

ORIGINAL RESEARCH ARTICLE

Environment

A bioassay of long-term stockpiled salvaged soil amended with biochar, peat, and humalite

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Funding information

Imperial Oil Resources Ltd

Abstract

Long-term stockpiling of soil salvaged from oil sands operations often leads to the deterioration of soil quality and poor reclamation results for disturbed land. This study assessed the efficacy of organic amendments in improving the quality and productivity of long-term stockpiled salvaged soil in Canada's Alberta oil sands region (AOSR). Dry soil samples (4.2 kg) from a 24-yr-old stockpile were placed into 5-L pots and amended with biochar, humalite, peat, and 50:50 mixtures of biochar and peat (BCP) and biochar and humalite (BCH) at rates corresponding to 0, 6.55, 13.1, and 26.2 g C kg⁻¹ soil. The pots were seeded with barley (*Hordeum vulgare* L.), fertilized, and placed in a growth chamber. Plants were allowed to grow for three crop cycles of 45 to 59 d each and harvested at the end of each cycle. Results showed that biochar and peat increased dry matter yield (DMY) by 38 and 40%, respectively, compared with the unamended soil. Humalite produced the highest N and P concentrations in plant tissue, but this did not translate to an increase in DMY. Biochar and peat offer the greatest promise for improving the productivity of long-term stockpiled salvaged soil, thereby enhancing the success of reclamation of disturbed sites.

1 | INTRODUCTION

Soil salvaging and stockpiling are techniques implemented to conserve topsoil for future reclamation of disturbed land. However, extended stockpiling often results in the quality of the salvaged soil deteriorating (Kundu & Ghose, 1997), partly due to the halt in organic matter inputs, which impacts the functioning of almost all aspects of

the soil (Larney & Angers, 2012). Microbial communities shift to anaerobically dominant ecosystems, which favors the accumulation of ammonium nitrogen (NH₄-N) (Williamson & Johnson, 1990). When stored soils are once again aerated, NH₄-N is rapidly transformed to NO₃-N, which is highly mobile in the soil and easily leached from the solum (Williamson & Johnson, 1990). Kundu and Ghose (1997) observed a significant reduction in the quality of a 1-yr stockpiled salvaged soil at an opencast coal mine site in southwestern Germany. They reported reductions of 47% in soil organic carbon (SOC), 31% in available N, 23% in available P, and 28% in extractable K during the first year of stockpiling. After 6–10 yr, these soil quality

Abbreviations: AOSR, Alberta oil sands region; BCH, 50:50 biochar/humalite mixture; BCP, 50:50 biochar/peat mixture; CEC, cation exchange capacity; DMY, dry matter yield; SOC, soil organic carbon; SOM, soil organic matter; WFPS, water-filled pore space.

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indicators had decreased by between 89 and 96% (Kundu & Ghose, 1997).

Organic amendments, such as peat, biochar, and humalite, have been used to improve soil quality (Hemstock et al., 2010; Turgay, Karaca, Unver, & Tamer, 2011; Zhang et al., 2012). However, the efficacy of these amendments depends on several factors. For example, the performance of peat can be affected by its biological and chemical composition, available nutrient mobilization, and mineral additives (Pietola & Tanni, 2003; Vestberg et al., 2009). Similarly, the feedstock, pyrolysis, rate of application, and nutrient additives have been shown to significantly impact biochar effectiveness (Jones, Rousk, Edwards-Jones, DeLuca, & Murphy, 2012; Major, Rondon, Molina, Riha, & Lehmann, 2010). Humalite is a concentrated form of fulvic and humic acids, with humic acid content ranging from 30 to 80% and overlays more compacted coal in coal mines; it has recently become a popular amendment for improving soil productivity (Sanli, Kardogan, & Tonguc, 2012; Turgay et al., 2011). However, unlike peat and biochar, it may not be readily available locally, which may result in prohibitive transportation and handling costs. Although these amendments have been used to improve soil quality, their effectiveness in improving the quality and productivity of stockpiled salvaged soils in cold climate regions is not well understood.

In the AOSR, there are more than $144 \times 10^6 \text{ m}^3$ of stockpiled salvaged soils (Laskosky, 2015). A better understanding of how organic amendments improve soil quality and productivity is vital to the successful reclamation of the disturbed lands in the region. Therefore, the overall objective of this study was to characterize the effectiveness of biochar, humalite, and peat in improving the quality and productivity of a long-term stockpiled salvaged soil from the AOSR.

2 | MATERIALS AND METHODS

2.1 | Soil sampling and preparation

Soil samples were collected from a 24-yr-old, 25 m W \times 50 m L \times 4 m H, stockpiled salvaged soil on Imperial Oil Limited's Cold Lake, AB, Canada Operations site (54°36' N, 110°28' E). The stockpiled soil was salvaged from the O and Ae horizons of an Orthic Gray Luvisol (Boralfs) on a 1-ha well pad. Salvage was conducted with bulldozers, which salvaged soil to the bottom of the A horizon (~20 cm in depth). The soil was then windrowed immediately outside the salvage area. Samples were taken from the 2- to 3-m layer of the stockpile using a small excavator (Hitachi ZAXIS33U) and placed into 20-L plastic pails that were then sealed with lids for transportation. The soil samples were screened through an 11-mm sieve and then

Core Ideas

- Biochar and peat improved the productivity of long-term stockpiled salvaged soil.
- Humalite produced the highest N and P concentrations in plant tissue.
- High soil NO_3^- concentrations from biochar application will assist initial revegetation.

composited and stored in pails at 20 °C for approximately 30 d until the start of the experiment.

2.2 | Laboratory analysis of soil

Initial soil NO_3^- -N concentration in the stockpiled salvaged soil (results shown in Table 1) was determined in 2 M KCl extracts with the Cd reduction method (Mulvaney, 1996) using a Lachat QuikChem 8500 Flow Injection Analyzer. Ammonium N was also determined using the same auto analyzer but a different channel, following extraction with 2 M KCl. Olsen P concentration was determined by the ammonium molybdate method following extraction with 0.5 M sodium bicarbonate at a pH of 8.5 (Olsen, Cole, & Watanabe, 1954), and then measured using an Utraspec 2100 Pro Spectrophotometer. Soil organic matter (SOM) concentration was determined via the loss on ignition method (Nelson & Sommers, 1996) using a Thermo Scientific Thermolyne muffle furnace, while pH was determined in a 1:1 soil/water suspension with a pH electrode (AB15–Fisher Scientific). Soil organic C concentration was estimated by dividing SOM by 1.72. Calcium, Mg, and Na were determined by atomic absorption spectroscopy (iCE 3300 AAS; Thermo Scientific) in ammonium acetate extracts. Micronutrients (Fe, Mn, Cu, and Zn) were extracted with 0.005 M DTPA and their concentrations measured using atomic absorption spectroscopy, while B was determined by the hot water extraction method (Keren, 1996). Cation exchange capacity was estimated by summing cation concentrations in the ammonium acetate extracts described above. Container moisture capacity of the soil was determined following addition of varying amounts of moisture to 100 g aliquots of the soil in clear plastic pill bottles and allowing the moist soils to sit for 24 h to allow for moisture equilibration (White & Mastalerz, 1966).

2.3 | Amendments and their chemical composition

The amendments used to improve stockpiled salvaged soil quality in this study were biochar, humalite, peat,

TABLE 1 Selected baseline chemical properties of the stockpiled, salvaged soil

pH	SOM	SOC	NO ₃ -N	NH ₄ -N	Olsen P	K	Cl	S	B	Zn	Fe	Mn	Cu	Mg	Ca	Na	CEC
	—g kg ⁻¹ —				mg kg ⁻¹ —												
7.5	25	14.5	1.5	1.4	15	117	4.5	8	0.8	2.1	157	56	1.2	244	1,633	14	10.6

Note. SOM, soil organic matter; SOC, soil organic carbon; CEC, cation exchange capacity.

TABLE 2 Initial chemical properties of the organic amendments added to stockpiled, salvaged soil

Amendment	pH	TOC	N	P	K	S	Ca	Mg	CEC	CCE	SAR	C/N ratio
		—g kg ⁻¹ —		mg kg ⁻¹ —					cmol _c kg ⁻¹	g kg ⁻¹		
Biochar	7.2	648	0.4	67	1,282	90	3,350	722	1.7	3	0.21	1,620
Humalite	4.3	375	10.7	103	175	4,169	14,136	2,140	21	2	5	35
Peat	4.5	273	7.2	157	6.0	792	7,490	1,076	4.6	3	0.18	38

Note. TOC, total organic carbon; N, total nitrogen; CEC, cation exchange capacity; CCE, calcium carbonate equivalent; SAR, sodium adsorption ratio.

a 50:50 mixture of biochar and humalite (BCH), and a 50:50 mixture of biochar and peat (BCP). Roller-milled biochar produced from Douglas fir [*Pseudotsuga menziesii* (Mirbel) Franco], white spruce [*Picea glauca* (Moench) Voss], and various pine species (*Pinus* spp.) waste wood from logging operations was acquired from Alterna Bio-carbon. The waste wood was pyrolyzed at a temperature of 380 °C and then immediately cooled with a water spray following carbonization. Humalite was acquired from Black Earth Humic LP (Calgary, AB). Peat (Sunshine horticultural peat) was obtained from Sun Gro Horticulture (Vancouver, BC) and was fibric in nature. The initial chemical composition of the amendments before adding to the stockpiled salvaged soil samples for assessment are presented in Table 2.

2.4 | Experimental setup

Air-dry soil samples (4.2 kg per pot) were thoroughly mixed with each of the five amendment treatments at equivalent rates of 0, 6.55, 13.1, and 26.2 g C kg⁻¹ (Table 3). The amended soils (including control) also received 0.143 g urea (34 mg kg⁻¹ soil), 0.338 g mono-ammonium phosphate (80 mg kg⁻¹ soil), and 0.787 g potassium sulfate (187 mg kg⁻¹ soil), after which they were placed into 5-L pots and seeded with 15 barley (*Hordeum vulgare* L.) seeds that were randomly distributed and placed at least 2 cm apart.

The pots were watered with reverse osmosis water to attain a water content of 0.230 kg H₂O kg⁻¹, which was equivalent to 50% water-filled pore space (WFPS), corresponding to the container capacity of the soil (White & Mastalerz, 1966). The seeded pots were then placed in a walk-in growth room that was maintained at 22 °C during

the 16-h photoperiod and 15 °C during the 8-h dark period. Light intensity in the growth room was 270 μmol m⁻² s⁻¹ during the photoperiod, while relative humidity was set at 65% at all times.

The pots were weighed every 2–3 d and watered to replace any moisture lost via evapotranspiration. Plants were allowed to grow until full heading, after which they were harvested by clipping the aboveground biomass at the soil surface. The cycle was repeated two more times, to give a total of three cycles (Cycle 1, Cycle 2, Cycle 3), each of which was 45–59 d long to mimic three growing seasons. The experimental design was a 5 × 3 factorial plus one

TABLE 3 Carbon and amendment rates applied to the stockpiled, salvaged soil

Amendment	Humalite or peat Biochar		
	—g kg ⁻¹ soil—		
Control	–	–	–
Biochar	6.55	–	10.1
50:50 Biochar/Humalite	–	8.8	5.1
50:50 Biochar/peat	–	12.0	5.1
Humalite	–	17.5	–
Peat	–	24.0	–
Biochar	13.1	–	20.2
50:50 Biochar/humalite	–	17.6	10.1
50:50 Biochar/peat	–	24.0	10.1
Humalite	–	35.0	–
Peat	–	48.0	–
Biochar	26.2	–	40.4
50:50 Biochar/humalite	–	35.2	20.1
50:50 Biochar/peat	–	48.0	20.1
Humalite	–	70.0	–
Peat	–	96.0	–

control, corresponding to five amendments, three amendment rates, and one non-amended control. All treatments were replicated three times, for a total of 48 experimental units (pots).

Between cycles and following harvest, a 10-d rest period was allowed, during which the uncropped pots were stored in the growth chamber. Roots from the previous crop were retrieved from the potted soil, cut into approximately 0.5- to 1-cm pieces, and uniformly mixed back into the soil prior to the start of the next crop cycle.

A micronutrient mixture consisting of 8 mg kg⁻¹ Zn, 7 mg kg⁻¹ Mn, 1 mg kg⁻¹ Mo, 5 mg kg⁻¹ B, 20 mg kg⁻¹ Cu, 26 mg kg⁻¹ Fe, and 203 mg kg⁻¹ S was added to each pot at the start of Cycle 2, along with the same rate of urea applied in Cycle 1, to prevent nutrient deficiencies in subsequent cycles. The same rate of urea was also applied at the beginning of Cycle 3.

2.5 | Determination of dry matter yield and chemical composition

Aboveground biomass was harvested at the end of each cycle to determine dry matter yield (DMY). The harvested samples were immediately weighed and then oven-dried at 60 °C for 48 h. The oven-dried samples were weighed to determine the DMY. Subsamples of the biomass were ground, passed through a 2-mm sieve, and analyzed for total N and total P concentrations. Total N concentration was determined by dry combustion with an Elementar Vario Max CN analyzer. Total P concentration in the plant tissue was determined in nitric acid and hydrogen peroxide digests using a Perkin Elmer 5400 ICP.

2.6 | Soil chemical composition after harvest

Twenty-gram soil samples were collected from each pot immediately after harvest at the end of each crop cycle. The samples were analyzed for concentrations of NO₃-N, NH₄-N, Olsen P, extractable cations (K, Ca, Mg, and Na), and CEC as described above.

2.7 | Statistical analysis

Analysis of variance was performed on all data using the generalized linear mixed models procedure (PROC GLIMMIX) of SAS 9.4 (SAS Institute, 2013). Amendment and rate were modeled as fixed effects, while sampling time was modeled as a repeated measures factor using the com-

pound symmetry (CS) covariance structure. Treatment means were compared using the Tukey–Kramer multiple comparison procedure. Treatment differences were considered significant if $P < .05$.

3 | RESULTS

3.1 | Dry matter yield

There was a significant amendment × cycle interaction where peat produced 24% greater DMY than BCP in Cycle 1, averaged across rates, but there were no significant differences between any of the other treatment pairs (Table 4, Figure 1a). In Cycle 2, peat produced 174% greater DMY than the control, whereas DMY did not differ significantly between the other four amendments and the control. For all amendments, there was an overall decreasing trend in DMY across the three growth cycles. In Cycle 2, DMY was 50–80% lower than in Cycle 1 for all amendments and the control, with the DMY lowest for the control, and the least difference occurring in the BCP and peat treatments. In Cycle 3, DMY was 65–71% lower than in Cycle 1, with the smallest difference occurring again for the BCP treatment and the highest difference occurring for the humalite and peat treatments.

Cumulative DMY was 38% greater with biochar and 40% greater with peat than the unamended soil (Table 4). Peat produced 22% more cumulative DMY than humalite, but peat and humalite did not differ significantly from the other amendments with respect to cumulative DMY.

3.2 | Aboveground plant nitrogen uptake

Amendment effects varied with cycle, as indicated by the significant amendment × cycle interaction (Table 4, Figure 1b). In Cycle 1, N uptake was 49% higher for peat than the control, while N uptake from the other amendments did not differ from the control in any of the cycles. Among the amended soils, there were no significant amendment differences within the cycles, except in Cycle 1, where N uptake was 35% greater for peat than for biochar.

Plant N uptake was significantly greater for the 6.55 g C kg⁻¹ peat (46% more N uptake), 13.1 g C kg⁻¹ peat (60%), and 13.1 g C kg⁻¹ HU (74%) treatments than the control (Table 4, Figure 2). When the rates were compared within each amendment, only humalite showed significant differences, with the 13.1 g C kg⁻¹ rate producing 44% greater N uptake than the 6.55 g C kg⁻¹ rate and 52% greater N uptake than the 26.2 g C kg⁻¹ rate. Among the amendments, humalite produced 41% greater plant N uptake than biochar at the 13.1 g C kg⁻¹ rate.

TABLE 4 Amendment, rate, and cropping cycle effects on dry matter yield (DMY) and residual soil chemical properties

Amendment	DMY	Total DMY	Plant N	Plant P	pH	NO ₃ -N	NH ₄ -N	Olsen P	CEC
	g pot ⁻¹		mg pot ⁻¹			mg kg ⁻¹			cmol _c kg ⁻¹
Biochar	12.4ab ^a	37.3ab	120	24.0	7.25	2.91	16.8	14.2	10.9
Humalite	10.3c	31.0bc	131	21.4	6.40	0.59	19.4	13.8	13.4
Peat	12.7a	38.0a	144	23.5	6.55	0.72	15.6	13.4	12.5
BCH	10.5c	31.5abc	119	20.0	6.82	1.81	16.6	13.9	12.0
BCP	10.8bc	32.3abc	121	21.3	6.90	1.65	16.8	13.7	11.2
Control	9.0c	27.1c	96.4	19.1	7.21	0.83	22.5	16.9	11.3
Rate									
0	9.0	27.1	96.4	19.1ab	7.21	0.83	22.5	16.9	11.3
6.55	11.4	34.2	124	21.9ab	7.08	1.20	18.3	15.0	11.6
13.1	11.5	34.6	140	23.6a	6.85	1.61	16.8	14.4	12.3
26.2	11.1	33.2	117	20.7b	6.42	1.80	16.1	12.0	12.1
Cycle									
1	19.8a	–	247	34.3	6.66b	1.49	17.5a	16.2	11.9ab
2	7.9b	–	80.9	17.5	6.71b	1.67	13.3b	14.1	12.4a
3	6.3b	–	53.7	14.4	6.97a	1.46	20.3a	13.1	11.7b
P value									
Amendment (A)	<.001	.007	.002	.017	<.001	<.001	.08	.68	<.001
Rate (R)	.62	.73	<.001	.02	<.001	<.001	.08	<.001	.02
A × R	.92	.97	.04	.53	<.001	<.001	.91	.01	.02
Cycle (C)	<.001	–	<.001	<.001	<.001	.03	<.001	<.001	.04
A × C	.04	–	.002	.042	.78	.58	.65	.22	.52
C × R	.07	–	.37	.54	.06	<.001	.43	.02	.10
A × R × C	.10	–	.81	.53	.31	.54	.58	.67	.86

Note. BCH, 50:50 biochar/humalite by mass (based on C content); BCP, 50:50 biochar/peat by mass (based on C content); CEC, cation exchange capacity; Total DMY, cumulative dry matter yield over three growth cycles.

^aMeans in the same column followed by the same letter are not significantly different at $\alpha = .05$ according to the Tukey multiple comparison procedure. Significant interactions are plotted in Figures 1–4.

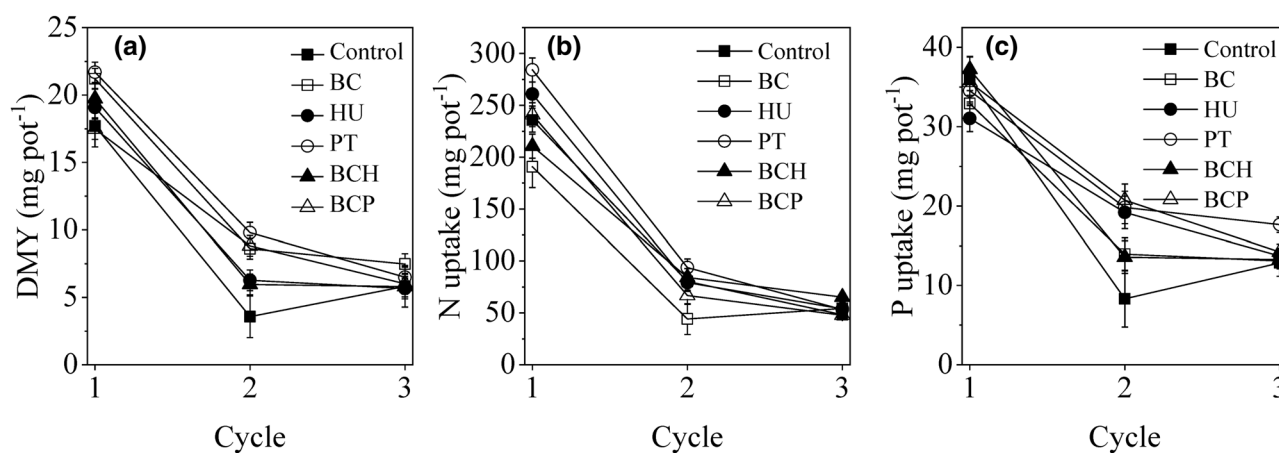


FIGURE 1 Barley dry matter yield (DMY) as affected by amendment application and cropping cycle. The amendments are biochar (BC), humalite (HU), peat (PT), a 50:50 biochar/humalite mixture (BCH), a 50:50 biochar/peat mixture (BCP), and a non-amended control. Vertical bars represent standard errors of the mean

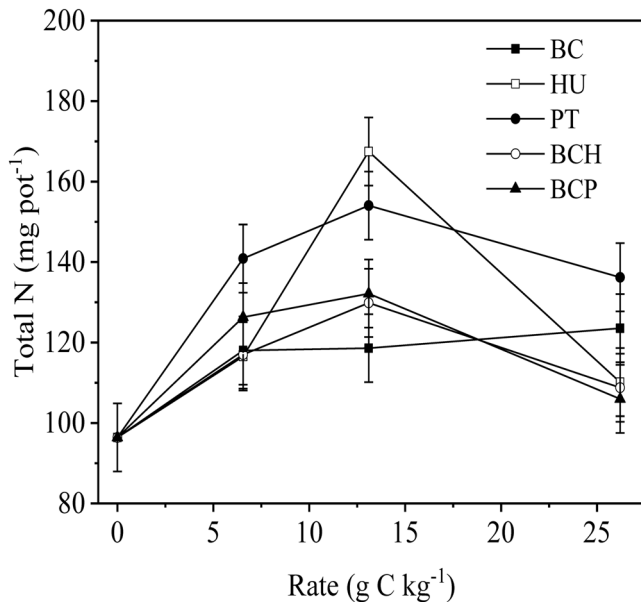


FIGURE 2 Plant N accumulation in barley plants as affected by amendment type and rate. The amendments are biochar (BC), humalite (HU), peat (PT), a 50:50 biochar/humalite mixture (BCH), and a 50:50 biochar/peat mixture (BCP). Vertical bars represent standard errors of the mean

3.3 | Aboveground plant phosphorus uptake

Amendment effects on plant P uptake varied with cycle, as indicated by the significant amendment \times cycle interaction (Table 4, Figure 1c). Phosphorus uptake decreased by between 62 and 174% in Cycle 2 compared with Cycle 1 across the five amendments and averaged across rates, while the control showed a 333% decrease in P uptake in Cycle 2. Overall, P uptake decreased by 96–181% in Cycle 3 relative to Cycle 1. The rate main effect was also significant, which was due to the 13.1 g C kg⁻¹ rate having a 14% greater P uptake than the 26.2 g C kg⁻¹ rate.

3.4 | Residual soil nitrate and ammonium

Amendment effects on residual soil NO₃-N concentration varied with amendment rate ($P < .001$ for the amendment \times rate interaction) (Table 4, Figure 3a). While biochar at all rates and BCH and BCP at the 13.1 and 26.2 g C kg⁻¹ rates produced significantly higher soil NO₃-N concentrations than the control across the three cycles, NO₃-N concentrations in soils amended with humalite and peat did not differ significantly from those in the control. Nitrate-N concentration in biochar-amended soil, across the rates,

was 140–333% higher than that in the control, with the 26.2 g C kg⁻¹ rate giving the highest concentration, while BCH and BCP produced 120–180% higher NO₃-N concentration than the control. Nitrate-N concentration was significantly greater for biochar than all the other amendments at all rates. The largest differences were between biochar vs. peat and humalite. Increasing the amendment rate from 0 through 6.55–13.1 g C kg⁻¹ resulted in a linear increase (56–83%) in NO₃-N concentration for biochar and, to a lesser extent, for BCH and BCP; however, doubling the amendment rate from 13.1 to 26.2 g C kg⁻¹ produced a slight and non-significant increase in the NO₃-N concentration.

Amendment rate effects on NO₃-N concentration varied with crop cycle ($P < .001$ for the rate \times cycle interaction; Table 4). In Cycle 1, NO₃-N concentration increased significantly with each successive increase in amendment rate, with the increase greater through the 13.1 g C kg⁻¹ rate (Figure 4a). Nitrate concentrations in Cycle 2 were, on average, greater than in Cycles 1 and 3 at amendment rates up to 13.1 g C kg⁻¹, but slightly lower at the 26.2 g C kg⁻¹ rate. There were no significant differences in NO₃-N concentration between Cycles 1 and 3 at any of the amendment rates.

Amendment type and rate main and interaction effects were not significant for NH₄-N concentration (Table 4). However, there was a significant main effect of crop cycle ($P < .001$), which was due to the NH₄-N concentration being significantly lower in Cycle 2 than in the other two cycles. Ammonium N concentration did not differ significantly between Cycles 1 and 3.

3.5 | Residual soil Olsen phosphorus

There was a significant amendment \times rate interaction for Olsen P concentration (Table 4, Figure 3b). Olsen P concentration decreased by 30–35% with peat, humalite, BCH, and BCP application at the 26.2 g C kg⁻¹ rate relative to the control, whereas biochar showed no significant rate effect. Among the amendments, humalite produced 28% lower Olsen P concentration than biochar at the 26.2 g C kg⁻¹ rate, but there were no significant amendment differences at the other rates.

Amendment rate effects on Olsen P concentration varied with cycle ($P = .02$ for the cycle \times rate interaction; Table 4, Figure 4b). In Cycle 1, amendment rate had no significant effect on Olsen P concentration. However, in Cycle 2, Olsen P concentration decreased by 38% at the 26.2 g C kg⁻¹ rate relative to the control. In Cycle 3, Olsen P concentration was 23–38% lower with amendment application compared with the control.

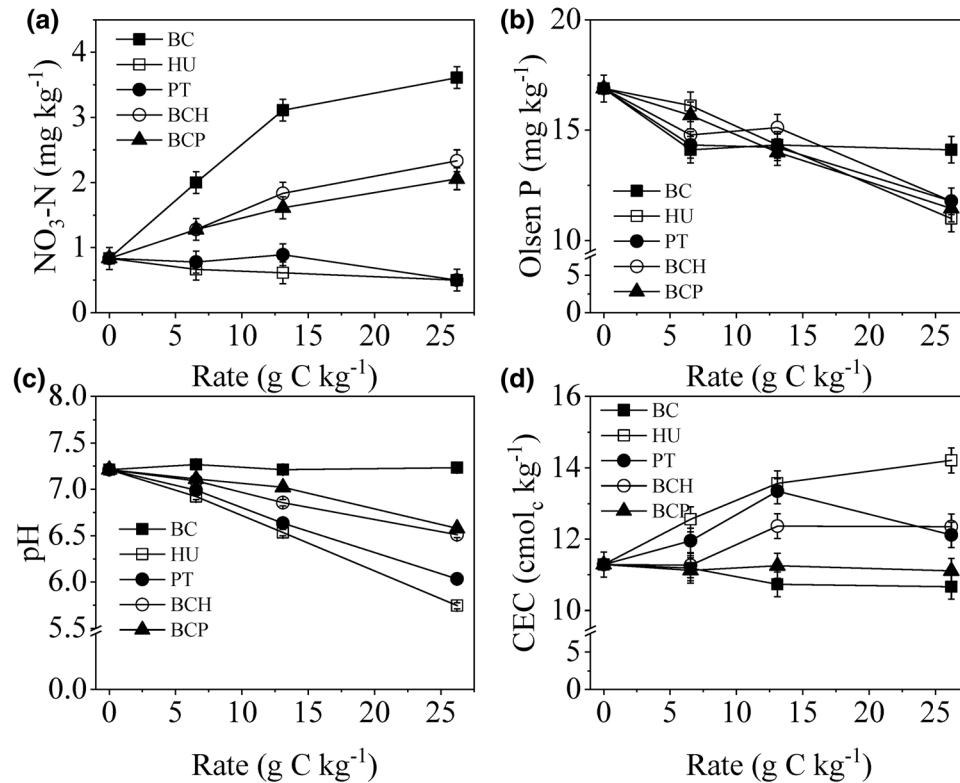


FIGURE 3 (a) Residual soil $\text{NO}_3\text{-N}$, (b) Olsen P, (c) pH, and (d) cation exchange capacity (CEC) as affected by amendment type and rate. The amendments are biochar (BC), humalite (HU), peat (PT), a 50:50 biochar/humalite mixture (BCH), and a 50:50 biochar/peat mixture (BCP). Vertical bars represent standard errors of the mean

3.6 | Soil pH

Amendment \times rate interaction ($P < .001$) and cycle main effects ($P < .001$) were significant for post-harvest soil pH (Table 4, Figure 3c). With the exception of biochar, which did not significantly change soil pH across the range of rates tested, soil pH decreased with rate for all the other amendments, with the decrease greatest for humalite and peat. The cycle main effect was also significant for soil pH ($P < .001$), with the pH significantly higher in Cycle 3 than in the first two cycles.

3.7 | Cation exchange capacity

There was a significant amendment \times rate interaction for CEC averaged across cycles (Table 4). Cation exchange capacity at harvest increased with humalite application across the range of rates tested, and increased with peat application through the 13.1 g C kg^{-1} rate, after which it decreased slightly (Figure 3d). Cation exchange capacity was 6% higher in Cycle 2 than in Cycle 3, but did not differ significantly from that in Cycle 1 ($P = .04$ for cycle main effect).

4 | DISCUSSION

4.1 | Dry matter yield

The overall higher DMY from amendment application relative to the control reflects the more favorable effects of these amendments on soil productivity. This was attributed in part to the high soil water holding capacity and nutrient availability associated with the increase in soil organic matter content. For example, Liu, Rong, Zhou, and Liang (2017) reported that improved rice (*Oryza sativa* L.) yields in their study were associated with higher nutrient availability and microbial biomass C, and enhanced enzyme and microbial community in the amended soil. Li, Parent, Karam, and Tremblay (2004) observed increases of up to 30% in potato (*Solanum tuberosum* L.) yield with the application of 48 Mg ha^{-1} peat in conjunction with mineral fertilizers and attributed the improved yield to improvements in soil moisture storage and nutrient availability.

Biochar and peat produced similar cumulative DMY in all cropping cycles and out-yielded the control and BCP in Cycle 1 and the control, humalite, and BCH in Cycle 2. While the superior performance of peat reflected the greater N uptake associated with this amendment

in Cycles 1 and 2, the similarly high DMY from biochar application were at variance with N uptake from biochar-amended soils, which was lower than all the other amendments in Cycles 1 and 2. This appears to suggest greater N use efficiency from biochar application, which is consistent with the higher residual $\text{NO}_3\text{-N}$ concentrations at all rates of biochar compared with the other four amendments. Averaged across Cycles 1 and 2, DMY was generally lower with humalite alone or mixed with biochar (BCH treatment). This was likely related to the toxicity symptoms (chlorosis, brown spotting) observed in soils receiving these amendment treatments. Bekele, Roy, and Young (2013), who used the same source of humalite as in the present study, reported similar symptoms from humalite application and noted that B (particularly the N/B ratio) was responsible for the toxicity symptoms because of the relatively narrow range between B deficiency ($0.5\text{--}1.0 \text{ mg kg}^{-1}$) and B toxicity (5.0 mg kg^{-1}).

4.2 | Nitrogen and phosphorus accumulation in aboveground plant tissue

Humalite and peat produced the highest plant N uptake during the first two cropping cycles and the least soil $\text{NO}_3\text{-N}$ concentration at all rates, suggesting that the N content of these amendments was mineralized and effectively taken up by plants, leaving lower levels in the soil. This is consistent with the less stable forms of C and lower C/N ratios of these amendments relative to those of biochar, which is known to contain highly stable C (Kuzayakov, Bogomolova, & Glaser, 2014). Biochar C typically has a very high alkyl C/O-alkyl C ratio, which is inversely related to N mineralization rate and, therefore, plant N uptake (Kuzayakov et al., 2014; Purwanto, Watanabe, Shoon, Kakuda, & Ando, 2005). Importantly, biochar contained lower N (0.4 g kg^{-1} vs. 10.7 g kg^{-1} for humalite and 7.2 g kg^{-1} for peat) while it was also applied at lower equivalent rates since it had a much higher C concentration (648 g C kg^{-1} vs. 375 g C kg^{-1} for humalite and 273 g C kg^{-1} for peat).

Significant amendment effects on P uptake observed in Cycle 2, where humalite, peat, and BCP produced higher P uptake than the control, and Cycle 3, in which peat resulted in greater P uptake than all the other amendments, reflect the higher content and greater lability of P in peat and humalite compared with biochar. However, the greater P uptake did not translate to superior yields from these amendments.

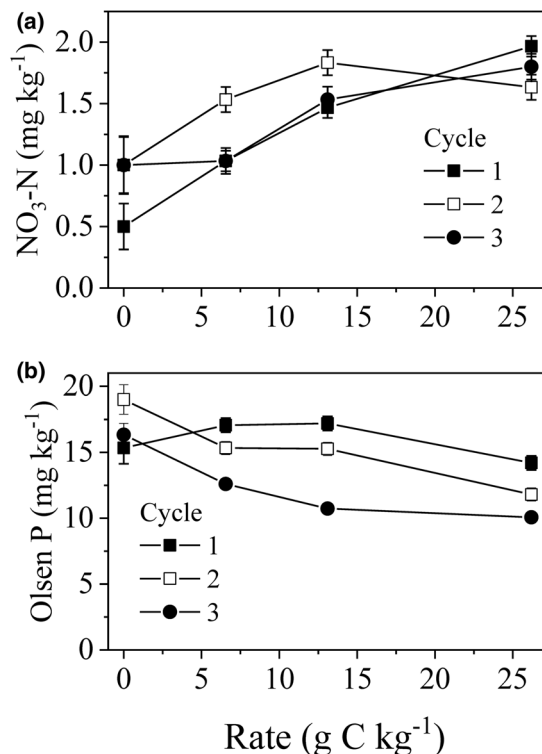


FIGURE 4 (a) Residual $\text{NO}_3\text{-N}$ and (b) Olsen P concentration as affected by amendment type and rate. The amendments are biochar (BC), humalite (HU), peat (PT), a 50:50 biochar/humalite mixture (BCH), and a 50:50 biochar/peat mixture (BCP). Vertical bars represent standard errors of the mean

4.3 | Residual nitrate- and ammonium-nitrogen and residual Olsen phosphorus in soil

The high $\text{NO}_3\text{-N}$ concentrations in soil amended with biochar was likely partly due to biochar increasing habitat for nitrifying bacteria (Jones et al., 2012). This finding contradicts a 3-yr field study by Jones et al. (2012), which showed no significant impact of biochar application on soil $\text{NO}_3\text{-N}$ concentration. This could be due to the highly productive agricultural soil in their study, which could have masked the benefits of biochar, in contrast to the relatively infertile soil used in our study. Further, the biochar in their study was coarse (83% > 2 mm in size) and was produced from European ash (*Fraxinus excelsior* L.), European beech (*Fagus sylvatica* L.), and English oak (*Quercus robur* L.) feedstock at a pyrolysis temperature of $450 \text{ }^\circ\text{C}$. The biochar in the present study, on the other hand, had larger pieces (>2 mm) removed by hand prior to application, which likely resulted in larger surface area (thus niche space) for soil microbes, and better soil-biochar contact. Additionally, biochar is known to

decrease denitrification at soil moisture contents <75% WFPS, which would minimize $\text{NO}_3\text{-N}$ loss from the soil (Nelson, Agudelo, Yuan, & Gan, 2011). Results from a related study showed that biochar significantly reduced cumulative N_2O emissions relative to all the other amendments, except for peat, over a 45-d period, which could further explain the higher $\text{NO}_3\text{-N}$ concentrations associated with biochar in the present study (Laskosky, 2015). The combined effect of increasing moisture retention and the ability to stabilize $\text{NO}_3\text{-N}$ in the soil makes biochar an appealing amendment in reclamation, as stockpiled soils tend to emit large flushes of $\text{NO}_3\text{-N}$ upon placement and aeration (Williamson & Johnson, 1990).

Nelson et al. (2011) observed a 4.2–8.8% reduction in $\text{NO}_3\text{-N}$ concentration in biochar-amended soils relative to non-amended soils, which occurred after an initial spike followed by a decrease in the soil $\text{NO}_3\text{-N}$ concentration. They attributed the decrease in $\text{NO}_3\text{-N}$ to volatile organic compounds in the biochar acting as a C source, which caused N immobilization.

Another observation made in the present study was that in almost all situations, residual $\text{NO}_3\text{-N}$ concentrations in biochar-amended soils were significantly higher than those in humalite-amended soils, despite the higher initial N concentration of humalite. However, the lower residual $\text{NO}_3\text{-N}$ concentrations in humalite-amended soil correlate well with the relatively high plant N uptake observed for humalite during the first two cropping cycles. This suggests that there was high N mineralization and nitrification in soil amended with humalite, and that the nitrate was being taken up by the barley plants. However, any DMY benefits that could have emanated from the high N uptake were offset by the suspected B toxicity observed in the humalite-amended soils, which reduced DMY.

Residual $\text{NH}_4\text{-N}$ concentrations in this study were much higher than $\text{NO}_3\text{-N}$ concentrations. While this is common in soils from the core of large stockpiles, such as that used in the present study, which are typically anaerobic, it is expected that rapid oxidation of $\text{NH}_4\text{-N}$ to $\text{NO}_3\text{-N}$ would occur following restoration of disturbed land using the soil. However, it is possible that anaerobic conditions in the potted soil resulting from inadvertent overwatering (unnoticed accumulation of water in the bottom layer of the potted soil) may have suppressed nitrification of $\text{NH}_4\text{-N}$ in the soil and from applied fertilizer. A similar result was reported by Johnson and Williamson (1994), who observed a rapid initial decline in $\text{NH}_4\text{-N}$ concentration, followed by an increase in the concentration after 29 wk. They attributed the increase to the large moisture content prevailing at the time of sampling, which would have inhibited nitrification. The general decrease of $\text{NH}_4\text{-N}$ concentrations with cycle regardless of amendment or rate of application was attributed to increased

nitrification. This is likely explained by the increased $\text{NO}_3\text{-N}$ concentrations observed, which increased with cycle.

The observed decrease in residual Olsen P concentration with increasing amendment rate reflects the observed general increase in plant P accumulation with amendment rate. The incremental additions of Ca and Mg (from increasing rates of amendment application) to a soil high in Ca and Mg concentrations may have resulted in a progressive decrease in Olsen P concentration, as Ca and Mg are known to form low solubility P precipitates (Wadu, Michaelis, Koeker, & Akinremi, 2013). However, humalite, which had the highest concentrations of Ca and Mg, produced the highest plant P accumulation and the second lowest residual Olsen P concentration relative to the control. Nevertheless, organic matter constituents, particularly humic and fulvic acids, which are abundant in humalite, are known to increase P availability to plants via competition for Fe/Al oxides, and replace H_2PO_4^- on anion exchange sites (Guppy, Menzies, Blamey, & Moody, 2005a, Guppy, Menzies, Moody, & Blamey, 2005b).

4.4 | Soil pH and cation exchange capacity

The difference in pH observed in amended soils reflected the pH values of the respective amendments. Biochar, which had a pH closest to that of the unamended soil, had the least effect on soil pH. Humalite and peat, on the other hand, had much lower pH values thereby reducing the pH of the amended soils. These findings corroborate those from previous studies (Nandakumar, Sarvanan, Sigaram, & Chandrasekaran, 2004; Sanli et al., 2012). As expected, the blended amendments, BCH and BCP, produced intermediate pH values.

The liming effect of biochar is well documented in the literature and is thought to be one of the main reasons why biochar improves soil fertility (DeLuca, MacKenzie, & Gundale, 2009; Jones et al., 2012; Laird et al., 2010). However, because of the pyrolysis temperature employed in the production of the biochar used in the present study, the observed pH was much more neutral than that in other studies (DeLuca et al., 2009, Laird et al., 2010; Jones et al., 2012) which ranges from 7.6 to 8.8. Nonetheless, benefits to soil quality, including improved soil $\text{NO}_3\text{-N}$ status of the amended soil, were still observed in the present study.

The higher overall CEC of soil amended with humalite relative to the other amendments across amendment rates are consistent with findings by Giannouli et al. (2009). Their study showed that soils amended with lignite (which is similar to humalite) tended to have higher CEC values than peat-amended soils, mainly because of their higher humic acid concentration (Giannouli et al., 2009).

5 | CONCLUSION

The unblended forms of biochar and peat provide the greatest potential for improving the quality of stockpiled salvaged soil intended for land reclamation, based on the biomass yields from soils treated with these amendments. The properties of peat make it a suitable amendment when recreating acidic, sandy Jack pine (*Pinus banksiana* Lamb.) ecosystems common throughout the AOSR, while biochar may be beneficial in creating neutral to basic, finer-textured, mixed-wood ecosystems. Should only one amendment be used for an entire site, biochar may prove to be the most beneficial as it often performed similarly to peat and did not show a decrease in Olsen P concentration at higher rates of application. Biochar's ability to maintain high soil nitrate concentrations, likely through providing habitat for soil microorganisms and nitrifying bacteria, makes it highly promising as this may help kick-start initial revegetation of reclaimed sites while providing a steady, sustained release of nitrate as nitrifying microbial communities colonize and flourish. With the expected increase in humic acids following plant establishment, as well as any acidifying or leaching effects from rainfall, biochar may be the better option, as it did not impact soil pH. Land reclamation in the AOSR still poses great concerns to the public, the environment, and reclamation planners; the use of organic amendments, particularly on upland ecosites, may substantially alleviate some of these concerns, and aid in the restoration of these disturbed ecosystems.

ACKNOWLEDGMENTS

We thank Rob Ellis and Anthony Buckley for their technical assistance, Nicholson Jeke for miscellaneous help, and Imperial Oil Resources Ltd. for funding.

CONFLICT OF INTEREST

The authors declare no conflict of interest.

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How to cite this article: Laskosky JD, Mante AA, Zvomuya F, Amarakoon I, Leskiw L. A bioassay of long-term stockpiled salvaged soil amended with biochar, peat, and humalite. *Agrosyst Geosci Environ*. 2020;3:e20068. <https://doi.org/10.1002/agg2.20068>