some information on the importance of absorbent materials quantity regulations in earlier studies. Cheng et al. [44] observed a rapid biodegradation of nonylphenol (petroleum compound) as the concentration of rice straw biochar was increased to an optimal point; after which, further in crement in the rice straw biochar concentration negatively affected the microbial activities in the soil. Cornelissen et al. [45] and Ghosh et al. [46] reported that increasing the concentration of carbonaceous materials (activated carbon, charcoal, biochar, etc.) could lead to a decline in microbial activities (biodegradation) due to reduction in bioaccessibility and bioavailability. Rhodes et al. [24] observed that activated carbon concentration, if not controlled, can reduced the biodegradation process in the soil; this it does due to its’ strong sorptive capacity and ability to increase the very slowly desorbing fractions, which leads to reduce contaminant availability [46]. According to Cheng et al. [44], biochar did not only degrade contaminants in the soils but also affects metabolite rate. The above-mentioned reasons showed the advantage of charcoal block over biochar or granular (powder) charcoal; this is because, charcoal blocks can be easily retrieved (removed) from the remediating soil if an overreaction (abnormal sorptive capacity) is observed.

Materials recovered from the replacement amendment can be recycled for further use. For instance, the replaced will sawdust contains significant amount of petroleum hydrocarbons; therefore, they can be used to produce insects’ resistant plywood boards. Petroleum based oils have been used as effective pesticides for centuries and have advantages over other artificial pesticides because they have; very low mammalian toxicity, low residual activity, and are less disruptive to natural enemies when compared to a broad spectrum of other insecticides [47-49]. Citing Carlos et al. [50] petroleum-based oils are effective and safe alternatives as insecticides and...
fungicides when compared to synthesized insecticides. Some important oils distilled from crude oil include paraffin, kerosene, etc. They can be used to manage pests and diseases.

**Conclusion**

This study was carried out to investigate the effect of wood charcoal block and sawdust on THC biodegradation, in petroleum products contaminated soil samples. Laboratory results obtained from the experiment showed that a combination of charcoal block and sawdust significantly biodegraded the THC concentration of the soil samples. Although the THC concentration of all the soil samples (both the amended and the control) generally declined during the experimental period; THC removal was higher (between 60% and 90%) in the amended contaminated soil samples, when compared to results (18.71%) obtained from the control soil samples. In all cases, it can be seen from the results that replacing the amendments every 10 experimental days significantly influenced biodegradation of the THC in the contaminated soil samples. This study indicated that there was an optimal charcoal THC adsorption efficiency in relation to the contact duration with the contaminated soil after, which adsorption efficiency of the charcoal block declined with time. Furthermore, the amendment affected the morphological performance of the honey bean plants growing in the contaminated soil samples. The results obtained from this study will provide useful information in the utilization of charcoal block and sawdust in bioremediation technique, for the cleaning up of petroleum products/crude oil contaminated sites. In addition, more research needs to also focus on the recycling and utilization of the high concentration THC wood charcoal blocks and sawdust products.

**Declaration of Competing Interest**

The authors declare that they have no conflict of interest.

**Credit Authorship Contribution Statement**

The authors declared that the following contributions are correct.

- Hilary Uguru: Design the research and writing the original draft.
- Ovie Isaac Akpokodje: Data analysis and review of the original draft.
- Esegbuyota D: Literature review and methodology.

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