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Biochar amendment as a remediation strategy for surface soils impacted by crude oil☆

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ABSTRACT

The impact of organic bulking agents on the biodegradation of petroleum hydrocarbons in crude oil impacted soils was evaluated in batch laboratory experiments. Crude oil impacted soils from three separate locations were amended with fertilizer and bulking agents consisting of biochars derived from walnut shells or ponderosa pine wood chips produced at 900 °C. The batch reactors were incubated at 25 °C and sampled at pre-determined intervals to measure changes in total petroleum hydrocarbons (TPH) over time. For the duration of the incubation, the soil moisture content was adjusted to 75% of the maximum water holding capacity (MWHC) and prior to each sampling event, the sample was manually stirred. Results show that the addition of fertilizer and bulking agents increased biodegradation rates of TPH. Soil samples amended with ponderosa pine wood biochar achieved the highest biodegradation rate, whereas the walnut shell biochar was inhibitory to TPH biodegradation. The beneficial impact of biochars on TPH biodegredation was more pronounced for a soil impacted with lighter hydrocarbons compared to a soil impacted with heavier hydrocarbons. This study demonstrates that some biochars, in combination with fertilizer, have the potential to be a low-technology and eco-friendly remediation strategy for crude oil impacted soils.

1. Introduction

Crude oil continues to be the major source of energy worldwide with annual demand increasing from 80.1 to 97.8 million barrels per day from 2003 to 2017 (IEA, 2011). During the exploration, production, and transport of crude oil, accidental environmental releases may occur. As these activities are conducted in many environments, novel, cost-effective remediation technologies are of significant interest to industry. Crude oil can be classified as light or heavy, with light crude oil containing a higher percentage of light hydrocarbons, having a lower specific density, and typically being more reliably degradable in soil. Crude oil found in soils is weathered by environmental processes, and differs considerably from freshly extracted crude oil. The weathered oil has undergone, evaporation, dispersion, dissolution, biodegradation, and emulsification processes that lead to changes in the physical and chemical properties of the extracted crude oil (Lehr, 2010). For example, most of the volatile hydrocarbons in fresh crude are lost to the atmosphere, resulting in the residual crude having larger, more condensed, and less volatile heavy hydrocarbons (HH). Similarly, dissolution and biodegradation also lead to progressively greater recalcitrance of the residual crude.

Crude oil releases to soil at some sites have led to temporary reductions of microbial activity and reduced plant growth, in part due to decreased availability of essential nutrients from the soil e.g., (Braddock et al., 1997). This can, in turn, temporarily reduce or

Abbreviations: List: HH, heavy hydrocarbon. TPH; total petroleum hydrocarbon. MWHC, maximum water holding capacity. Fert: fertilizer. P900; ponderosa pine biochar (900 °C). WA900, walnut shell biochar (900 °C). ABT; antibacterial treatment. WC, ponderosa pine wood chips.

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inhibit natural degradation rates of petroleum compounds (Abii and Nwosu, 2009; Hickman and Reid, 2008). Crude oil can also reduce water and air permeability in the soil pores, potentially affecting plant rooting (Knapp, 1979).

Biodegradation of petroleum compounds in soil can often be enhanced using relatively simple bioremediation strategies that include the addition of fertilizer and bulking agents, such as sawdust or compost (Lodge, 1994; Cajthaml et al., 2002) and frequent watering and mechanical treatment (tilling). This approach is particularly effective for relatively light crude oils (McMillen et al., 2001). However, bioremediation is typically less effective for soils with heavy crude oil that contain a relatively high percentage of poorly biodegradable or recalcitrant TPH components. Conventional bioremediation strategies for such soils may require a very long time to reach a target TPH end-points or may not be able to achieve TPH targets within an acceptable timeframe (McMillen et al., 2001). In these cases, landfarming may not be an option, or a simpler, more efficient, and cost-effective method is desirable. We hypothesize that biochar could be part of a viable remediation strategy to enhance landfarming practices.

Biochar is a form of pyrogenic carbon which is a byproduct of pyrolysis or gasification of organic waste to produce biofuels, as well as an intentional product of partial pyrolysis. The use of biochar as an agricultural soil amendment has received increased attention since the discovery of the Terra Preta de Indio soils in the Amazon. These soils received historical applications of pyrogenic carbon (charcoal), and today have higher organic C and improved soil fertility (Uretsky et al., 1975; Liang et al., 2006). There are, however, still many unknowns about biochar because source material and pyrolysis conditions affect molecular biochar structure and therefore affect its persistence and reactivity as an sorbent (Brewer et al., 2009; Keiluweit et al., 2010). Examination of different biochar source materials and pyrolysis temperatures has shown distinctively different biochar products (Brewer et al., 2009; Novak et al., 2009a,b; Keiluweit et al., 2010; Mukome et al., 2013).

Due to their highly porous and hydrophobic nature, biochars are often found to be favorable ad/absorbents for a variety of natural and synthetic organic chemicals and heavy metals, as well as a favorable substrate for colonization by bacteria and fungi (Thies and Rillig, 2009), the key drivers of bioremediation. The addition of biochar to soil has been shown to increase sorption of organic compounds such as pesticides (Spokas et al., 2009), heavy metals (Jones et al., 2016) and polycyclic aromatic hydrocarbons (PAHs) (Chen et al., 2008; Zhang et al., 2010). Biochar produced from pine needles (100–700 °C) showed a high affinity for naphthalene, nitrobenzene, and *m*-dinitrobenzene from water (Chen et al., 2008). In that study, sorption increased linearly with biochar aromaticity, which increased with pyrolysis temperature. Increased sorption of a phenanthrene (a relatively low molecular PAH) was also observed with increasing pyrolysis temperature (350–700 °C) of Monterey Pine (*Pinus radiate*) (Zhang et al., 2010). Consistent with these results, biochar addition to a polluted soil greatly reduced both metal and PAH concentrations in the soil pore water (Beesley et al., 2010), and the application of birch wood biochar enhanced the sorption of phenanthrene in 20 agricultural soils from Denmark (Kumari et al., 2014).

Specific to crude oil, Pignatello and co-workers (Nguyen and Pignatello, 2013) showed that four commercial hardwood biochars and six synthesized biochars from maplewood (anoxic, 300–700 °C) could absorb several times their own weight of Texas, South Louisiana, or Qua-Iboe Nigeria light crude oils floating on seawater. The oil absorption capacity, determined in dip tests, ranged from 3.6 to 6.3 g/g. Absorption capacity peaked at the production temperature of about 400 °C and correlated poorly with % C, H/C ratio, O/C ratio, surface area, and porosity. They also observed an increase in CO_2 evolved from oil-on-seawater mixtures to which biochar had been added. The authors suggest that swelling, as a consequence of oil absorption, in addition to macropore filling, may be primarily responsible for the high oil capacities of biochar. They concluded that biochar may prime biodegradation by providing a favorable solid support for growth of bacterial degrader biofilms that can utilize the hydrocarbons imbibed by the biochar particles.

Biochar addition to soils has been shown to increase petroleum hydrocarbon partition coefficients, while at the same time increasing their rates of degradation (Bushnaf et al., 2011). One study demonstrated improved hydrocarbon degradation via an immobilized microorganism technique using biochar as the carrier of PAH-degrading bacteria (Chen et al., 2012). Qin et al. (2013) studied bioremediation of oil-contaminated soil in 180 day lab tests and reported that degradation efficiency was significantly increased when rice straw biochar was added; they monitored TPH, saturated hydrocarbons, aromatics and the polar fraction. They surmised that biochar addition may increase biodegradation of some chemcial species by sequestering the polar intermediates that otherwise may inhibit those reactions. Another study showed that biochar (feedstock and production parameters not provided) combined with rhamnolipid was effective in reducing TPH levels in landfarming experiments. The suggestion that biochar can enhance biodegradation and serve both as a sink and a site for biodegradation of crude oil hydrocarbons forms the basis for a promising *in situ* remediation strategy for oil contaminated soils. However, it is unknown if this process will also result in a decrease in TPH, because sorption alone does not degrade any HH, and extraction solvents used for TPH analytical methods may be able to desorb most HHs from the biochar surfaces.

The central hypothesis addressed by this study is that biochar will promote reduction of TPH concentration when added to soil. The specific objectives are: 1) to evaluate the effectiveness of different biochars in enhancing the biodegradation of weathered crude oil, and 2) to compare the efficacy of this biochar in soils contaminated with light and heavy weathered crude oil under realistic conditions. Mechanistically, we postulate that biochar promotes TPH reduction by serving as an absorptive sink for HH and by providing a favorable substrate for colonization by microbial hydrocarbon degraders.

2. Materials and methods

2.1. Soil and biochar

The soils for this study were composite surface soil grab sample blends of HH contaminated soils collected from multiple sites and supplied by Chevron. After collection, each composite was homogenized by thoroughly mixing the soils. Two composites were contaminated by light crude (Soils A and B), and a third composite was contaminated by heavy crude (Soil C). Key chemical and physical properties of the soils are summarized in Table 1.

Two biochars from different feedstocks were produced at 900 °C. The first, WA900, was made from walnut shells (*Juglans californica)* in a gasifier; details on its physical and chemical characteristics and manufacturing given by Mukome et al. (2013). The second, P900, was made from ponderosa pine (*Pinus ponderosa*) under controlled conditions in a laboratory reactor located in the Environmental Engineering Biomass Laboratory at UC Davis. The reactor was a 0.1 × 2 m internally circulating fluidized bed reactor with gas extraction and purification capacity, and a controlled temperature/atmosphere/residence time single-screw moving bed reactor with liquids and char recovery. Biochars produced at 900 °C were selected as they can be produced during bioenergy

Table 1
Physical and Chemical properties of the soils used for the study.

Sample ID	TPH (mg/kg)	Crude Oil	Sample Description	Total organic matter (%)	CEC (meq/100g)	Moisture (%)	pH
Soil A	24000	Light	Sand/silt/clay mix, with some free product.	1.8	18.3	8.8	**7**
Soil B	16000	Light	Silty matrix with fine sand, water saturated with some free product.	4.7	16.5	26	**7.4**
Soil C	21000	Heavy	Sand/silt/clay mix, dry with no free product.	4.3	30.4	2.3	**7.9**

Table 2
Physical and Chemical properties of biochars used for the study.

Biochar	Feedstock	Production	pH	% Moisture	% Ash	%C	C/N	H/C	B.E.T. Surface area (m^2/g)
P900	Ponderosa Pine	Slow Pyrolysis	9.9	1.2	2.7	92.4	308	0.07	127
WA900	Walnut Shell	Gasification	9.7	3.1	46.4	55.3	117.7	0.22	75

production and thus their production has added value; in fact, WA900 is a bioenergy byproduct. Complete details on its physical and chemical characteristics and manufacturing are given by (Li et al., 2018). Key properties of the biochars are listed in Table 2.

2.2. Incubations

Incubations were performed in 1 quart Mason jars, each containing the appropriate soil (50 g) and other additives (Tables S1 and S2). All treatments, except appropriate controls, received a fertilizer solution (2.08 mL) with a C:N:P ratio of 800:13.3:1 made from KH_2PO_4, K_2HPO_4, and NH_4NO_3. This nutrient ratio was calculated using an established method that takes into account the soil organic carbon content (Infante et al., 2010). Water was added at the start of the incubations to achieve 75% of the maximum water holding capacity (MWHC) of the soils. This water content was maintained for the duration of the study through weekly adjustments. After incorporation of the biochar (see Table S1), the jars were capped and placed in a randomized block design in an incubator at 25 °C (Soil A, biochar selection trial) and 30 °C (Soil B and C, main experiment) in the dark and without further mixing during the study to mimic a no-till situation in the field. All treatments were conducted in triplicate. After destructive sampling, subsamples of the soils were immediately transferred to volatile organic carbon (VOC) sample holders and stored at 4 °C before analysis for TPH following EPA methods 8015M and 9071B. Short-term testing of 60 days was performed on Soil A to select a biochar, and long-term testing of 230 days was performed on Soil B and Soil C to study the efficacy of the selected biochar.

2.2.1. Biochar selection

Incubations to compare the efficacy of the WA900 and P900 biochars were performed over a period of 60 days with Soil A. The treatments included soil only (no amendments); soil plus fertilizer; biochar (no soil) plus fertilizer; and soil plus biochar at two different rates (5% and 10% by dry weight) plus fertilizer. Experimental controls of 5% biochar and an antibacterial treatment (ABT) containing sodium azide and 10% ethanol were also included. The specific compositions of the treatments are shown in Table S1. An adequate number of microcosms were setup to allow for destructive sampling after 10, 30 and 60 days.

2.2.2. Biochar efficacy of light and heavy weathered crude contaminated soils

For this efficacy trial, the best performing biochar selected based on the 60 day incubations, shown in Fig. 1, (P900) was incubated with Soil B and Soil C, soils contaminated with light and heavy weathered crude oil, respectively, for 230 days. In this case, the efficacy of the biochar was compared with a soil bulking agent (pine wood chips, 5% by dry weight). The specific composition of the treatments is shown in Table S2. An adequate number of microcosms were setup to allow for destructive sampling after 30 60, 90, 150, and 230 days.

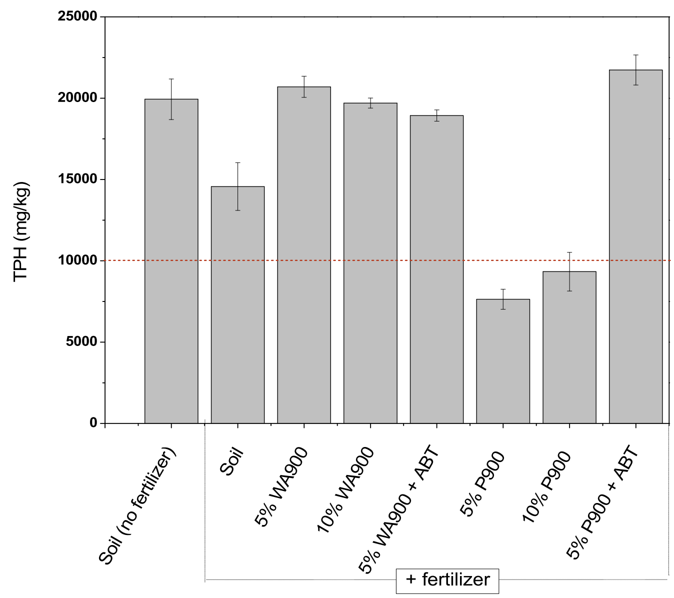

Fig. 1. Change in TPH concentration of Soil A (light crude) amended with two biochars (WA900 and P900) after 60 days of incubation to assess biochar efficacy. All samples were maintained at 55–60% MWHC and received fertilizer except the one represented by the far left bar. Averages and standard errors of the triplicate incubations are shown. Dotted line at 10,000 mg/kg TPH indicates typical remediation target level. WA900: walnut shell biochar (900 °C); P900: ponderosa pine biochar (900 °C); ABT: antibacterial treatment.

2.3. Analyses of total petroleum hydrocarbons (TPH) in soil or water

Analysis of TPH as motor oil and diesel from hexane soil extracts was performed by Kiff Analytical (Davis, CA) following EPA methods 8015M and 9071B. According to this methodology, TPH is a measure of a specific fraction of solvent extractable organics present, not necessarily the sum of all hydrocarbons, and does not differentiate between breakdown products or bioavailable and non-bioavailable hydrocarbons that may still have been extractable in the TPH test.

3. Results and discussion

Table 2 shows that the two biochars had similar pH values, but differed markedly in their ash content, % C, C/N ratio, H/C ratio, and surface area. As mentioned by Mukome et al. (2013), the basicity is related to ash content of the biochar; the C/N ratio gives the nutrient ratio, and the H/C ratio is a proxy for the relative aromaticity of the biochars. The biochars were selected to compare biochar made at similar temperature but from different feedstocks. High temperature pyrolysis leads to lower biochar yield, higher surface area, higher pH, higher ash content, lower surface charge, higher percent C in the biochar, and increased aromaticity. The C content of biochars can range widely (36–94%; dependent on feedstock and charring temperature), with C content increasing with higher pyrolysis temperatures (Novak et al., 2009a,b; Keiluweit et al., 2010). Mackay and Roberts (1982) found that the micropore system of chars is established by 500 °C but access to internal pore volume is restricted in such chars by mass that would be volatilized at higher temperatures.

3.1. Biochar comparison incubations

The TPH data from incubations with Soil A (Fig. 1) indicate that the presence of pinewood biochar (P900) biochar can enhance TPH reduction in this soil contaminated with light crude oil. However, the walnut shell biochar (WA900) was ineffective – or perhaps even inhibitory when compared to soil with fertilizer, as all samples with WA900 had TPH levels close to the soil control with no fertilizer. In another study, it was shown that WA900 had a much high higher aryl hydrocarbon receptor dependent gene expression, compared to P900, indicating the possible presence of dioxins or polycyclic aromatic hydrocarbons that could impact microbial activity (Li, 2018). This result highlights how different biochars have varied physical and chemical properties which impact the reactions that can take place in the soil. Thus, screening of biochars for specific outcomes is important before selecting biochars for any given purpose.

No significant reduction (~1%) in TPH was observed in the P900 abiotic control (5% P900 + ABT; Fig. 1) indicating that biodegradation is the main process responsible for the decrease in soil TPH concentrations. Additionally, CO_2 emissions were much greater in the P900 treatment, than the WA900 (Fig. S1), further suggesting an inhibitory impact on microbial activity with WA900 (Shang, 2015). The addition of P900 biochar and fertilizer was more effective in reducing TPH than the addition of fertilizer alone over the course of the 60-day experiment. This could suggest that P900 stimulates biodegradation of oil components, however no direct microbial analyses were performed. Biochar addition to soil has previously been shown to increase microbial activity and biomass in some cases (Wardle et al., 2008; Kolb et al., 2009), but to decrease them in others (Dempster et al., 2012; Liang et al., 2010). This varied impact supports our hypothesis that P900 was favorable for microbial activity and WA900 was inhibitory when incubated with Soil A.

The TPH results from the current study reveal that after 60 days, treatments with 5% and 10% P900 were below the United States Environmental Protection Agency (USEPA) regulatory soil clean-up goal of 10,000 mg/kg (USEPA, 1996). Given the apparent lack of additional benefit of adding a greater amount of biochar, and the poor performance of WA900, 5% P900 was selected for subsequent experiments.

3.2. Efficacy of biochar in remediation of light and heavy weathered crude contaminated soils

The changes in TPH after 30 and 230 days in different microcosms are shown in Fig. 2a and b for Soils B and C, respectively. TPH reduction is most extensive for soils contaminated with light (Soil B) than heavy (Soil C) in all microcosms tested. This is expected, as light crude oils are more biodegradable than heavy crude oils (McMillen et al., 2001). They further show that, for both soils, the 5% P900 in combination with fertilizer was the most effective in reducing TPH concentrations, particularly at the 30 day sampling point. Indeed, for soil B TPH concentrations reached the US EPA clean-up standard of 10,000 mg/kg after 30 days and soil C reached this threshold after 230 days. The more rapid reduction of TPH in the light crude contaminated soil is in consistent with prior studies. According to Leahy and Colwell (1990), weathering of oil occurs through the depletion of the aliphatic or light aromatic fractions, leaving behind viscous fractions enriched in high-molecular-

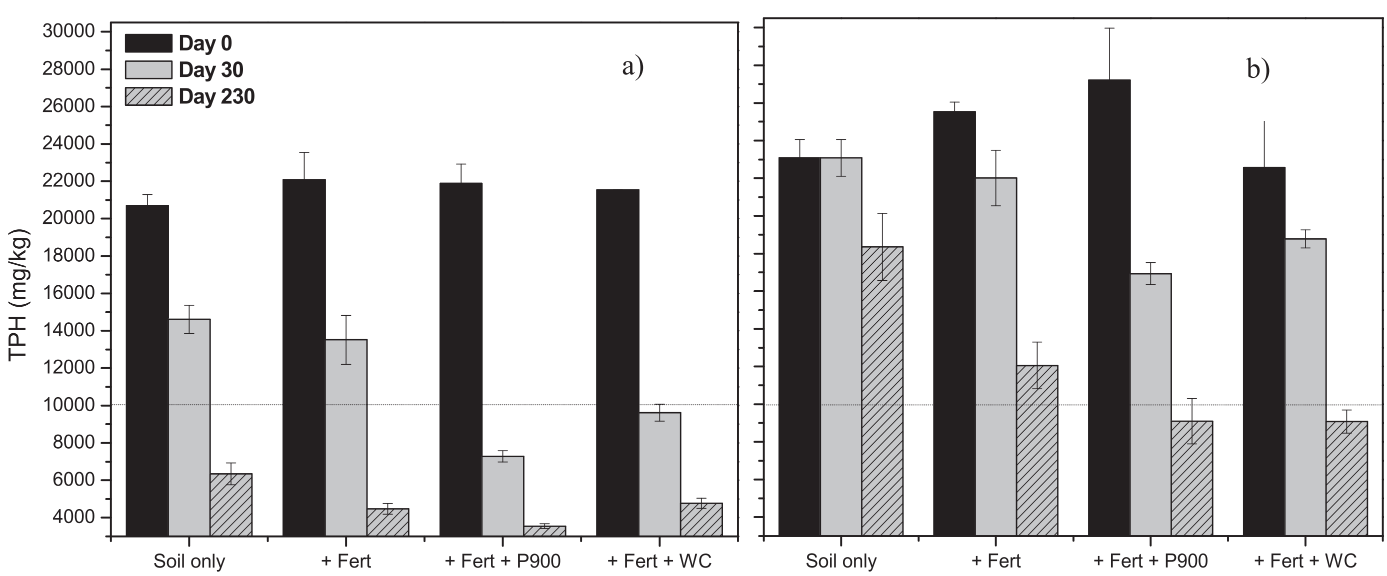

Fig. 2. Change in TPH concentration in a) Soil B (light crude) and b) Soil C (heavy crude) after a 30 and 230 day incubation period. Averages and standard errors of the triplicate incubations are shown. Dotted line at 10,000 mg/kg TPH indicates remediation target level. Fert: fertilizer, P900: ponderosa pine biochar (900 °C), WC: ponderosa pine wood chips.

weight aromatics, polar resins, and asphaltenes that are considered more recalcitrant to biodegradation.

The incubation results also show nutrients are critical for sustaining rapid HH degradation regardless of crude oil type. The role of fertilizer, as nutrients for microbial communities, is particularly evident in the comparison of treatments of the soil contaminated with the heavy crude (Soil C) which shows a significant difference in the final soil TPH after 230 days between the soil only and the soil plus fertilizer cases (Fig. 2b). Other studies have also shown that certain types of fertilizers enhance oil remediation on impacted beaches (Jiménez et al., 2006; Nikolopoulou et al., 2007). In a study of a coal tar-containing soil near a manufactured gas plant, biodegradation of PAHs by indigenous microorganisms was greatly accelerated by addition of inorganic nutrients (N, P, K, and trace metals) (Li et al., 2005). Since the soil had been impacted for many decades, these results suggest that inorganic nutrient limitation may have been a key factor in the persistence of these compounds, and that an *in situ* remediation approach might benefit from nutrient supplementation (N, P, K, and trace metals). However, it should be noted that optimized biodegradation often requires soil aeration and balancing of moisture content, so that addition of fertilizer alone may be insufficient to promote microbial activity (Brown et al., 2017). Thus, if biochar amendment technology is utilized in the field, periodic fertilizer reapplication, tilling, and watering may be required for optimal efficacy.

Bulking or composting is often utilized to facilitate bioremediation of petroleum contaminated soils in techniques called biopiles (Hazen et al., 2003). For example, field studies of biopiles utilizing mature compost only as bulking agents showed a 52% reduction in TPH and 74–82% reduction when combined with an inoculation of a commercial consortia of microbes (Gomez and Sartaj, 2014). Woodchips are typically a readily-available and inexpensive bulking agent that improves air exchange and makes conditions more favorable for microbial activity. We conducted experiments using the same woodchips used to make P900 as a bulking agent (WC). The results show that WC was less effective than P900 in combination with fertilizer for Soil B (Figs. 2 and 3), but was comparably effective for Soil C, suggesting that aeration may be a more important factor for soils impacted heavy crude oils. However, it should be noted that the heavy hydrocarbons in Soil C are more difficult to degrade than Soil B; at 230 days TPH levels in soil C remained substantially higher for all treatments compared to Soil B. Fig. 3 shows more clearly, over the course of 230 days, the benefical effect of fertilizer on TPH reductions. For the light crude oil impacted soils P900 is more effective than WC in reducing TPH over the first 90 days, suggesting that P900 could be used to increase initial landfarming efficiency. For the heavy crude oil, the fertilizer combined with biochar performed better than the other treatments at all time points, with the difference being again most pronounced at the early points. Finally, it is notable that P900 plus fertilizer amendment resulted in a faster initial rate of TPH reduction (≤30 d) than either fertilizer alone or WC plus fertilizer. The change in degradation rates is likely correlated with nutrient availability, and it is possible that a second addition of fertilizer between 30 and 230 days may have been beneficial in increasing TPH reduction in all fertilizer treatments.

Although use of the pine wood biochar was effective in our study, a recent bioremediation study utilizing wheat straw biochar observed no significant difference between the control of crude oil spiked soil and the treatment with added 1% biochar in overall TPH removal (Han et al., 2016). The high ash content and low surface area of the biochar may have impacted its effectiveness. Also, the biochar content was much lower than used here. This is also similar to our experiments with the WA900 biochar, which showed no benefits in lowering TPH in Soil A. Results such as these emphasize the need for more studies of the importance of feedstock, pyrolysis temperature, and amendment rate.

4. Conclusion

Biochar amendment has potential as a simple and sustainable remediation strategy for shallow soils impacted by weathered crude. This study demonstrated a significant difference in rates of achievement of the US EPA clean up standard of 10,000 mg/kg TPH in soil by biochar co-amended with fertilizer compared to fertilizer alone and bulking agents with fertilizer, particularly for soils containing light crude oil. However, the efficacy of the individual biochars must be carefully ascertained prior to deployment for this type of strategy as some have the potential of inhibiting TPH degradation. Additional studies are warranted to investigate the

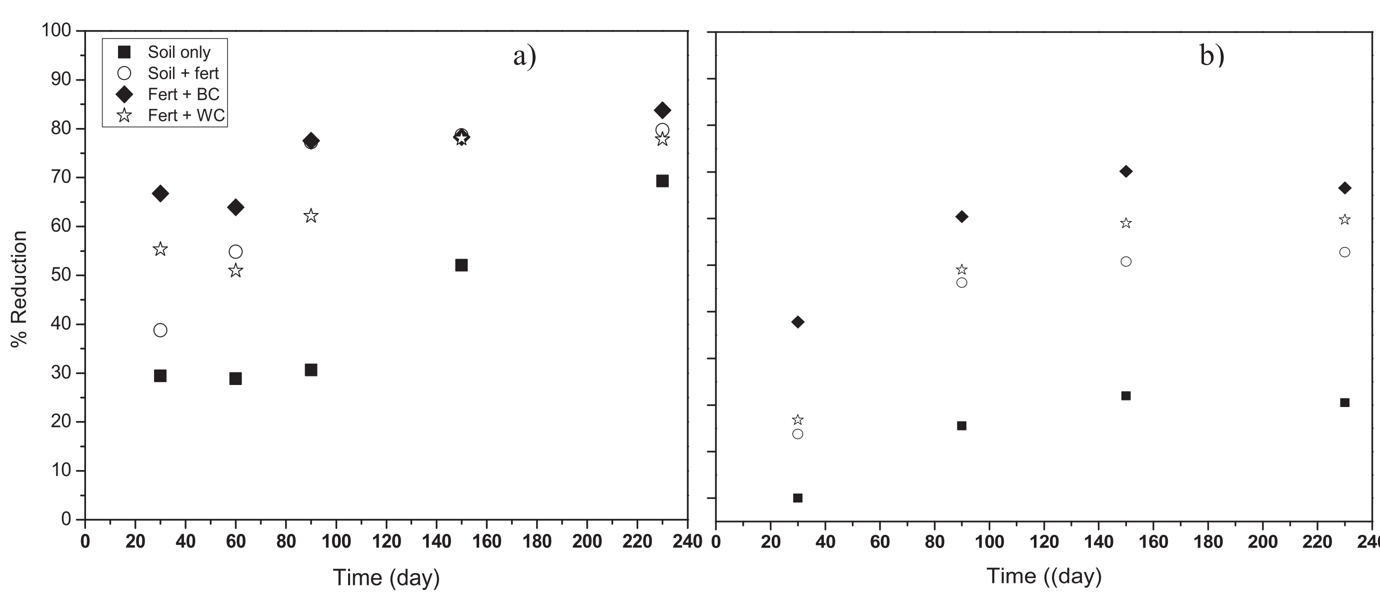

Fig. 3. Percentage reduction in TPH concentration in a) Soil B (light crude) and b) Soil C (heavy crude) over 230 days of incubation. BC is P900 and WC is wood chips.

ideal biochar addition ratio, feedstock, and pyrolysis conditions. Future studies aimed at optimizing biochar amendments for other soils, climates, and available biochars would be highly beneficial.

Credit author statement

Fungai N.D. Mukome: Methodology, Investigation, Visualization, Writing - original draft. **Maya C. Buelow:** Methodology, Investigation, Visualization, Data curation. **Junteng Shang:** Methodology, Investigation. **Juan Peng:** Methodology, Investigation, Writing - review & editing. **Michael Rodriguez:** Methodology, Investigation. **Douglas M. Mackay:** Methodology, Investigation, Project administration, Writing - review & editing, Funding acquisition. **Joseph J. Pignatello:** Methodology, Investigation, Writing - review & editing, Funding acquisition. **Natasha Sihota:** Grants program administration, Writing - review & editing. **Thomas P. Hoelen:** Grants program administration, Writing - review & editing. **Sanjai J. Parikh:** Conceptualization, Writing - original draft, Writing - review & editing, Methodology, Investigation, Visualization, Resources, Supervision, Project administration, Funding acquisition.

Declaration of competing interest

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

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.envpol.2020.115006

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