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Biosorption Potential of Breadfruit Husk for Remediating Crude Oil-Contaminated Soil

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ABSTRACT

Background and Objective: Crude oil pollution is an issue of prime concern in most developing countries of the world. The study was conducted to evaluate the potential of breadfruit seed husks in remediating soil contaminated with heavy metals. Soil contamination with heavy metals and crude oil poses significant environmental concerns, necessitating effective remediation strategies. This study aimed to determine the efficiency of breadfruit husk extract in reducing heavy metal concentrations and improving soil quality over time. **Materials and Methods:** Equal volumes of soil samples were collected and spiked with 100 mL of crude oil to simulate contamination. The samples were left undisturbed for 2 weeks to mimic major spill conditions. Five different treatments were applied to the contaminated soil: 50, 100, and 150 g of breadfruit husk extract, along with polluted unamended and natural soil as controls. Each treatment was set up in triplicate and monitored for 5 months. Physicochemical properties and selected heavy metals (Lead, Cadmium, Arsenic, and Copper) were analyzed using Atomic Absorption Spectroscopy (AAS). Data were analyzed using ANOVA at a 0.05% probability level with SAS and IBM SPSS Statistics 20 software. **Results:** Two weeks after crude oil contamination, alterations in the physicochemical properties of the soil were observed. Heavy metal concentrations in both the control and breadfruit husk-treated soils decreased over time. The total hydrocarbon content was reduced by 20.33, 50.9, and 665.09% for soils treated with 50, 100, and 150 g of breadfruit husk extract, respectively. The highest reductions in heavy metal concentrations were observed in the treated soils compared to controls. Optimum remediation was achieved at 150 and 100 g of breadfruit husk extract within 5 weeks. **Conclusion:** Breadfruit husk extract proved to be an effective remediation material for heavy metal-contaminated soil. The study demonstrated that increasing the concentration of breadfruit husk extract enhances the remediation process, with the highest efficiency observed at 150 g. These findings suggest that breadfruit husk extract could be utilized as a natural and sustainable soil remediation strategy.

KEYWORDS

Biosorption, breadfruit, husk, remediation, crude oil, contaminated soil

INTRODUCTION

Crude oil, also known as petroleum, is a naturally occurring, unrefined petroleum product composed of hydrocarbon deposits and other organic materials[1]. Petroleum hydrocarbons are major environmental pollutants generated by wide-scale production, transport, coastal oil refining, shipping activities, offshore

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oil production, and accidental spilling[2]. Anthropogenic activities such as municipal run-off, sand liquid release from industries, cause petroleum hydrocarbon pollution, which impacts the environment and poses a direct or indirect health hazard to different forms of life[3].

When oil is spilled into the environment, it causes different kinds of pollution that have serious adverse effects on man, animals, and plants[4]. These adverse impacts on the ecosystems and the long-term environmental pollution call for an urgent need to develop a wide range of materials for the clean-up of oil from impacted areas[5]. The treatment/clean-up process employed by the oil companies is not very effective or sometimes dangerous to the aquatic life and affects the food chain[6]. 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 collecting and removal of the oil from the oil spill site[7].

The possibilities of cleaning oil pollution by sorbents based on 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 the removal of oil from oil-spill-polluted soil. The study aimed to evaluate the potential of breadfruit seed husks in remediating soil contaminated with heavy metals. It sought to determine the effect of breadfruit husk extract on soil physicochemical properties, assess its efficiency in reducing heavy metal concentrations (Lead, Cadmium, Arsenic, and Copper), and analyze the reduction in total hydrocarbon content. Additionally, the study aimed to identify the optimal concentration and duration required for effective soil remediation.

MATERIALS AND METHODS

Collection and preparation of a soil sample: The study was conducted from June to October, 2024. The soil used in this study was collected from a fallow land behind the 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 a 0-20 cm depth and mixed. The collected soil was sun-dried for 48 hrs and sieved using a 5 mm sieve to remove debris and large stones. The crude oil samples were collected from the Nigerian National Petroleum Corporation (NNPC) in Port Harcourt, Nigeria.

Remediating substances 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, and 100 mL of crude oil was thoroughly mixed for even distribution and allowed to age for two weeks to simulate conditions of a major spill. Based on the setup, the treatment combinations used in this study were:

- 20 kg topsoil+100 mL of crude oil+50 g breadfruit husk
- 20 kg topsoil+100 mL of crude oil+100 g breadfruit husk
- 20 kg topsoil+100 mL of crude oil+150 g breadfruit husk
- 20 kg topsoil untreated+unamended (natural soil)
- 20 kg topsoil+100 mL of crude oil unamended

The 5 sets of experiments were sampled at weekly intervals for 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 the concentration of the selected heavy metals (Pb, As, Ni, and Cd), and total petroleum hydrocarbons[8].

Determination of physicochemical properties of soil: Soil samples were collected from each container every week to analyze 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[8]. The nitrogen content of the sample was determined by the Kjeldahl method[8]. The organic carbon present in the sample was determined using the chromic acid wet oxidation[8]. The available phosphorus in the sample was determined using the colorimetric Molybdenum blue procedure[9]. The amount of potassium was determined using a flame photometer[10]. The total hydrocarbon content was determined spectrophotometrically at a wavelength of 460 nm.

Determination of heavy metals in soil

Reagents and materials: Standard stock solutions of Pb, As, Ni, and Cd at a 1000 ug/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 and Ba) and internal standard solution (including Ge, In, Rh and Bi). In 2% nitric acid aqueous solution was prepared. Calibration standards were prepared by diluting the mixed standard solution to reach the quantitative concentrations, which added the internal standard solution to the concentration level of 50 µg/L.

Sample preparation: Approximately 0.2 g of soil samples or spiked soil were weighed into a PTFE beaker. About 6 mL of nitric acid, 2 mL of hydrochloric acid, and 2 mL of hydrofluoric in a combination were 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 min and held for 3 min
- Raising the temperature to 150°C and maintaining for 5 min
- Increase the temperature to 190°C, keeping for 35 min

After cooling, 2 mL of H_2O_2 was added to the digested mixture, then taken to a heating block at 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 the internal standard method and the standard addition method.

Instrumentation: The inductively coupled plasma mass spectrometry was carried out to analyze the contents of target elements in the standard mode. Operating conditions (parameters) are summarized[10].

ICP-MS operating conditions and acquisition parameters

Operating conditions[10]:

- Nebulizer gas flow 0.88 mL/min
- Auxiliary gas flow 1.20 mL/min
- Plasma gas flow 18.00
- Deflector voltage-11.00 v
- ICP RF power 1250 w
- 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 μg/L: 5-50, 20-200, 100-1000, 5-500 and 10-100
- Internal standard concentration μg/L: 50, 50, 50, 50, 50, 50 used as an internal standard element

Standard addition method: Add a series of standard solutions (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 a standard curve which not pass the zero point from the calibration equation, and can calculate each heavy metal level. The sample and blank were analyzed in triplicate.

Adsorption studies: Equilibrium and kinetic studies were conducted to ascertain the efficiency of breadfruit husk in adsorbing hydrocarbons[5].

Estimation of percentage degradation of metals: The percentage of remediation of selected heavy metals (Pb, As, Cd, and Cr) was estimated using the formula[6]:

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

Where:
C_0 = Initial metal level
C_1 = Final metal level

Statistical analysis: Data collected was subjected to statistical analysis, ANOVA at a probability level of 0.05% using the Statistical Analytical System (SAS) software, IBM SPSS Statistics 20.

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 0.169-0.147% and from 7.54-7.31%, respectively. A significant increase in the total organic carbon (2.09 to 3.22%) and total hydrocarbon content was observed from 0.078-96.77 mg/kg.

Phytochemical characterization of breadfruit husk: The quantitative phytochemical analysis for breadfruit husk confirmed the presence of Alkaloids (8.063%), Cyanogenic glycoside (6.315%), Flavonoids (16.367%), Saponin (6.905%), Oxalate (0.205 mg), Phenol (9.256%), Phytate (5.337 mg), Steroids (13.256 mg), Tannin (13.916%), and Terpenoids (4.0%), respectively. The results also indicated a higher number of Steroids (13.256 mg) in the phytochemicals, while the least was observed in Terpenoids (4.0%). The results of the quantitative phytochemical compositions of breadfruit seed husk are shown in Table 2.

Variation in physicochemical properties of soil exposed to different concentrations of breadfruit husk

pH: The pH variation of the soil samples exposed to different concentrations of breadfruit husk with time is shown in Fig. 1. The pH of the control sample showed a slight decrease in the course of the 5 weeks of treatment. The pH of the sample with 20 kg of topsoil+100 mL of crude oil+100 g breadfruit husk and 20 kg of topsoil+100 mL of crude oil+150 g of breadfruit husk increased from 5.44 to 6.95 and 7.01, respectively, within the 5 weeks of the experiment. The soil sample that had 150 g of breadfruit husk

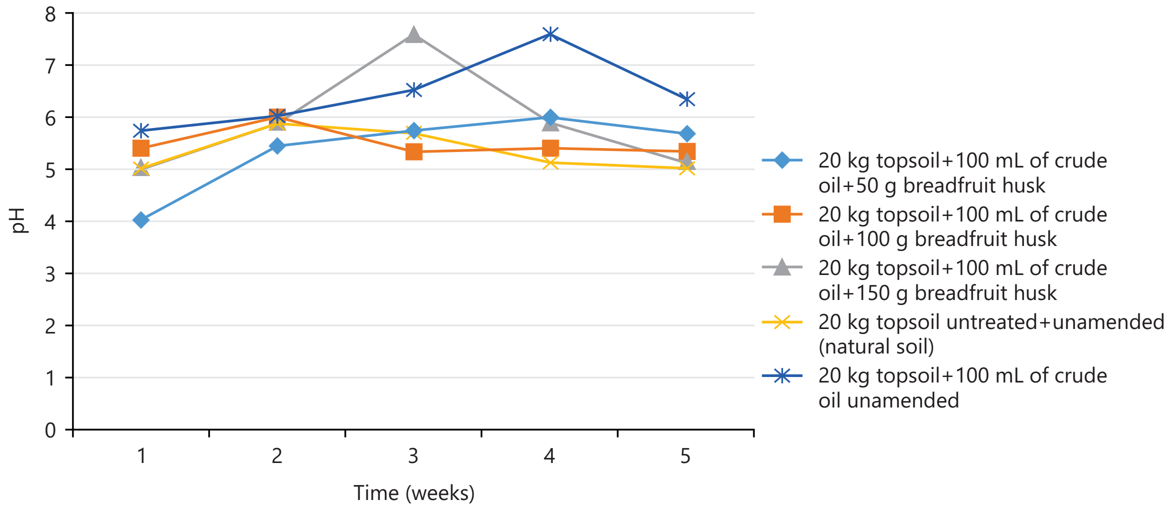

Fig. 1: Changes in pH of soil samples over the study period (5 weeks)

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

Parameter	Before contamination	2 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

Table 2: Phytochemical properties of breadfruit husk sample

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

generally had the highest pH values, and the soil sample that had no treatment had the lowest pH values. The soil sample treated with 150 g (20 kg topsoil+100 mL of crude oil+150 g breadfruit husk) had the highest final pH value, and the soil sample that had no treatment (20 kg 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, the soil sample treated with 50 g of breadfruit husk was observed to be alkaline, while the soil sample with 100 and 150 g of breadfruit husk was observed to be less acidic compared to the control samples.

Total hydrocarbon content: Figure 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 50, 100, and 150 g) after 5 weeks. The control samples (polluted, unamended) did not show a significant reduction in the total hydrocarbon content. At the end of the 5-week treatment period, the reduction in hydrocarbon content of the polluted soil samples was 50.9 and 665.09% for sample 100, and 150 g bread fruit husk-treated soils, respectively,

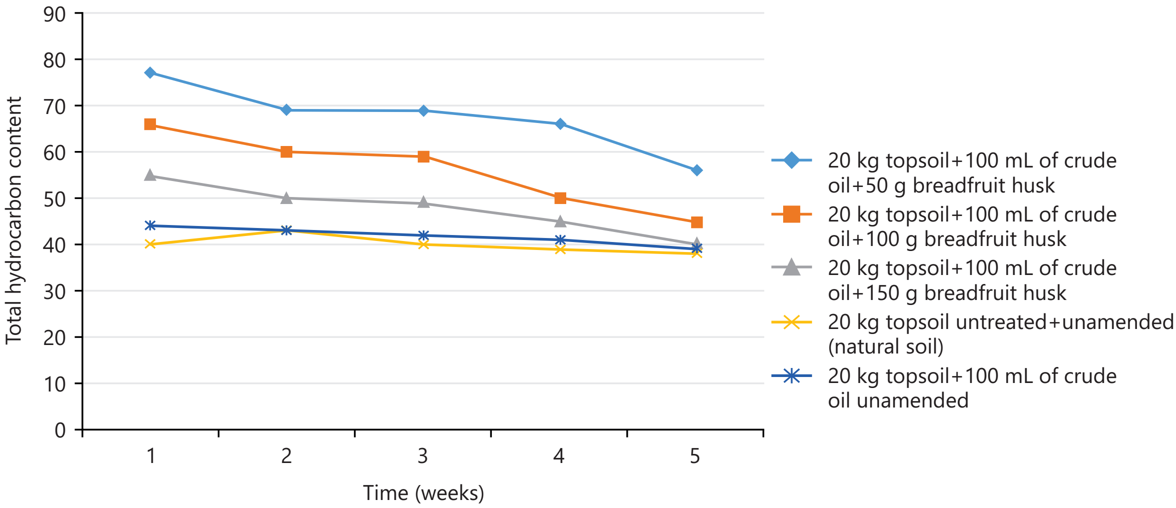

Fig. 2: Changes in TPH concentration of soil samples over the study period (5 weeks)

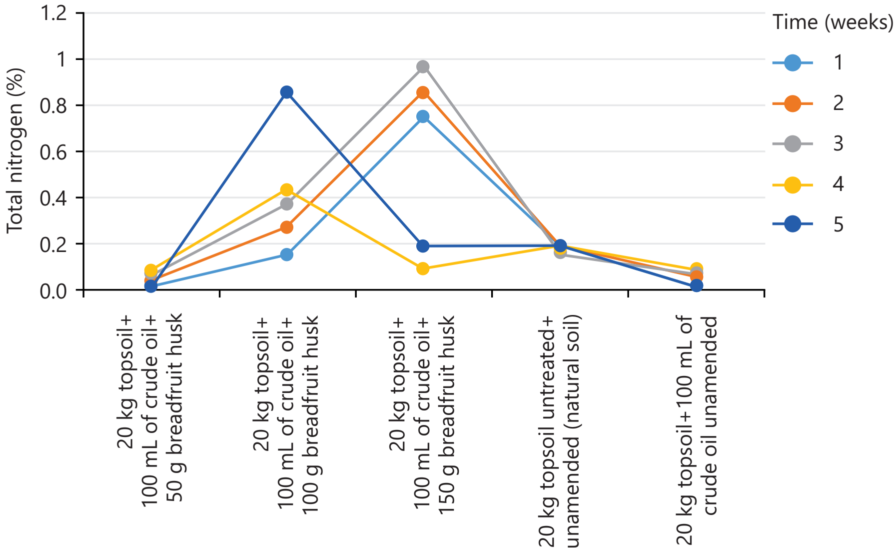

Fig. 3: Variation of total nitrogen over the study period

while that for 50 g was 20.33%. Residual hydrocarbon reduction was significantly enhanced and was more significant in the soil sample treated with 150 g bread breadfruit husk-treated soil.

Total nitrogen content: Changes in total nitrogen content in the soil samples with time are shown in Fig. 3. It was observed that there was a continuous decrease in the nitrogen content with time after an initial slow increase in the 1st week. The soil remediated with 150 g of breadfruit husk had a greater reduction potential in nitrogen content compared to other treatments. The rate of reduction of nitrogen content of the sample treated with 50 g of breadfruit husk was lower than that of the samples 100 and 150 g.

Total organic carbon: Changes in total organic carbon over the study period with time are presented in Fig. 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 100 g had a higher total organic carbon compared to the soil sample remediated with 150 and 50 g of breadfruit husks. The results for all the samples after five weeks of remediation showed reductions from the initial value of 0.22-1.45%, 1.31-1.52%, and 2.08-3.51% for samples treated with 50, 100, and 150 g, respectively.

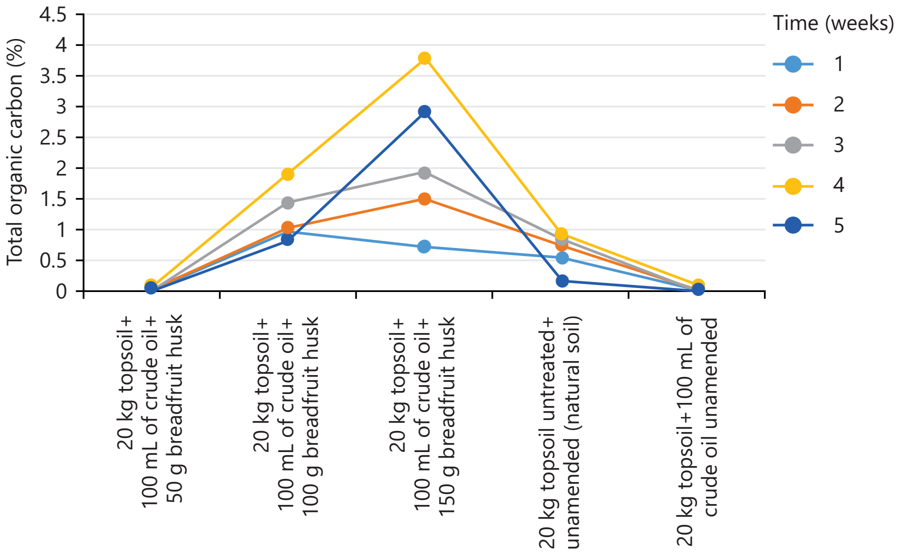

Fig. 4: Variation of total organic carbon over the study period

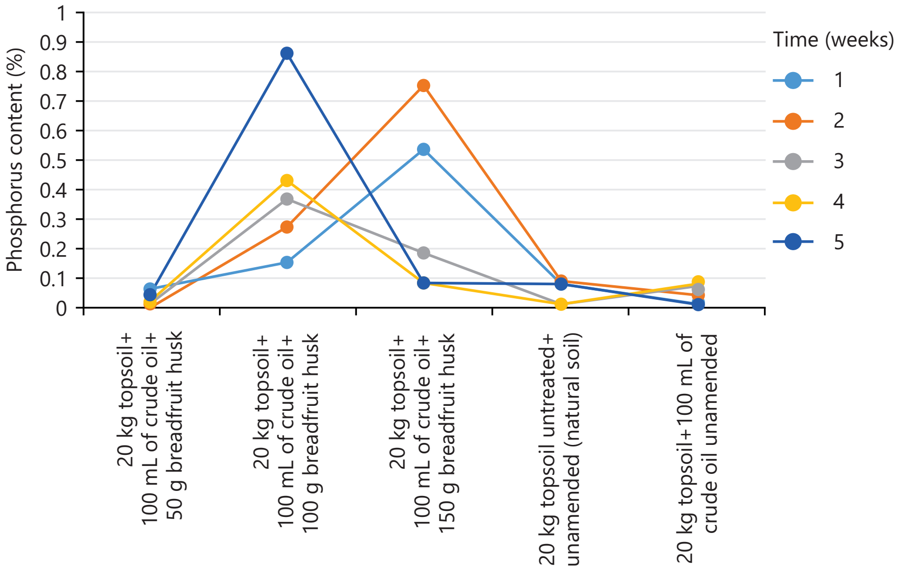

Fig. 5: Variation of phosphorus over the study period

Phosphorus content: Figure 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 20 kg of topsoil untreated+unamended (natural soil) and 20 kg of topsoil+100 mL of crude oil unamended. The results further revealed that there was a gradual increase in the phosphorus contents from the initial value of 0.09-0.8, 0.8-0.6, and 0.67-0.57% for samples treated with 50, 100, and 150 g, respectively.

Variation in heavy metal concentration of crude oil-polluted soil exposed to different concentrations of breadfruit husk

Concentration of Pb in soil: Figure 6 illustrates the temporal variation in lead (Pb) concentration across crude oil-contaminated soils treated with 50, 100, and 150 g of breadfruit husk over five weeks. All treated samples showed a progressive decline in Pb levels, with the 150 g treatment achieving the most significant reduction, from an initial concentration (not explicitly stated) to a 76.1% decrease by week 5. The untreated control showed only a marginal decrease (maximum 0.09%). This suggests that the Pb removal efficiency increased with higher breadfruit husk dosage and longer exposure time. Despite this, lead

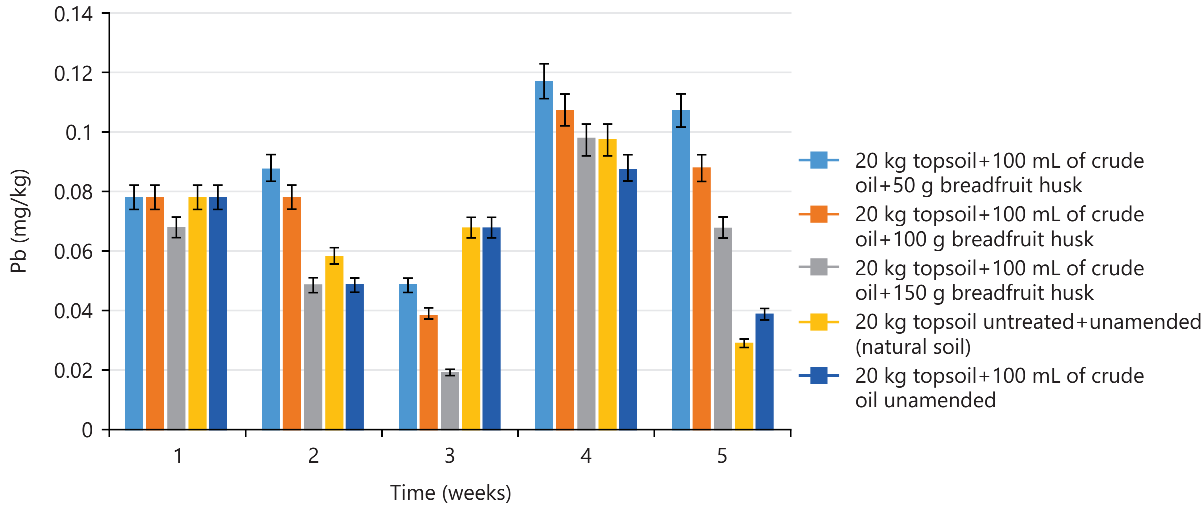

Fig. 6: Pb concentration in soil with time for different concentrations of bread fruit husk

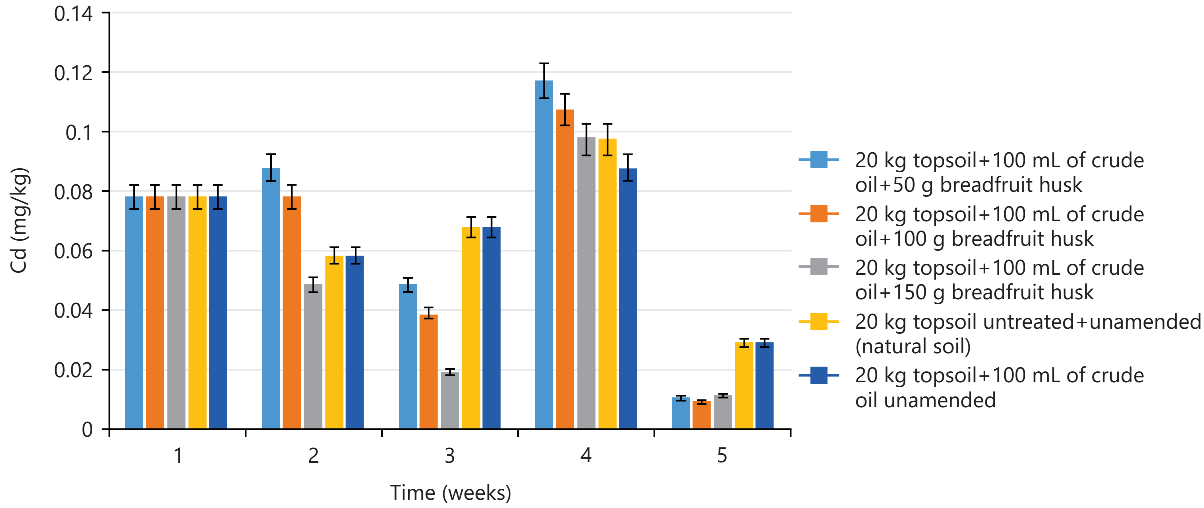

Fig. 7: Cd concentration in soil with time for different concentrations of remediating substance

exhibited the least reduction compared to the other heavy metals studied, indicating relatively lower binding or biosorption affinity of breadfruit husk for Pb.

Concentration of Cd in soil: Figure 7 presents the cadmium (Cd) concentration trends in treated and control soil samples. The 150 g breadfruit husk treatment led to the highest remediation, reducing Cd concentration by 94 mg/kg at week 5. The 100 and 50 g treatments also showed substantial decreases, while the control achieved only 30.2 mg/kg reduction. A clear dose-response relationship was observed, where higher amounts of husk resulted in greater cadmium removal. Compared to Pb, cadmium exhibited higher removal efficiency, likely due to its greater mobility in soil and higher susceptibility to biosorption by the phytochemicals present in breadfruit husk.

Concentration of As in soil: Figure 8 shows the decline in arsenic (As) concentrations over 5 weeks across the control and husk-treated samples. The 150 g treatment again performed best, with a total reduction of 87.4 mg/kg, followed by 80.8 and 65.9 mg/kg reductions for the 100 and 50 g treatments, respectively. The control sample recorded a much lower reduction of 40.8 mg/kg. The steady downward trend indicates that breadfruit husk effectively facilitated arsenic removal from the polluted soil, with efficiency improving

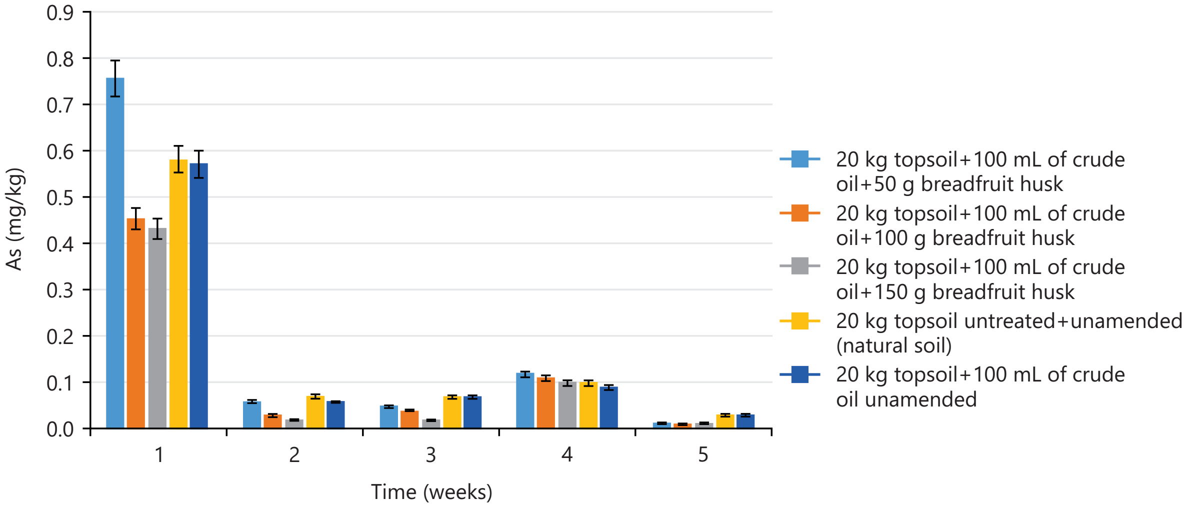

Fig. 8: As concentration in soil with time for different concentrations of remediating substance

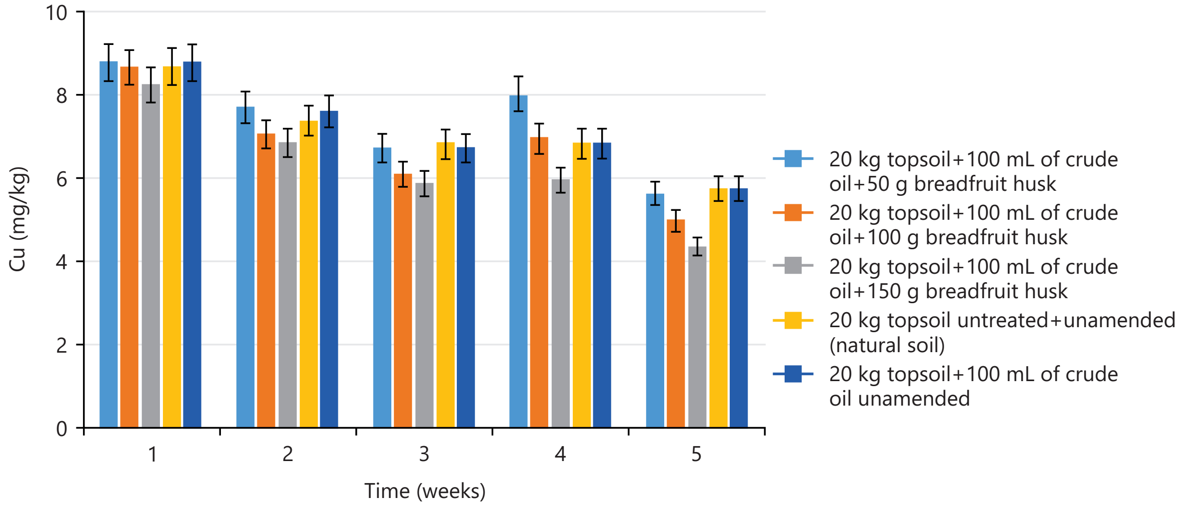

Fig. 9: Cu concentration in soil with time for different concentrations of remediating substance

as the application rate and exposure time increased. Compared to cadmium and copper, arsenic exhibited moderate biosorption efficiency.

Concentration of Cu in soil: Figure 9 depicts the copper (Cu) concentration changes with time across soil samples. Among all the metals tested, copper exhibited the greatest reduction, especially under the 150 g breadfruit husk treatment, which reduced Cu levels by 95.7 mg/kg by week 5. Even the 100 g treatment achieved a high removal rate, while the control sample recorded a modest reduction of 59.1 mg/kg. This pronounced reduction suggests a strong affinity between breadfruit husk components and copper ions, possibly due to complexation or chelation mechanisms facilitated by phenolics, flavonoids, and saponins in the husk.

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 because crude is slightly acidic, as reported by Agbogidi *et al.*[7]. This result is consistent with earlier reports of Agbogidi *et al.*[8]. The addition of the crude oil led to an increase in the amount of organic carbon from an initial value of 0.22-1.45%, 1.31-1.52%, and 2.08-3.51% for samples treated with 50, 100, and 150 g, 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 0.169-0.147%; 7.54-11.31; and 0.31-0.30 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. The 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 Akubugwo *et al.*[11].

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

At the end of the experiment, the soil sample with 50 g of breadfruit husk was observed to be alkaline, while the soil sample with 100 g bread breadfruit husk 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. 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 Bhargava *et al.*[13], who observed a decrease in total hydrocarbon contents. According to Yahaya *et al.*[14], 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 is relevant to the findings of Banks *et al.*[15]. 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 150>100>50 g, implying that the higher the concentration of the breadfruit husk extract in soil, the more effective 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 week in the highest (150 g) 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[15-18].

CONCLUSION

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. 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 the 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>00>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.

SIGNIFICANCE STATEMENT

Crude oil pollution is an issue of global concern in developing countries of the world. Available remediation methods are expensive and not accessible. This study has established the remediation of crude oil-polluted soil. The use of breadfruit husk in crude oil pollution can be replaced with the synthetic method of oil cleanup in developing countries. This study will assist in the reclamation of crude oil-polluted agricultural soils in the tropics.

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