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Biosorption Potentials of Breadfruit Husk in Remediation of Crude Oil Contaminated Soil

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Abstract: *The study was carried out to evaluate the potentials of bread fruit seed husks in remediating a soil contaminated with heavy metals. Equal volume of soil samples was collected and spiked with 100ml of crude oil. The samples were left for two weeks to simulate conditions of major spill. Five different treatments were added to the soil in 50g, 100g, 150, polluted unamended and natural soil respectively and monitored for five (5) months. Each mixture was setup in triplicates. Physicochemical and selected heavy metals (Lead, Cadmium, Arsenic, and Copper), were assayed using AAS. Results obtained revealed alteration in physicochemistry of the soil samples two weeks after crude oil pollution. Results obtained showed that the concentration of the heavy metals in soil decreased with increasing time for both Control and breadfruit husk extract treated soils. The total hydrocarbon content reduced by 50.9% and 665.09% for sample 100g and 150g bread fruit husk treated soils respectively, while that for 50g was 20.33%. However, higher reductions of the heavy metals were observed in the breadfruit husk extract treated soils compared to controls. Among the treated soils, the optimum remediation occurred at breadfruit husk extract concentration of 150g and 100g at a period of 5 weeks. Therefore, it is concluded that breadfruit husk extract is an effective remediating material for a soil contaminated with heavy metals.*
Keywords: biosorption, breadfruit, husk, remediation, crude oil, contaminated soil

INTRODUCTION

When oil is spilt into the environment, it causes different kinds of pollution that have serious adverse effects on man, animals and plants. These adverse impacts to the ecosystems and the long-term environmental pollution calls for urgent need to develop a wide range of material for clean-up of oil from impacted areas. The management of hydrocarbon is an essential environmental management tool especially in mining companies that deal with large volumes of hydrocarbons and hydrocarbon-related wastes [1]. Soil contaminated with oil is a concern because the contaminated soils are not suitable for agricultural, industrial, or recreational uses and are possible sources of surface and ground water contamination [2]. Agriculture which happens to be the major source of food in Nigeria and all over the world is hindered because of this contamination [2]. Through the expanding population of the country, shortages of food could lead to poor health and poor standards of living. Current public concern and rising costs regarding the conventional clean-up procedures demonstrates the need for a less expensive remediation option such as breadfruit husk.

The treatment/clean-up process employed by the oil companies are not very effective or sometimes dangerous to the aquatic life and affect the food chain [3]. Therefore, there is an urgent need to develop a wide range of materials for cleaning up oil from oil impacted areas. Absorbent materials are attractive for some applications because of the possibility of collection and removal of the oil from the oil spill site [4]. The possibilities of cleaning oil pollution by sorbent on the basis of fibers, polymers and wood products, however, have not yet been sufficiently investigated. Conversion of undervalued and neglected agricultural waste residues such as African breadfruit seed husk, to valuable sorbents for oil spill clean-up has the potential of providing economic incentives. This study will substitute the expensive synthetic sorbent materials used in removal of oil from oil-spill polluted soil.

MATERIALS AND METHODS

Collection and preparation of Soil sample

The soil used in this study was collected from a fallow land behind Chemistry laboratory, Hezekiah University Umudi, Imo State. The site has been lying fallow for over a decade and is surrounded by residential buildings. The soil sample was randomly collected from three spots within 0 – 20 cm depth and mixed together. The collected soil was sun-dried for 48 hours and sieved using a 5mm sieve to remove debris and large stones. The crude oil samples was collected from Nigerian National Petroleum Corporation (NNPC) Port-Harcourt, Nigeria.

Remediating substance and contaminated soil

The remediating substance (breadfruit husk) was obtained from Nweke market in Umudi, Imo State. The breadfruit husk was kept in a polythene bag for 30 days to sun dry. Thereafter the breadfruit husk was taken to the laboratory for analysis.

Experimental design

The experimental pots were filled with 20 kg topsoil, 100 ml of crude oil was thoroughly mixed for even distribution and allowed to age for two weeks to simulate conditions of major spill. Based on the setup, the treatment combinations used in this study were:

20kg topsoil + 100ml of crude oil + 50g breadfruit husk

20kg topsoil + 100ml of crude oil + 100g breadfruit husk

20kg topsoil + 100ml of crude oil + 150g breadfruit husk

20kg topsoil untreated + unamended (natural soil)

20kg soil + 100ml of crude oil unamended

The five (5) sets of experiments were sampled at weekly interval for one (1) month.

Variables

The variables in this experiment were Breadfruit husk concentration, contact time, soil moisture content and temperature.

Analytical methods

Soil analysis; a pre and post soil analysis was carried out to determine concentration of the selected heavy metals (Pb, As, Ni and Cd), and total petroleum hydrocarbons.

Determination of physicochemical properties of soil

Soil samples were collected from each container on a weekly basis to analyse for pH, organic carbon, potassium, nitrogen, phosphorus and total hydrocarbon content. The pH of the soil samples was determined using a glass electrode pH meter and the procedure was as described by McLean (1982). The nitrogen content of the sample was determined by the Kjeldahl method as described in Bremner, (1960). The organic carbon present in the sample was determined using the chromic acid wet oxidation method of Walkley and Black (1965). The available phosphorus in the sample was determined using the colorimetric Molybdenum blue procedure as reported in Bray and Kurtz, (1945). The amount of potassium was determined using a flame photomer (Toth et al., 1948). The total hydrocarbon content was determined spectrophotometrically at a wavelength 460 nm.

Determination of heavy metals in soil

Reagents and materials

Standard stock solutions of Pb, As, Ni and Cd at the 1000ug/mL concentration were obtained. The commercially available nitric acid. Hydrochloric acid, hydrofluoric acid, hydrogen peroxide and ultrapure water. Mixed working solution (containing Ni, Mn, Cr, Cu, Pb, Ba) and internal standard solution (including Ge, In, Rh. Bi).. In 2% nitric acid aqueous solution were prepared. Calibration standards were prepared by diluting mixed standard solution to reach the quantitative concentrations, which added internal standard solution to the concentration level of 50ug/L.

Sample preparation

Approximately 0.2g Soil samples （or spiked soil were weighed into a PTFE beaker. 6 ml of nitric acid, 2 ml of Hydrochloric acid and 2 ml of hydrofluoric in a combination have been used for the simultaneous extraction of a large number of metals in soils. The solution was digested by the Microwave digestion instrument.

Following procedure:

Heated to 120°C in 8 minutes and holding 3 min ;

Raising the temperature to 150°C maintaining 5min ;

Increase the temperature to 190 °C keeping 35 min .

After cooling, 2 ml of H2O2 was added to the digested mixture then taken to heating block in 140°C until the residue solution left about 1 mL.

Finally, the solution was transported into 50 volumetric flasks, brought to volume with water and misted fully. The determination of metals was performed by ICP-MS with internal standard method and standard addition method.

Instrumentation

The Inductively coupled plasma mass spectrometric was carried out to analyze the contents of target elements in the standard mode. Operating conditions (parameters) are summarized.

ICP-MS operating conditions and acquisition parameters.

Operating conditions

Nebulizer Gas Flow 0.88ml/min

Auxiliary Gas Flow 1.20ml/min

Plasma Gas Flow 18.00

Deflector Voltage -11.00v

ICP RF Power 1250w

Analyzer Vacuum 5.0×10-7

Acquisition parameters

Pb, As, Ni and Cd Measured m/z 206,208 63,65 53 55 137,138 60

Calibration range/ug/L5-50 5-50 20-200 100-1000 5-500 10-100

Internal std concentration50 50 50 50 50 50 used as an internal standard element

Standard addition method:

Adding a serials of standard solution (the final solution is equal to the internal standard method calibration range for each element) into the same sample, then scan the standard solution. Draw standard curve which not pass zero point, from the calibration equation we can calculate the each heavy metal level. Sample and blank were analyzed in triplicate.

Adsorption studies

Equilibrium and kinetic studies were determined to ascertain the efficiency of breadfruit husk in adsorbing hydrocarbons using the method of [5]

Estimation of percentage degradation of metals

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Percentage remediation of selected heavy metals (Pb, As, Cd and Cr) was estimated using the formula according to [6]

$$\frac{C_0 - C_1}{C_0} \times \frac{100}{1}$$

Where, c_0 = initial metal level
C_1 = final metal level

Statistical Analysis

Data collected will be subjected to statistical analysis of ANOVA using statistical analytical system (SAS) software, IBM SPSS statistic 20.

RESULTS AND DISCUSSION

Results

Physicochemical properties of the loamy soil used for the study before and after crude oil pollution

Table 1 shows the physicochemical properties of the soil used for the study before and after contamination with crude oil. It was observed that there was a decrease in pH from 5.14 to 5.24. The results revealed that there was a decrease in the nitrogen and potassium contents from 0.169% to 0.147 % and 7.54% to 7.31% respectively. A significant increase in the Total organic carbon (2.09 to 3.22%) and total hydrocarbon content was observed from 0.078 mg/kg to 96.77 mg/kg.

Table 1: Physicochemical properties of the loamy soil used for the study before and after contamination

Parameters	Before Contamination	Two weeks After Contamination
pH	5.14	5.24
Total organic carbon (%)	2.09	3.22
Total nitrogen content (%)	0.169	0.147
Phosphorus content (mg/kg)	7.54	7.31
Potassium content (ppm)	0.31	0.30
Total hydrocarbon content (mg/kg)	0.078	96.77

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Phytochemical characterization of bread fruit husk

The quantitative phytochemical analysis for Bread fruit husk confirmed the presence of Alkaloids (8.063%), Cyanogenic glycoside (6.315 %), Flavonoids (16.367 %), Saponin (6.905 %), Oxalate (0.205mg), Phenol (9.256%), Phytate (5.337mg), Steroids (13.256mg), Tannin (13.916 %), and Terpenoids (4.0%) respectively. The results also indicated higher amount of Steroids (13.256mg**)** in the phytochemicals while the least was observed in Terpenoids (4.0 %). Results of the quantitative phytochemical compositions of bread fruit seed husk is shown in Table 4.2.

Table 2: Phytochemical properties of bread fruit husk sample

Parameters	**Value (%)**
Alkaloids	8.063
Cyanogenic glycoside	6.315
Flavonoids	16.367
Saponin	6.905
Oxalate	0.205mg
Phenol	9.256
Phytate	5.337mg
Steroids	13.256mg
Tannin	13.916
Terpenoids	4.0

Variation in physicochemical properties of soil exposed to different concentrations of bread fruit husk

pH

The pH variation of the soil samples exposed to different concentrations of bread fruit husk with time is shown in Figure 4.1. The pH of the control sample showed a slight decrease in the course of the 5 weeks of treatment. The pH of sample with 20kg topsoil + 100ml of crude oil + 100g breadfruit husk and 20kg topsoil + 100ml of crude oil + 150g breadfruit husk increased from 5.44 to 6.95 and 7.01 respectively within the 5 weeks period of the experiment. The soil sample that had 150g of bread fruit husk generally had the highest pH values and the soil sample that had no treatment had the lowest pH values. The soil sample treated with 150g (20kg topsoil + 100ml of crude oil + 150g breadfruit husk) had the highest final pH value and the soil sample that had no treatment (20kg topsoil untreated + unamended (natural soil) had the lowest final pH value. On contaminating the sample (i.e. week 0), all samples were acidic. Over time, an increasing trend was observed. At the end of the experiment, soil sample treated with 50 g of bread fruit husk was observed

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to be alkaline while soil sample with 100g and 150g bread fruit husks was observed to be less acidic compared to the control samples.

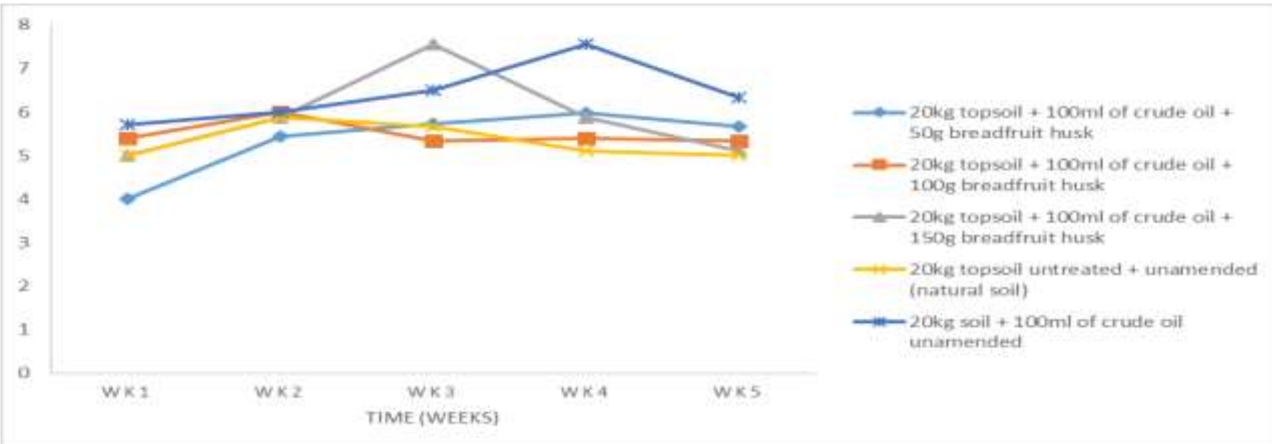

Figure 1: Changes in pH of soil samples over the study period (5 weeks)

Total Hydrocarbon Content

Figure 4.2 shows the variation of the total hydrocarbon content in the soil samples with time. A considerable decrease in the total hydrocarbon concentration occurred in both treated samples compared to the control samples (samples treated with 50g, 100g and 150g) after 5 weeks. The control samples (polluted unamended) did not show significant reduction in the total hydrocarbon content. At the end of the 5 weeks treatment period, the reduction in hydrocarbon content of the polluted soil samples were 50.9% and 665.09% for sample 100g and 150g bread fruit husk treated soils respectively, while that for 50g was 20.33%. Residual hydrocarbon reduction was significantly enhanced and was more significant in the soil sample treated 150g bread fruit husk treated soils.

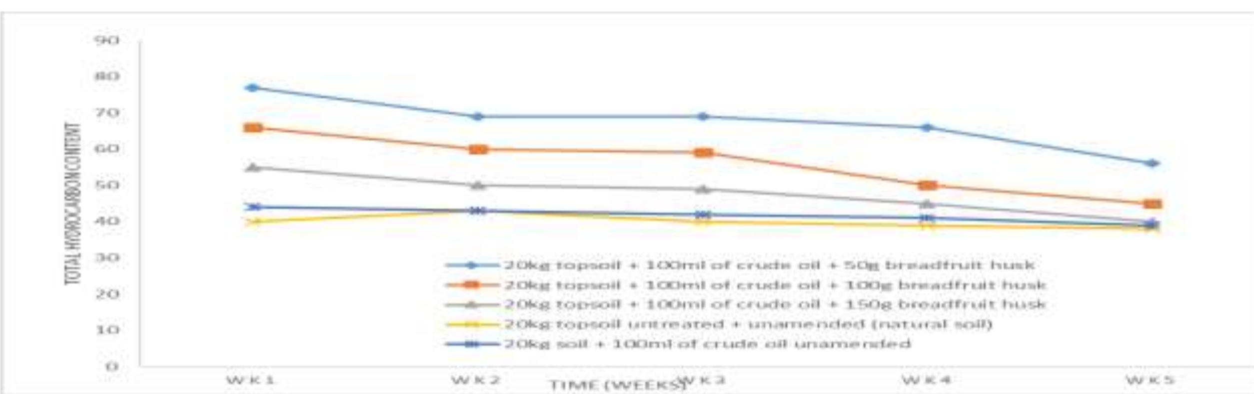

Figure 2: Changes in TPH concentration of soil samples over the study period (5 weeks)

Total Nitrogen Content

Changes in total nitrogen content in the soil samples with time is shown in Figure 4.3. It was observed that there was a continuous decrease in the nitrogen content with time after an initial slow increase in the first week. The soil remediated with 150g of bread fruit husk had a greater reduction potentials in nitrogen content compared to other treatments. The rate of reduction of nitrogen content of sample treated with 50g of bread fruit husk was lower than that of sample 100g and 150g.

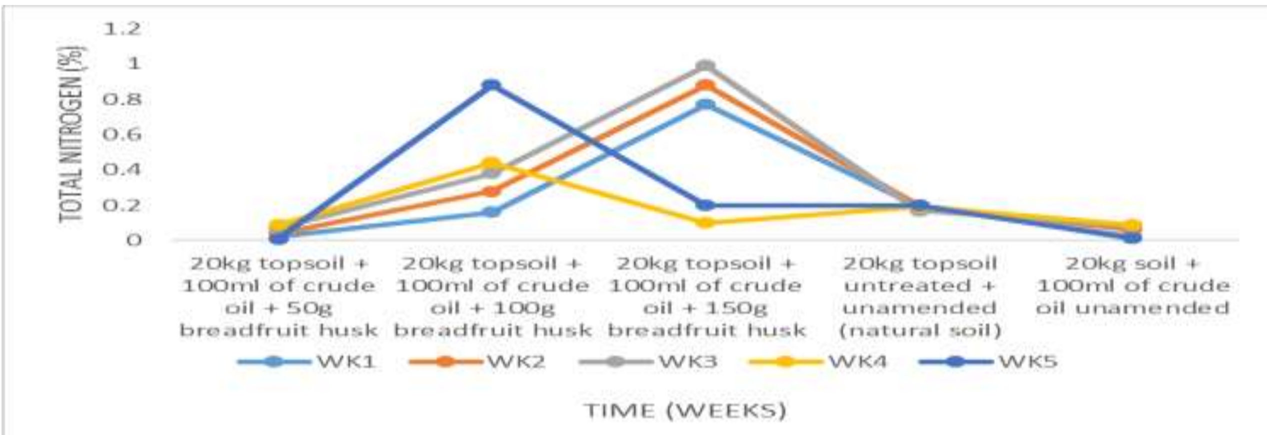

Figure 3: variation of total nitrogen over the study period.

Total organic carbon

Changes in total organic carbon over the study period with time is presented in Figure 4.4. The results revealed that there was a decrease in total organic carbon in the treated samples after five weeks of remediation. At the end of the treatment period, the soil sample containing 100g had a higher total organic carbon compared to soil sample remediated with 150g and 50g of bread fruit husks. The results for all the samples after five weeks of remediation showed reductions from the initial value of 0.22% to 1.45%, 1.31% to 1.52%, and 2.08% to 3.51% for samples treated with 50g, 100g and 150g respectively.

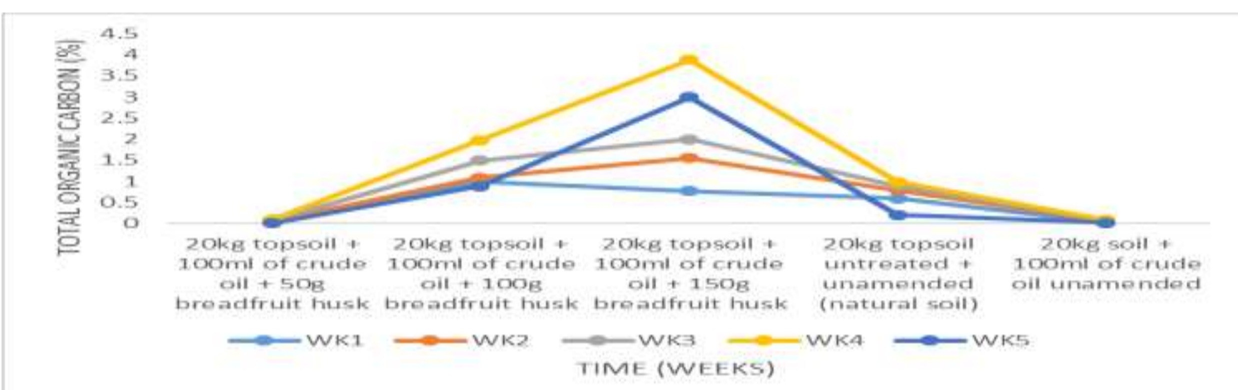

Figure 4.4: Variation of total organic carbon over the study period

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Phosphorus Content

Figure 4.5 shows the variation of phosphorus content in the soil samples with time. Results obtained showed that there was a reduction in phosphorus content of the soil samples treated with 20kg topsoil untreated + unamended (natural soil) and 20kg soil + 100ml of crude oil unamended. The results further revealed that there was a gradual increase in the phosphorus contents from the initial value of 0.09 mg/kg to 0.11 mg/kg, 0.11 mg/kg to 0.16 mg/kg and 0.18 to 0.57% for samples treated with 50g, 100g and 150g respectively.

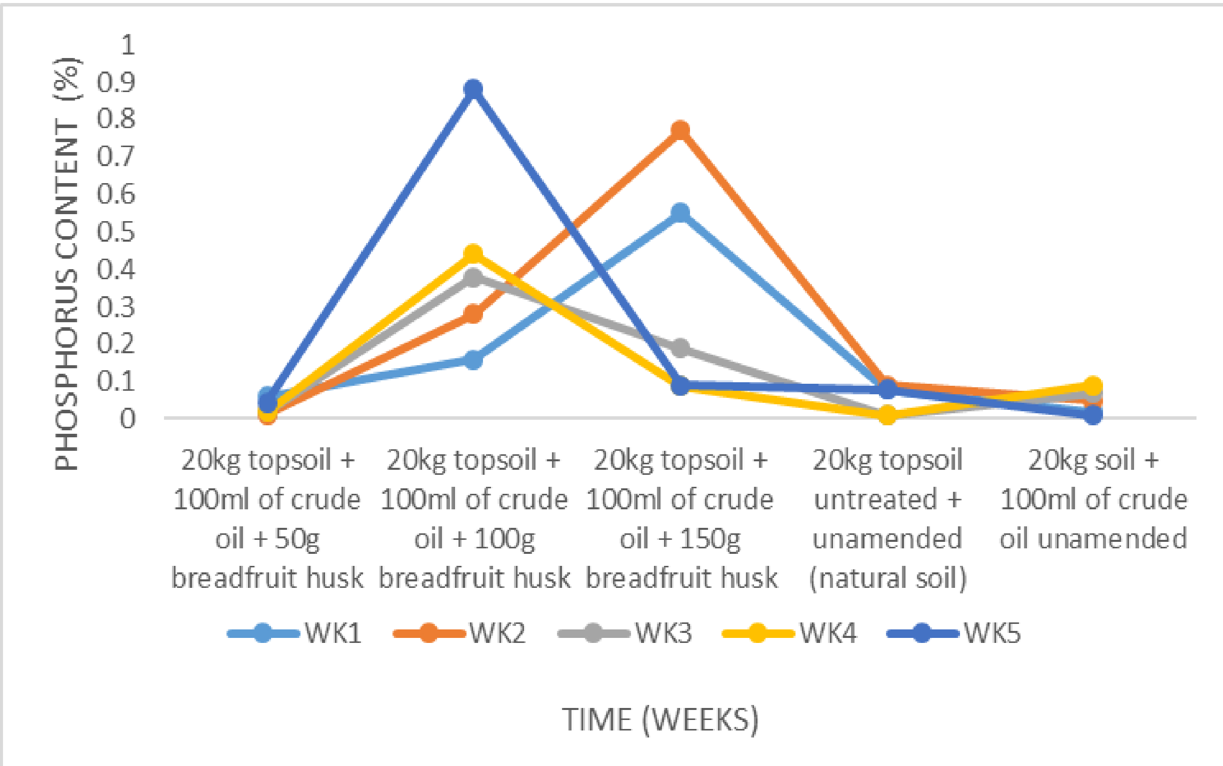

Figure 5: Variation of total organic carbon over the study period

Variation in heavy metal concentration of crude oil polluted soil exposed to different concentrations of bread fruit husk

Concentration of Pb in soil

The concentrations of Pb in soil with time for different concentrations of breadfruit husk extract is shown in Figure 4.6. The concentration of Pb in soil decreased with increasing time and concentration of the breadfruit extract. The control with the least reduction of Pb decreased by 3.7, 15.6, 23.8 and 39.2% while the 150g breadfruit husk treatment with the highest reduction of Pb decreased by 30.6, 58.2, 69.1 and 76.1% for 2, 3, 4 and 5 weeks, respectively.

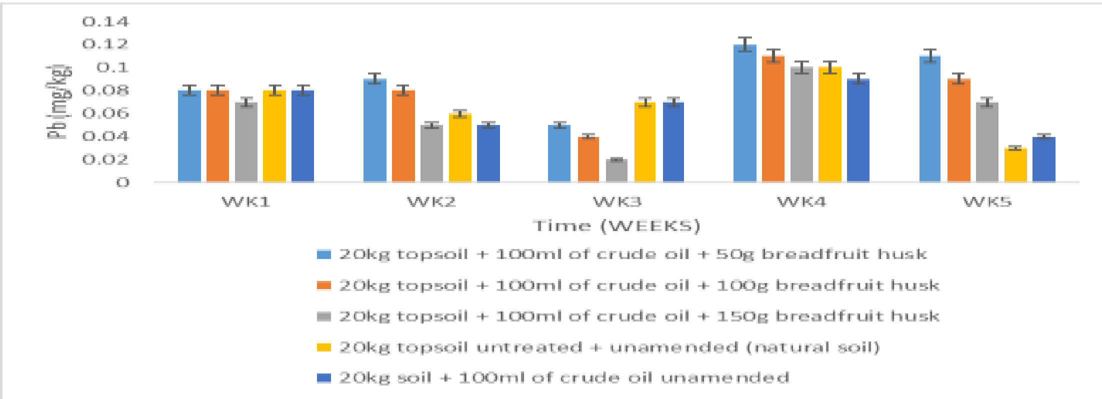

Figure 4.6: Pb concentration in soil with time for different concentrations of bread fruit husk

Concentration of Cd in soil

The concentrations of Cd in soil with time for different concentrations of breadfruit husk extract are shown in Figure 4.7. The concentration of Cd in soil also decreased with increasing time and concentration of the breadfruit husk extract. The control with the least reduction of Cd decreased by 4.0, 13.6, 23.6 and 30.2% while the breadfruit husk extract treatment with the highest reduction of Cd decreased by 40.4, 66.2, 83.2 and 94.0% for 2, 3, 4 and 5 weeks, respectively.

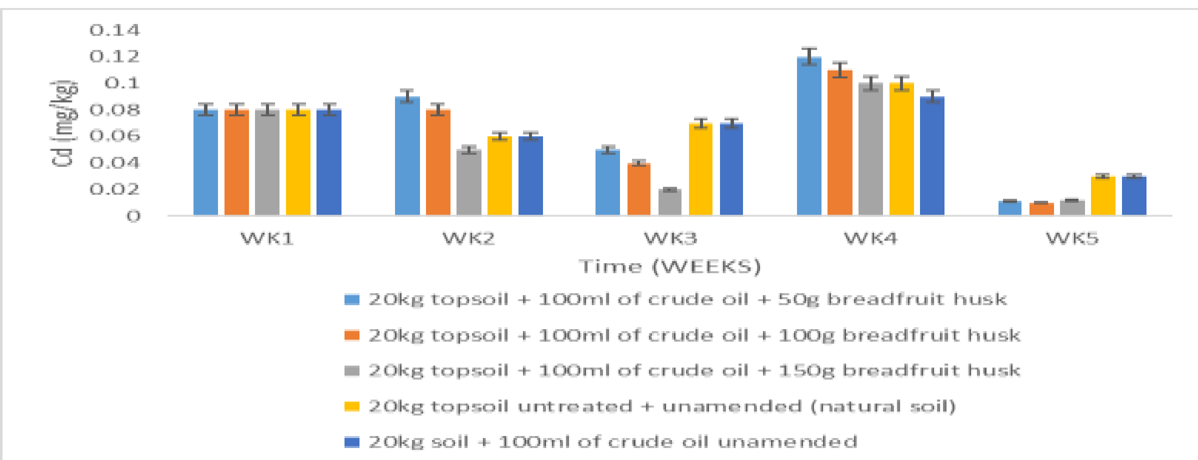

Figure 7: Cd concentration in soil with time for different concentrations of remediating substance

Concentration of As in soil

The concentrations of As in soil with time for different concentrations of breadfruit husk extract is shown in Figure 4.8. The reduction in the concentration of As in soil similarly decreased with increasing time and concentration of the breadfruit husk extract. The control with the least reduction of As decreased by 6.6, 17.1, 32.2 and 40.8% while the treated breadfruit extract treatment with the highest reduction of As decreased by 40.0, 65.9, 80.8 and 87.4% for 2, 3, 4 and 5 weeks respectively.

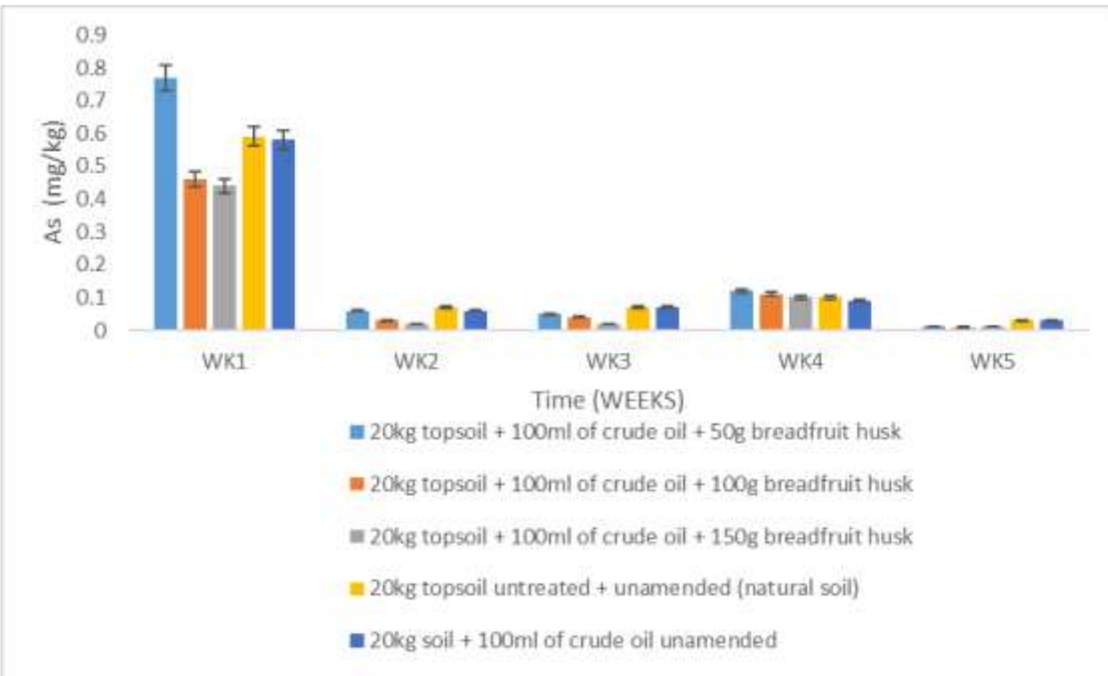

Figure 8: As concentration in soil with time for different concentrations of remediating substance

Concentration of Cu in soil

The concentrations of Cu in soil with time for different concentrations of breadfruit husk extract is shown in Figure 4.9. The reduction in the concentration of Cu in soil similarly decreased with increasing time and concentration of the breadfruit husk extract. The control with the least reduction of Cu decreased by 14.5, 25.1, 39.1 and 59.1% while the 100g breadfruit husk extract treatment with the highest reduction of Cu decreased by 61.4, 74.3, 87.2 and 95.7% for 2, 3, 4 and 5 weeks respectively.

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Figure 4.9: Cu concentration in soil with time for different concentrations of remediating substance

DISCUSSION

The result obtained from the physicochemical analysis of the soil before and after soil contamination showed that the crude oil altered the physicochemical properties of the soil. This could be due to the fact that crude is slightly acidic as reported by previous authors [7]. This result is consistent with earlier reports [8]. The addition of the crude oil led to an increase in the amount of organic carbon from initial value of 0.22% to 1.45%, 1.31% to 1.52%, and 2.08% to 3.51% for samples treated with 50g, 100g and 150g respectively. This could be due to the high carbon content in the crude oil that may have been converted to soil organic carbon. This may also be attributed to changes in the metabolic processes of the soil microflora following crude contamination, thereby reducing its carbon mineralizing capacity [9]

It was also observed that there was a decrease in the nitrogen, phosphorus and potassium contents of the crude oil contaminated soil from 0.169 to 0.147%; 7.54 to 11.31; and 0.31 to 0.30 mg/kg respectively. Crude contamination may have also led to the inactivation of nitrogen fixing bacteria soil and other microbes responsible for organic decomposition in the soil thereby leading to a reduction in the soil nitrogen level. Reduction in potassium content may have been caused by the temporal immobilization of this nutrient by soil microbes as a result of crude contamination of the soil [10]. Nutrient immobilization following oil pollution of soil has also been reported by [11]

A significant increase in the total hydrocarbon content was observed from 0.078 mg/kg to 96.77 mg/kg. This is expected as crude oil is essentially made up of hydrocarbons [3]. This finding supports earlier reports from [12] who noted increase in hydrocarbon content in crude oil polluted soil in Southern Nigeria.

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At the end of the experiment, soil sample with 50 g of bread fruit husk was observed to be alkaline while soil sample with 100g bread fruit husks was observed to be less acidic compared to the control sample. This suggests that the treatment tended to decrease the acidity of the soil samples. This could have resulted from the biodegradation of crude oil by microorganisms under anaerobic conditions in the soil pores. A similar trend was also reported by [13] where pH increased averagely from 5.21 to 10.1 for the remediation of crude oil polluted soil. The pH values observed throughout the 5 weeks of remediation for all samples were within the optimum range of 6-9 as stipulated by the Federal Environmental Protection Agency (FEPA) (FEPA, 2002). A considerable decrease in the total hydrocarbon concentration occurred in both treated samples compared to the control samples after 5 weeks. This conformed to the results obtained by Tanee and Albert (2011) who observed a decrease in total hydrocarbon contents. According to De Souza (2016), breadfruit contains minerals like potassium, phosphorus, magnesium, calcium, manganese, copper and iron which improve soil fertility.

The concentration of Lead (Pb), Chromium (Cr), Arsenic (As), and Copper (Cu) in soil decreased with increasing time for both control and treated soils. However, higher reductions were observed in the treated soils than in the control. This suggests that the breadfruit husk extract is an effective remediating material for heavy metals in the soil. Among the treated soils, the reduction of the heavy metals was found to be in the order of 150g>100g>50, implying that the higher the concentration of the breadfruit husk extract in soil, the more effective would be the remediation of the heavy metals. The percentage of reduction of the heavy metals at 5 weeks in the control experiment was generally equivalent to the percentage reduction at 1 weeks in the highest (150g) breadfruit husk extract treatment. The reduction of the heavy metals as a result of treatment by breadfruit husk extract was found to be in the order of Cu > Cd > As > Pb, indicating that breadfruit husk extract treatment would be most effective in copper contaminated soil. Further study is recommended to fully explain the reasons for the difference in reduction among the heavy metals.

Overall, the result of this study shows that breadfruit husk extract is an effective remediating material for a soil contaminated with heavy metals, and the effectiveness depends on the concentration of the breadfruit husk extract and the time allowed for remediation. The main advantage of this method is that it requires less time to remediate a soil contaminated with heavy metals compared to the widely advertised chemical method of remediation of crude oil polluted soil [14].

CONCLUSION AND RECOMMENDATION

The effectiveness of breadfruit husk extract in remediating a soil contaminated with crude oil polluted soil was studied. The analysis of the soil samples before and after crude oil pollution showed alteration in physicochemistry of the soil. Results obtained showed that the concentration of the heavy metals in soil decreased with increasing time for both control and treated soils, signifying that heavy metals in soil could be reduced via natural attenuation as well as breadfruit husk extract. However, higher reductions of the heavy metals were observed in soils with breadfruit husk extract than in the control, suggesting that the breadfruit husk extract is more effective than natural attenuation. In the soils mixed with breadfruit husk extract, the reduction of the heavy metals in the mixture was in the order of 150 g > 100 g> 50 g, implying that the higher the concentration of the breadfruit husk extract in soil, the more effective would be the remediation of the heavy metal contaminated soil. The reduction of the heavy metals as a result of treatment by breadfruit extract was found to be in the order of Cu > Cd > As > Cr > Pb, indicating that breadfruit extract treatment would be most effective in copper contaminated soil.

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Conclusively, the result of this study showed that breadfruit extract is an effective remediating material for a soil contaminated with heavy metals, and the effectiveness depends on the concentration of the fermented breadfruit extract and the time allowed for remediation.

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