



The Effect of Pyrolysis Temperature of *calotropis procera* Biochar on Dynamics of Petroleum Hydrocarbons Degradation, Carbon Emission, and Ammonia Volatilization in Artificial Petroleum-Contaminated Soil

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Abstract

Pollution by petroleum hydrocarbons (PHCs) is a current environmental challenge all over the world. Therefore, this study was established to evaluate the impact of *calotropis procera* biochar (CPB) produced at various pyrolysis temperatures on carbon emission, ammonia volatilization, degradation of PHCs, and properties of artificial petroleum-contaminated soil. An incubation experiment was performed with CPB produced at 400 °C (CPB400) and 650 °C (CPB650). The artificial petroleum-contaminated soil was amended with biochar at doses of 1, 2.5, and 5% along with the unamended soil (control) and incubated for 78 days. The results demonstrate that the CO₂-C emission rate in petroleum-contaminated soil increases significantly with all biochar applications compared to the control treatment. Adding CPB400 into the soil, at all doses, decreased cumulative NH₃ emission significantly, in comparison with control and CPB650 treatments during all incubation periods. Total PHCs concentration in the soil reduced significantly with CPB400 application at 2.5% compared to the control treatment. CPB400 was more effective in declining total PHCs content in the soil than biochar produced at 650 °C. Available phosphorus and potassium in petroleum-contaminated soil increased significantly under biochar applications compared with the control treatment. Using *Calotropis procera* biochar produced by low-temperature pyrolysis as an amendment can play a vital role in the remediation of PHCs in polluted soils, it is also a promising way to improve the quality indicators of petroleum-contaminated soil.

Keywords Ammonia volatilization · Biochar · Carbon emission · Petroleum hydrocarbons · Pyrolysis temperature

1 Introduction

Petroleum hydrocarbons (PHCs) represent a vast group containing hundreds of chemical compounds which were originated from crude oil and compounds produced from it (Ossai et al. 2020). Globally, increasing PHCs production and consumption increased the risks of environmental pollution that reached water surfaces, groundwater, soils, and air (Ambaye et al. 2022; Liu et al. 2021). The transfer of PHCs to the ecosystem occurs through petrochemical industries during many practices such as exploration, transportation, refining, and production of petroleum

products (Hussain et al. 2018; Karthick et al. 2019). PHCs consist of several pollutants for the environment including aromatic hydrocarbon, saturated hydrocarbon, and non-hydrocarbon compounds. Furthermore, aromatic hydrocarbon is classified as a major organic pollutant and is greatly spread in many environmental systems (Kuppusamy et al. 2020; Zahed et al. 2021). Sixteen types of polycyclic aromatic hydrocarbons have been identified by the United States Environmental Protection Agency and the European Union and they are known for their dangerous mutagenic, carcinogenic, and teratogenic effects (Kusmierz et al. 2016). Because PHCs and their products exist in large quantities in the ecosystem, they are one of the most dangerous types of pollution (Abbaspour et al. 2020; Dike et al. 2021; Saeed et al. 2021). The presence of PHCs in the environment has harmful effects on human health because it leads to skin inflammation, increased

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frequency of psychopathological symptoms, kidney disease, liver diseases, changes in semen, gastritis, elevated levels of serum creatinine, hematologic neoplasms, leukemia, exudative tracheobronchitis, intrapulmonary hemorrhage, carcinogenic, and teratogenic effects (Kuppusamy et al. 2020).

The existence of petroleum materials in the soils leads to the breakdown of soil aggregates, which causes the degradation of its structure and decreased porosity, in addition, it affects soil properties like soil reaction, cation exchange capacity, base saturation, aeration, and moisture (Hussain et al. 2018). All of these characteristics lead to poor plant growth, decreasing photosynthesis, and deterioration of the biological balance in those soils (Hoang et al. 2022). Also, another problem of contaminated soil with PHCs is impeding seed germination of many plants, such as maize, brachiaria, and perennial soybean (Gürtler et al. 2018). The compounds of PHCs have a hydrophobic characteristic which causes their high retention on surfaces of soil colloids, consequently, the soil turns to storage for these compounds and they are constant contaminants and toxic to plants, microorganisms, earthworms, as well as insects (Kusmierz et al. 2016).

Biochar is characterized by several properties such as high surface area, large porosity, surface functional groups, high nutritional content, pH, and exchange capacity that qualify it to be effective in environmental remediation and modern agriculture as a low-cost and eco-friendly amendment (Kamali et al. 2022). Applying biochar is a promising technique that can be used to remediate petroleum-contaminated soils (Bushnaf et al. 2011; Ni et al. 2017). Adding biochar to petroleum-contaminated soils increased the degradation of PHCs by microorganisms (Kong et al. 2018; Ni et al. 2017). The removal of inorganic and organic contaminants from the soil by using biochar amendment occurs through several mechanisms such as cation exchange, complexation, electrostatic interaction, precipitation, hydrogen-bond interaction, pore-filling, hydrophobic interactions, and cation/ π - π interactions which are dependent highly on contaminant type and biochar production conditions (Qiu et al. 2022). There have been no studies investigating how biochar produced from *calotropis procera* at different pyrolysis temperatures can be used to remediate petroleum-contaminated soils and its effect on soil properties. The hypotheses for which this study was designed are that the pyrolysis temperatures of biochar produced from *calotropis procera* are expected to have a significant impact on PHCs degradation, carbon emission, ammonia volatilization, and nutrient availability in artificial petroleum-contaminated soil. Therefore, the present study aims to assess the influences of *calotropis procera* biochar produced at pyrolysis temperatures of 400 and 650 °C on carbon emission, ammonia volatilization,

PHCs degradation, and nutrient availability in artificial petroleum-contaminated soil.

2 Materials and Methods

2.1 Materials

Spent engine oil was taken from a mechanic workshop. Hexane, hydrochloric acid, sulfuric acid, and sodium hydroxide were purchased from Alphachem, Egypt. Sodium bicarbonate, potassium dichromate, and boric acid were purchased from El Nasr Pharmaceutical Chemicals Company, Egypt. Ferrous sulfate was purchased from Alpha Chemika, India. Ammonium acetate was purchased from Prolabo, France. *Calotropis procera* biochar was produced at the laboratory.

2.2 Preparation of Artificial Petroleum-Contaminated Soil

An artificial petroleum-contaminated soil was prepared in the laboratory by treating a calcareous sandy soil with spent engine oil (as a source of petroleum hydrocarbons). Then, contaminated soil was mixed thoroughly, sieved through a sieve (<2 mm), and air-dried. Table 1 shows some important physical and chemical properties of artificial petroleum-contaminated soil.

2.3 A Laboratory Incubation Study

The incubation experiment and all soil analyses were performed in the Soil Chemistry Laboratory, Department of Soils and Water, Faculty of Agriculture, Assiut University, Assiut, Egypt. Initially, 100 g of artificial

Table 1 Physico-chemical characteristics of artificial petroleum-contaminated soil

Property	Unit	Value $\pm$ SD
Sand	g kg ⁻¹ soil	952.00 $\pm$ 0.00
Silt	g kg ⁻¹ soil	10.00 $\pm$ 2.38
Clay	g kg ⁻¹ soil	38.00 $\pm$ 2.38
Texture		Sandy
Ox. OC	g kg ⁻¹ soil	57.89 $\pm$ 0.37
CaCO ₃	g kg ⁻¹ soil	202.35 $\pm$ 1.77
pH (1:2)	-	7.82 $\pm$ 0.04
EC (1:2)	dS m ⁻¹	0.67 $\pm$ 0.03
Available phosphorus	mg kg ⁻¹ soil	8.98 $\pm$ 0.33
Available potassium	mmol kg ⁻¹ soil	1.22 $\pm$ 0.01
TPHCs	g kg ⁻¹ soil	46.81 $\pm$ 1.44

All data are shown as the mean $\pm$ standard deviation (SD). Ox. OC, oxidizable organic carbon; EC, electrical conductivity; TPHCs, total petroleum hydrocarbons

petroleum-contaminated soil, were set in each plastic jar whose volume is 330 ml. Calotropis Procera biochar (CPB) was produced at pyrolysis temperatures (400 and 650 °C). Some chemical properties of biochar are illustrated in Table 2. Biochar was applied to the soil at doses of 1, 2.5, and 5% (W/W) along with the unamended soil (control treatment). The soil in jars was mixed well with biochar. The soil under study was treated with ammonium sulfate as solution form which is applied at a dose of 420 mg N kg⁻¹ soil. Applying distilled water to the soil was at field capacity. This experiment was incubated for 78 days in darkness under normal laboratory conditions as well as recorded temperature ranged from 14 to 25.5 °C. The treatments in this experiment were placed in a completely randomized design with three replications. The CO₂ flux from the soil was captured by 1 M of NaOH (Hopkins 2008) and the ammonia emission was trapped by 4% boric acid placed in small plastic bottles, which were put into each of the incubation jars (Amin 2020a). The NaOH solution in the plastic bottles was replaced after 4, 13, 26, 56, and 78 days. While the boric acid in plastic bottles was replaced after 6, 21, and 78 days. At the same time, two small plastic bottles without soil containing NaOH and boric acid were established as a blank at every incubation time. After finishing the incubation period, the studied soil was prepared for analysis.

2.4 Soil Chemical Properties

The CO₂-C was captured in NaOH and the excess of NaOH was titrated with 0.57 M HCl in the presence of 1 M of BaCl₂ using the phenolphthalein indicator. Moreover, the CO₂-C emission rate (mg C kg⁻¹ soil day⁻¹) and cumulative CO₂-C emission (mg kg⁻¹ soil) were calculated (Hopkins 2008). The emitted ammonia was captured in a solution containing boric acid and NH₃ was titrated with 0.008 M HCl, in addition to calculating the rate of NH₃ emission (mg NH₃ kg⁻¹ soil day⁻¹) and cumulative NH₃ emission (mg NH₃ kg⁻¹ soil) (Amin 2020a). Organic carbon in the

soil and biochar was determined by the dichromate oxidation method; in this method, organic carbon is oxidized by dichromate in the presence of concentrated sulfuric acid with heating and then left to cool, while the excess of dichromate was back-titrated by standard ferrous sulfate (Skjemstad and Baldock 2008). According to Schwartz et al. (2012), the soil pH was estimated in suspension at 1:2 (soil: distilled water, w/v) using a pH meter (HI 3110, HANNA, Romania) and the electrical conductivity was determined in an extract of the same suspension with manual stirring using a conductivity meter (EDT, BA 380, UK). The available phosphorous was extracted from the soil by 0.5 M NaHCO₃ at pH = 8.5 (Olsen et al. 1954). The phosphorus in soil extracts was measured by the colorimetric method (Jackson 1973). Available potassium in the soil was extracted with 1 M of ammonium acetate (pH = 7), and potassium (K) in the extracts was measured by flame photometry (Jackson 1973).

2.5 Determination of Total PHCs

The concentration of total PHCs in the soil under study was extracted using n-hexane as solvent extraction. Five grams of the air-dried soil were set in a 50 ml centrifuge tube and 20 ml of n-hexane was added, shaken for 30 min end-over-end, and centrifuged for 5 min (4000 rpm). The supernatants were filtered using filter paper. Then this step was repeated with 10 ml of n-hexane, to ensure that all total PHCs are extracted (Arimieari and Ezeilo 2019). The extracts obtained from the two steps were collected in beakers. The TPH in each beaker was determined gravimetrically after solvent evaporation in a water bath at 85 °C. After evaporating the solvent, PHCs concentrations were estimated by the weighing method (Galitskaya et al. 2021; Tang et al. 2012). The following formula is used for calculating the degradation of TPHCs (%):

$$\text{Removal of total PHCs (\%)} = \left(\frac{W_0 - W_{\text{final}}}{W_0} \right) \times 100$$

where W_0 is the initial concentration of soil TPHCs (g kg⁻¹ soil) and W_{final} expresses the concentration of residual TPHCs at the end of incubation (g kg⁻¹ soil) according to (Saeed et al. 2021; Zhang et al. 2019).

2.6 Statistical Analysis

The obtained data were analyzed by the MSTAT-C program (version 2.10). The effects of biochar and incubation periods on emissions of carbon and ammonia were tested via Two-way ANOVA. The influence of biochar amendment on total PHCs and soil properties was examined by One-way ANOVA. Trait means were separated with Tukey's honestly

Table 2 Some chemical characterizations of biochar (Data were mean ± standard deviation)

	Unit	CPB400	CPB650
pH (1:10)	-	8.91 ± 0.02	10.12 ± 0.02
EC (1:10)	dS m ⁻¹	2.04 ± 0.01	7.00 ± 0.11
Ox. OC	g kg ⁻¹ biochar	85.56 ± 9.02	44.60 ± 11.68
Available phosphorus	mg kg ⁻¹ biochar	721.01 ± 5.37	542.24 ± 9.09
Available potassium	mmol kg ⁻¹ biochar	398.95 ± 0.64	580.66 ± 3.34

CPB400: calotropis procera biochar pyrolyzed at 400 °C; CPB650: calotropis procera biochar pyrolyzed at 650 °C. EC, electrical conductivity; Ox. OC, oxidizable organic carbon

significant difference test (Tukey's HSD) at a significance level of ≤ 0.01 .

3 Results

3.1 Carbon Emissions

The rate of $\text{CO}_2\text{-C}$ emission in petroleum-contaminated soil increased significantly ($p \leq 0.01$) with all types of biochar applications compared to the control treatment (Fig. 1). The $\text{CO}_2\text{-C}$ emission rate showed a significant rapid increase during the first 13 days of incubation and reduced significantly thereafter until the end of incubation for all treatments. The $\text{CO}_2\text{-C}$ emission rate declined significantly with increasing pyrolysis temperatures of the applied biochar at the same incubation period. Increasing levels of biochar resulted in a significant increase in the $\text{CO}_2\text{-C}$ emission rate (Fig. 1). The maximum values of $\text{CO}_2\text{-C}$ emission rate were recorded at day 13 of the incubation period for all treatments. Then there was a gradual decrease in the rate of $\text{CO}_2\text{-C}$ emission in all treatments. While the maximum value of $\text{CO}_2\text{-C}$ emission rate ($56.24 \text{ mg CO}_2\text{-C kg}^{-1} \text{ soil day}^{-1}$) was noticed at day 13 of the incubation period for 5% CPB400 treatment and the lowest value ($17.48 \text{ mg CO}_2\text{-C kg}^{-1} \text{ soil day}^{-1}$) was observed for control treatment at the end of the incubation (Fig. 1).

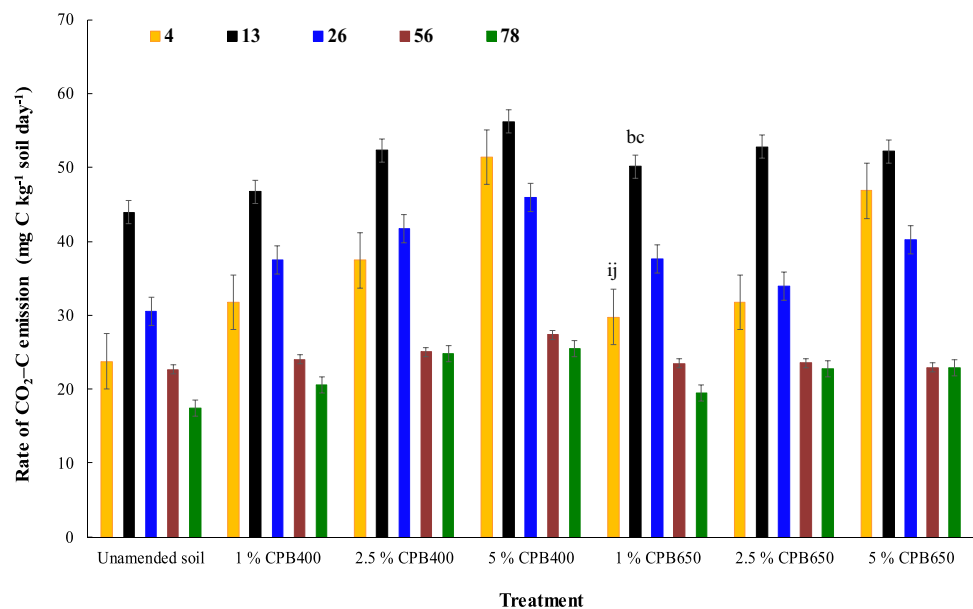
Biochar additions increased cumulative $\text{CO}_2\text{-C}$ emission significantly from petroleum-contaminated soil in comparison with the control (Fig. 2). Generally, the cumulative $\text{CO}_2\text{-C}$ emission increased significantly with increasing doses and incubation periods in all treatments, while it decreased significantly with increasing pyrolysis

temperatures of applied biochar at the same incubation period (Fig. 2). At the beginning of the incubation the cumulative $\text{CO}_2\text{-C}$ emission varied between 95.00, 126.95, 149.77, 205.68, 118.97, 126.95, and 187.42 $\text{mg C kg}^{-1} \text{ soil}$ for control, 1% CPB400, 2.5% CPB400, 5% CPB400, 1% CPB650, 2.5% CPB650, and 5% CPB650, respectively (Fig. 2). Then these values increased to 1951.59, 2209.46, 2461.05, 2690.40, 2192.34, 2252.25, and 2372.62 $\text{mg C kg}^{-1} \text{ soil}$ for control, 1% CPB400, 2.5% CPB400, 5% CPB400, 1% CPB650, 2.5% CPB650, and 5% CPB650, respectively, at incubation end. The largest value of cumulative $\text{CO}_2\text{-C}$ emission ($2690.40 \text{ mg C kg}^{-1} \text{ soil}$) was noticed at the incubation end for 5% CPB400 (Fig. 2).

3.2 Ammonia Emissions

Compared to the control, the addition of CPB400 at all levels caused a significant decrease in ammonia emission rate in petroleum-contaminated soil (Fig. 3). But, adding CPB650 at a 5% level after 6 days of incubation increased significantly ammonia emission compared to control and CPB400 treatments (Fig. 3). Decreasing ammonia emission rate from this soil was significant with increasing incubation periods in all studied treatments. At the beginning of the incubation period, the ammonia emission rate decreased from $1.46 \text{ mg NH}_3 \text{ kg}^{-1} \text{ soil day}^{-1}$ (control treatment) to 0.92, 0.80, 0.76, and $1.39 \text{ mg NH}_3 \text{ kg}^{-1} \text{ soil day}^{-1}$ for 1% CPB400, 2.5% CPB400, 5% CPB400, and 1% CPB650, respectively, as well as increased to 1.54 and $2.03 \text{ mg NH}_3 \text{ kg}^{-1} \text{ soil day}^{-1}$ for 2.5% CPB650, and 5% CPB650, respectively. After 78 days of incubation, the ammonia emission rate decreased from $0.30 \text{ mg NH}_3 \text{ kg}^{-1} \text{ soil day}^{-1}$ (control treatment) to 0.16, 0.09, 0.01,

Fig. 1 Rate of $\text{CO}_2\text{-C}$ emissions in artificial petroleum-contaminated soil as affected by pyrolysis temperature and doses of biochar. Vertical bars indicate the standard error of the mean ($n=3$ replicates). Different lowercase letters on each bar denote the significant differences among treatments according to Tukey's Honestly Significant Difference test at $p \leq 0.01$



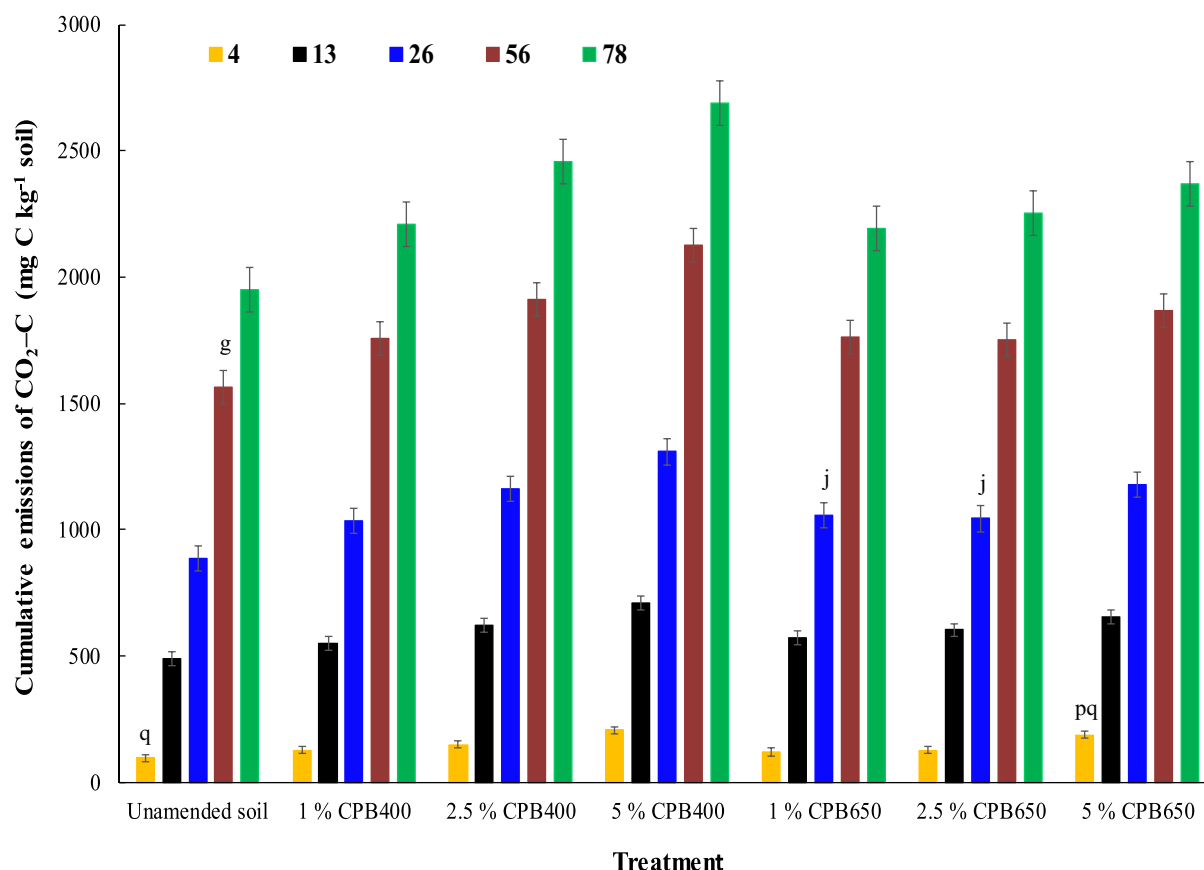


Fig. 2 Changes in cumulative $\text{CO}_2\text{-C}$ emission in artificial petroleum-contaminated soil as influenced by pyrolysis temperature and doses of biochar. Vertical bars indicate the standard error of the mean ($n=3$

replicates). Different lowercase letters on each bar denote the significant differences among treatments according to Tukey's Honestly Significant Difference test at $p \leq 0.01$

and 0.28, 0.24, and 0.04 $\text{mg NH}_3 \text{ kg}^{-1} \text{ soil day}^{-1}$ for 1% CPB400, 2.5% CPB400, 5% CPB400, 1% CPB650, 2.5% CPB650, and 5% CPB650, respectively (Fig. 3).

Overall, the cumulative NH_3 emission increased significantly accompanied by increasing incubation periods in all studied treatments (Fig. 4). Applying CPB400 at all levels caused a significant decrease in cumulative NH_3 emission in petroleum-contaminated soil compared to the control and CPB650 treatments during all incubation periods. After 21 and 78 days of incubation, the application of CPB650 at level 5% decreased significantly the cumulative NH_3 emission compared to the control treatment (Fig. 4). The lowest value of cumulative NH_3 emission (4.53 $\text{mg NH}_3 \text{ kg}^{-1} \text{ soil}$) was recorded in the 5% CPB400 at 6 days of incubation. Meanwhile, the highest value of cumulative NH_3 emission (38.71 $\text{mg NH}_3 \text{ kg}^{-1} \text{ soil}$) was recorded in the unamended treatment at 78 days of incubation (Fig. 4). Biochar prepared at low-temperature was more effective in decreasing ammonia emissions from petroleum-contaminated soil than high-temperature biochar.

3.3 Degradation of Total PHCs

Adding CPB400 at 2.5% decreased the concentrations of the total PHCs significantly ($p \leq 0.01$) in comparison with the control treatment (Table 3). Generally, adding biochar to petroleum-contaminated soil decreased the contents of the total PHCs. At the end of incubation, total PHCs reduced from 44.12 $\text{g kg}^{-1} \text{ soil}$ (control treatment) to 41.85, 34.51, 39.76, 43.90, 42.16, and 40.22 $\text{g kg}^{-1} \text{ soil day}^{-1}$ for 1% CPB400, 2.5% CPB400, 5% CPB400, 1% CPB650, 2.5% CPB650 and 5% CPB650, respectively (Table 3). The largest degradation of total PHCs occurred in this soil when CPB400 treatment was applied at a level of 2.5%. The order of the concentrations of total PHCs in this soil was: control > 1% CPB650 > 2.5% CPB650 > 1% CPB400 > 5% CPB650 > 5% CPB400 > 2.5% CPB400. Biochar prepared at low-temperature is more effective in decreasing the content of total PHCs than high-temperature biochar. Degradation rates of PHCs (%) in various treatments were presented in Fig. 5. At the end of incubation, the relative degradation of PHCs was 5.75, 10.59, 26.28, 15.06, 6.22, 9.93, and 14.08% for control,

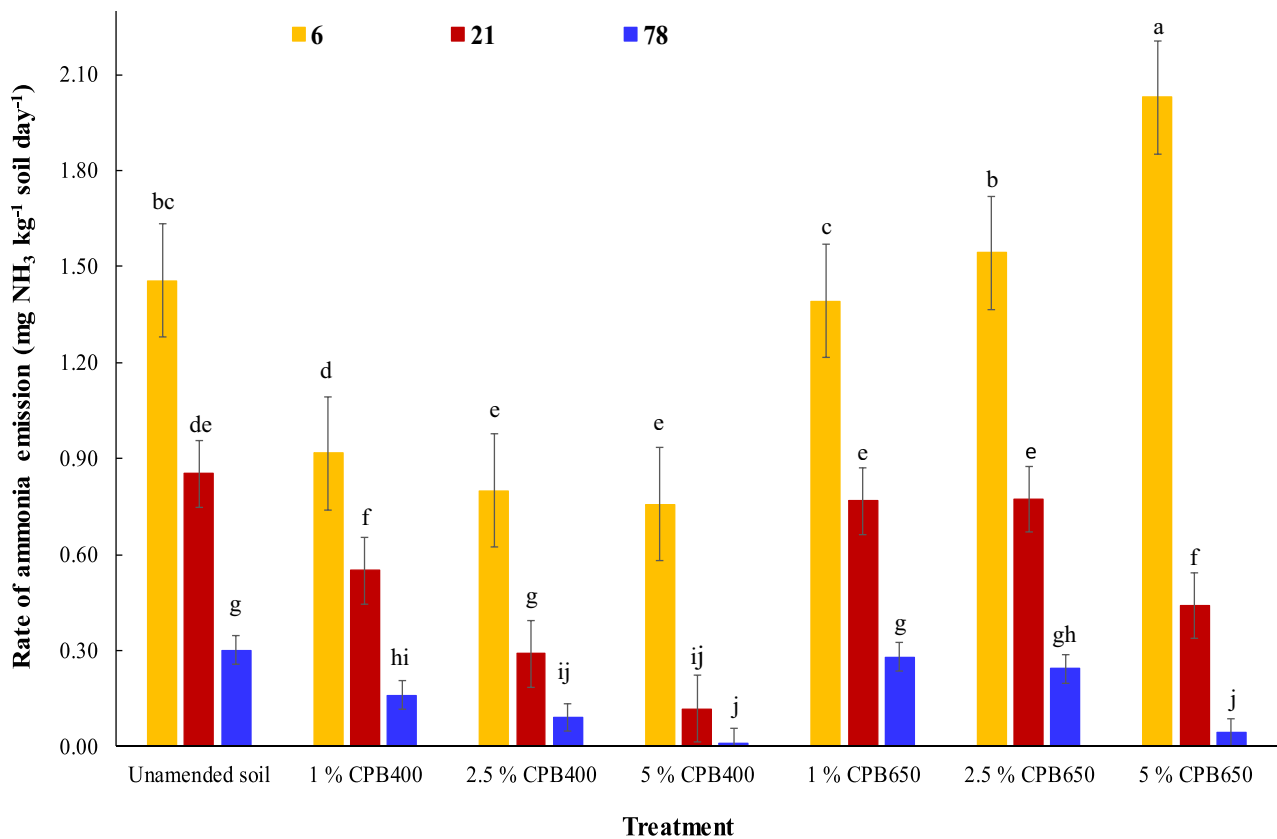


Fig. 3 Ammonia emission rate varied in artificial petroleum-contaminated soil as functions of pyrolysis temperature and doses of biochar. Vertical bars indicate the standard error of the mean ($n=3$ replicates).

Different lowercase letters on each bar denote the significant differences among treatments according to Tukey's Honestly Significant Difference test at $p \leq 0.01$

1% CPB400, 2.5% CPB400, 5% CPB400, 1% CPB650, 2.5% CPB650, and 5% CPB650, respectively (Fig. 5). The highest value of PHCs degradation was noticed at 2.5% CPB400 treatment.

3.4 Properties of Petroleum-Contaminated Soil

Compared with the control, the pH of petroleum-contaminated soil decreased significantly after adding 1% CPB400, 2.5% CPB400, 2.5% CPB650, and 5% CPB650 ($p \leq 0.01$), but, the application of 5% CPB400 treatment led soil pH to increase significantly. Soil pH values decreased from 7.35 for control to 7.29, 7.29, 7.32, 7.22, and 7.18 for 1% CPB400, 2.5% CPB400, 1% CPB650, 2.5% CPB650, and 5% CPB650, respectively (Table 3). High-temperature biochar effectively decreased soil pH more than low-temperature biochar under application levels of 2.5 and 5%. The addition of biochar into the soil increased the electrical conductivity (EC) significantly (Table 3). EC values increased from 1.83 dS m⁻¹ for control to 2.03, 2.26, 2.52, 2.24, 2.53, and 3.31 dS m⁻¹ for 1% CPB400, 2.5% CPB400, 5% CPB400, 1% CPB650, 2.5% CPB650, and 5% CPB650, respectively.

High-temperature biochar increased effectively the EC values than low-temperature biochar.

Available phosphorus increased significantly in petroleum-contaminated soil under biochar applications for all biochar treatments, except for 1% CPB650 where the increase was non-significant compared to the control (Table 3). Available phosphorus concentrations increased from 3.22 mg kg⁻¹ soil for control to 6.40, 6.56, 12.48, 4.82, 6.46, and 8.25 mg kg⁻¹ soil for 1% CPB400, 2.5% CPB400, 5% CPB400, 1% CPB650, 2.5% CPB650, and 5% CPB650, respectively (Table 3). Low-temperature biochar effectively improved phosphorus availability than biochar produced high-temperature when adding a similar dose. All biochar additions into petroleum-contaminated soil increased significantly available potassium compared to the control treatment. Available potassium enhanced from 1.04 mmol kg⁻¹ soil (control) to 4.12, 8.60, 14.58, 6.35, 14.64, and 23.41 mmol kg⁻¹ soil for 1% CPB400, 2.5% CPB400, 5% CPB400, 1% CPB650, 2.5% CPB650, and 5% CPB650, respectively. High-temperature biochar increased effectively potassium availability than low-temperature biochar when applying a similar dose (Table 3).

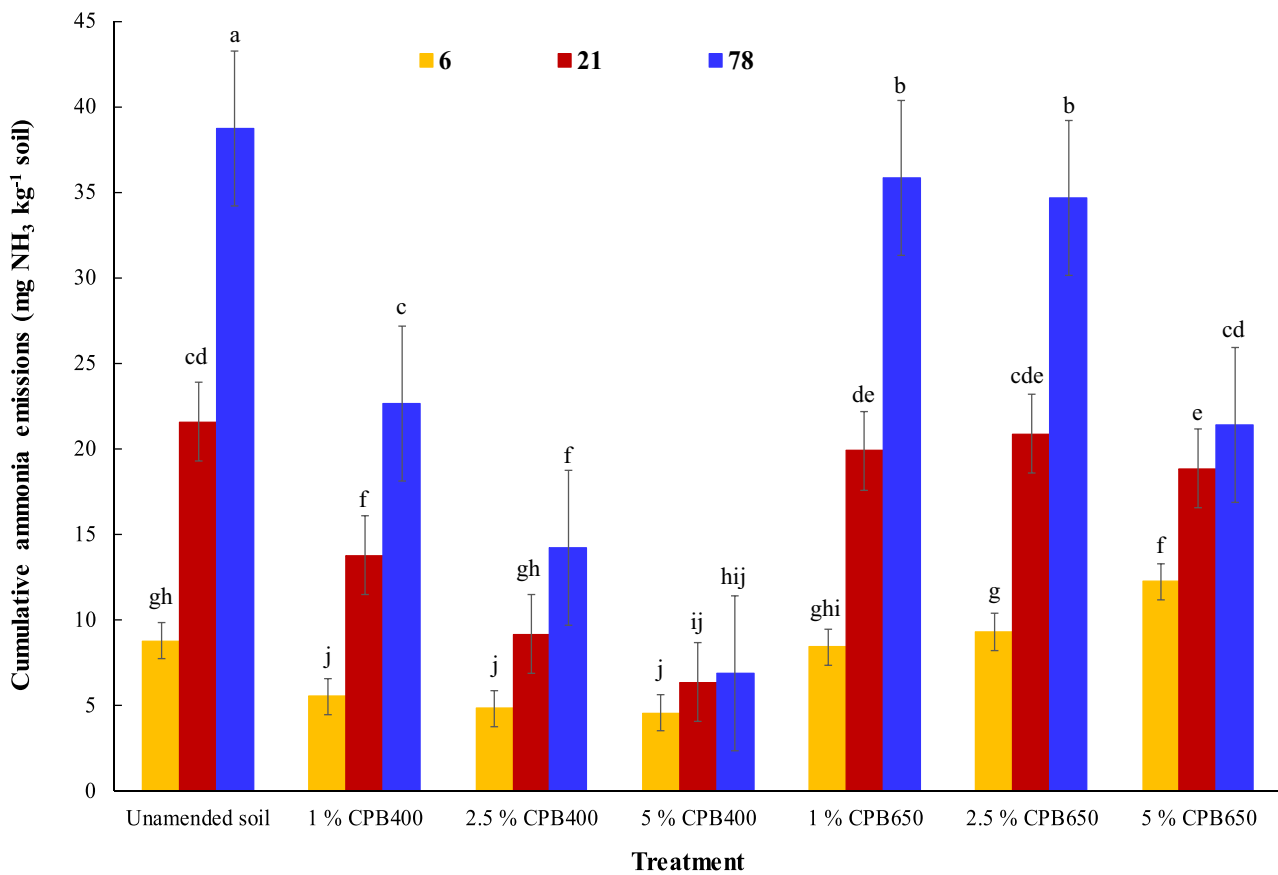


Fig. 4 Effects of pyrolysis temperature and doses of biochar on changes of cumulative NH₃ emission in artificial petroleum-contaminated soil. Vertical bars indicate the standard error of the mean ($n=3$

replicates). Different lowercase letters on each bar denote the significant differences among treatments according to Tukey's Honestly Significant Difference test at $p \leq 0.01$

Table 3 Effect of pyrolysis temperature and doses of biochar on total petroleum hydrocarbons degradation and chemical properties of artificial petroleum-contaminated soil. (Data were mean $\pm$ standard deviation)

Treatment	TPHCs (g kg ⁻¹ soil)	pH	EC (dS m ⁻¹)	Olsen-P (mg kg ⁻¹ soil)	Available K (mmol kg ⁻¹ soil)
Control	44.12 $\pm$ 3.04 a	7.35 $\pm$ 0.01 b	1.83 $\pm$ 0.04 e	3.22 $\pm$ 0.17 d	1.04 $\pm$ 0.05 f
1% CB400	41.85 $\pm$ 5.10 ab	7.29 $\pm$ 0.02 c	2.03 $\pm$ 0.05 d	6.40 $\pm$ 0.85 bc	4.12 $\pm$ 0.16 e
2.5% CB400	34.51 $\pm$ 1.67 b	7.29 $\pm$ 0.00 c	2.26 $\pm$ 0.05 c	6.56 $\pm$ 0.03 bc	8.60 $\pm$ 0.22 c
5% CB400	39.76 $\pm$ 1.08 ab	7.47 $\pm$ 0.04 a	2.52 $\pm$ 0.05 b	12.48 $\pm$ 1.12 a	14.58 $\pm$ 0.48 b
1% CB650	43.90 $\pm$ 1.30 a	7.32 $\pm$ 0.01 bc	2.24 $\pm$ 0.01 c	4.82 $\pm$ 0.34 cd	6.35 $\pm$ 0.03 d
2.5% CB650	42.16 $\pm$ 1.04 ab	7.22 $\pm$ 0.01 d	2.53 $\pm$ 0.03 b	6.46 $\pm$ 0.43 bc	14.64 $\pm$ 0.19 b
5% CB650	40.22 $\pm$ 1.36 ab	7.18 $\pm$ 0.02 d	3.31 $\pm$ 0.06 a	8.25 $\pm$ 0.06 b	23.41 $\pm$ 0.32 a

TPHCs, Total petroleum hydrocarbons; EC, electrical conductivity. Different lower case letters in each column indicate the significant differences among treatments according to Tukey's Honestly Significant Difference test at $p \leq 0.01$

4 Discussion

Generally, soil microorganisms contribute 99% of CO₂ emissions resulting from the decomposition of organic matter (Rahman 2013). Respiration rates of

microorganisms in soils as a form of CO₂ are indicators of petroleum hydrocarbon biodegradation resulting from the decomposition of organic matter to get the demanded energy for growth. CO₂ and water are the final outcomes caused by aerobic degradation during energy-producing reactions (Barati et al. 2017). Compared to the unamended

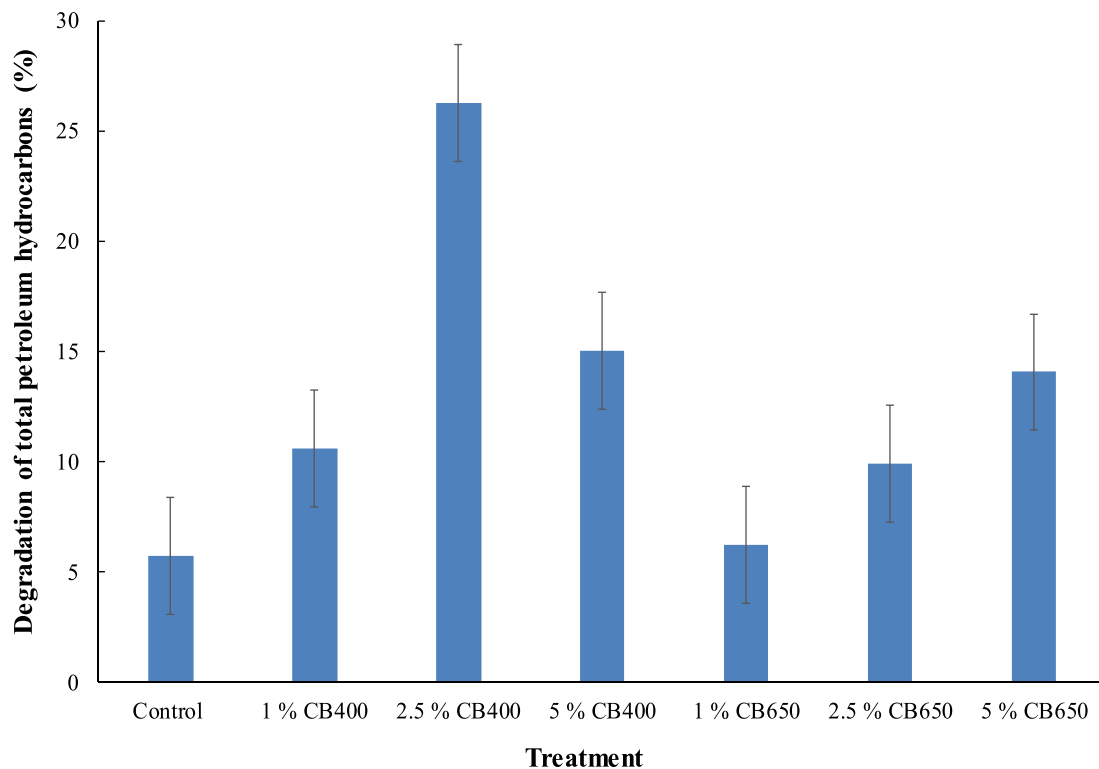


Fig. 5 Degradation of total petroleum hydrocarbons (%) in artificial petroleum-contaminated soil as influenced by pyrolysis temperature and doses of biochar. Vertical bars indicate the standard error of the mean ($n = 3$ replicates)

soil, biochar applications caused higher CO_2 -C emissions because the biochar contains high carbon content (Yang et al. 2019). Decreasing CO_2 -C emissions with increasing pyrolysis temperatures (Amin 2020a; Deng et al. 2021) has resulted from the content of dissolved organic carbon, released from biochar, which is reduced with increasing pyrolysis temperatures and increased stable carbon (Deng et al. 2021). Biochar applications increased the carbon dioxide emission from petroleum-contaminated soil and the release of CO_2 increased with the increasing incubation period (Zhen et al. 2021). In petroleum-contaminated soil, CO_2 emissions can be used to determine microbial biomass, metabolic rates, and microbial activity. Moreover, the respiration rate of soil microbes was high at 14 days of incubation which provides further evidence that nutrients released by biochar enhance the metabolism of soil microbes and increase petroleum hydrocarbons biodegradation (Guo et al. 2022).

Ammonia volatilization from the soils treated with biochar relies on the biomass type, application doses, and pyrolysis temperatures (Amin 2020a, 2023; Mandal et al. 2018). Numerous experiments found various results for the influence of biochar addition on the volatilization of ammonia from soils. Some studies showed that biochar amendment effectively increased the volatilization of NH_3 (Feng et al.

2018; Wang et al. 2017), this increase is attributed to increasing soil pH and nitrification reduction (Feng et al. 2017). In contrast, some other studies showed that biochar applications caused a decrease in ammonia volatilization (Amin 2020a; Mandal et al. 2018). (Amin 2020a) reported that calotropis biochar produced at 400 °C has a higher cation exchange capacity than that produced at 650 °C. Therefore, the ability of biochar prepared at 400 °C to retain ammonia is much higher than biochar prepared at 650 °C. This in turn leads to a decline in ammonia volatilization. Biochar addition into the soil led to increasing pH and cation exchange capacity as well as improving oxidation of ammonia, consequently, this reduces the volatilization of ammonia from the soil (Sun et al. 2019). Several mechanisms are responsible for ammonium retention in biochar-amended soils such as adsorption at the cation exchange site, sorption at surface functional groups, absorption in micropores, and microbial processes (Mandal et al. 2016, 2018).

Physico-chemical characteristics of biochar as structure, decomposition stability, high surface area, and cation exchange capacity, qualify it to be suitable for the remediation of soils (Dike et al. 2021; Zahed et al. 2021). In the soils, hydrocarbons degradation is controlled by several factors: pH, salinity, aerobic conditions, temperature, pollutant types, oxidation-reduction reactions, nutrient

concentration, organic matter content, moisture content, and microbial communities (Zahed et al. 2021). Adding biochar increased significantly PHCs degradation because biochar has many properties that qualify it to be appropriate as a soil amendment and also enhanced microorganisms' activity in the soil (Barati et al. 2017; Kong et al. 2018), however, biochar applications did not lead to a significant increase in petroleum hydrocarbon degradation (Han et al. 2016). In one other study, the additions of ponderosa pine wood biochar increased the degradation rate, but the walnut shell biochar prevented PHCs degradation (Mukome et al. 2020). Applying biochar to soils increased partition coefficients and degradation rates of PHCs (Bushnaf et al. 2011). All this depends primarily on the feedstock type which biochar is produced from (Han et al. 2016; Mukome et al. 2020). The pyrolysis temperatures of producing biochar play a major role in influencing the rate of PHCs degradation (Kong et al. 2018) because the pyrolysis temperatures of biochar have a great effect on bacteria communities which degraded petroleum hydrocarbon degradation in contaminated soil. Increasing pyrolysis temperature caused a reduction of bacteria communities responsible for petroleum hydrocarbon degradation (Guo et al. 2022). Substantial studies suggested that the residues of agriculture and biochar were utilized to immobilize carrier materials (Deng et al. 2016; Xiong et al. 2017; Zhang et al. 2019) because these amendments are distinguished by a high water holding capacity, oxygen transfer, and enhancement of enzymatic activities (Wang et al. 2019; Zhang et al. 2019). While, biochar is preferred to agricultural residues due to its richness in carbon, high surface area, high stability, nutrient retention, and immobilization of bacteria (Zhang et al. 2019). The microorganisms decompose low molecular weight polycyclic aromatic hydrocarbons more readily and volatile than the higher molecular weight polycyclic aromatic hydrocarbons, which are known for their high resistance to microbial degradation. This resistance is due to the high hydrophobicity of aromatic rings (Aydin et al. 2017; Kong et al. 2018; Sui et al. 2021) and electron transfer needs more energy to take place (Aydin et al. 2017). Fertilizer additions to the soils caused increasingly significant degradation of PHCs because it enhances microorganisms' community as well as their activity (Han et al. 2016; Nikolopoulou and Kalogerakis 2009). The degradation of petroleum hydrocarbon increased with increasing amounts of phosphorus addition in uncultivated soil (Hoang et al. 2022). The removal of hydrocarbons from petroleum-contaminated soils by biochar is attributed to several mechanisms such as sorption in low concentration, partition in high concentration, partitioning dominant at low pyrolysis temperature, adsorption-dominant at high pyrolysis temperature, adsorption by nano-porous, hydrophobic partition, physisorption,

chemisorption, hydrophobic interactions, entrapment in pore of biochar, irreversible sequestration, immobilization, and biodegradation (Wei et al. 2024).

The application of PHCs to the soil increased electrical conductivity and decreased pH (Shaoping et al. 2021). Hydrocarbons are a source of necessary carbon for microorganisms' growth in the soil, and this leads to the production of organic acids and carbon dioxide, causing a decrease in the soil pH. Then, the remains of CO₂ release, consumption of organic acids, sulfides, and short-chain alkanes contributed to soil pH increment (Shaoping et al. 2021). The lower pH of CPB650 than CPB400 in this study may be attributed to the presence of salts such as sulfates and chlorides of calcium, magnesium, and sodium that caused an increase in the activity of hydrogen ions (Brady and Weil 1999). Biochar has electrical conductivity values that are highly influenced by biomass type and pyrolysis temperature (Amin 2020a, b; Senbayram et al. 2019; Zhang et al. 2017). Adding several biochar types increased the electrical conductivity in the soils (Amin 2020a, 2022) because of biochar's high content of dissolved ions (Smider and Singh 2014). Values of electrical conductivity increased with increasing levels of biochar (Amin 2022; Smider and Singh 2014). The electrical conductivity in the soils under *calotropis* biochar addition increased under increasing pyrolysis temperature from 400 to 650 °C (Amin 2020a). With increasing pyrolysis temperatures, the EC values increased which can be attributed to the incremented concentrations of soluble salts and loss of acidic functional groups in biochar (Narzari et al. 2017; Zhang et al. 2020). Biomass type and pyrolysis temperature affect greatly concentrations of phosphorus and potassium in biochar (Cao et al. 2018; Liao et al. 2018). Increasing biochar applications to some soils increased the available phosphorus and potassium (Amin 2016, 2022; Song et al. 2019). Most types of biochar are rich in their content of alkaline cations, particularly potassium (Amin 2016; Xu et al. 2013). Applying *calotropis* biochar prepared at 400 °C led to an increase in the available phosphorus higher than the biochar prepared at 650 °C, however, adding *calotropis* biochar increased the available potassium with increasing pyrolysis temperature (Amin 2020a).

5 Conclusions

Global population growth and industrial development have increased the demand for petroleum products, hence increasing environmental pollution risk. All this led to the urgent need for new strategies to remediate this pollution. The importance of this study lies in that it provides a new vision for using biochar to rehabilitate and reclaim petroleum-contaminated soils. Therefore, the goals of the study are to test using *calotropis procera* biochar produced at pyrolysis temperatures of 400

and 650 °C on petroleum hydrocarbons degradation and quality indicators in artificial petroleum-contaminated soil. Applying *calotropis procera* biochar to artificial petroleum-contaminated soil increased the degradation of petroleum hydrocarbons. Biochar prepared at 400 °C showed more effectiveness in degrading petroleum hydrocarbons in the soil than biochar prepared at 650 °C. Biochar produced by low-temperature pyrolysis has played an important role in reducing ammonia volatilization from the soil. Low-temperature biochar effectively ameliorates phosphorus availability in artificial petroleum-contaminated soil than biochar produced by high-temperature. High-temperature biochar increases the electrical conductivity than low-temperature biochar. Our results support the hypotheses of this study that pyrolysis temperatures of biochar have a significant impact on petroleum hydrocarbons degradation, ammonia volatilization, and nutrient availability. Accordingly, biochar prepared at low temperatures is a more suitable strategy for the remediation of petroleum-contaminated soil while biochar prepared at high temperatures consumes large amounts of energy and loses some nutrients. The results of the current study contribute to finding sustainable solutions for the rehabilitation of petroleum-contaminated soil through the applications of biochar. Biochar is currently used as a soil amendment because of its several agricultural, economic, and environmental benefits.

Author Contributions I contributed to the study's conception and design. Material preparation, data collection, and analysis were performed by me. The first draft of the manuscript was written by me and I commented on previous versions of the manuscript. I read and approved the final manuscript.

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Data Availability The datasets generated during and/or analyzed during the current study are available from the corresponding author on reasonable request.

Declarations

Ethics Approval Not applicable.

Consent to Participate Not applicable.

Consent to Publish Not applicable.

Competing Interests The authors have no relevant financial or non-financial interests to disclose.

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