



Ameliorating Effects of Leucaena Biochar on Soil Acidity and Exchangeable Ions

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ABSTRACT

The amendment effect of Leucaena biochar was investigated in incubation study in acidic soil (pH (water (H₂O)) = 4.5) of northwest India. The biochar (BC) was mixed at 2%, 4%, and 6% with soil and change in soil pH, electrical conductivity (EC), ammonium (NH₄⁺) and nitrate nitrogen (NO₃⁻-N), exchangeable bases (calcium + magnesium, potassium, sodium) (Ca + Mg, K, Na), aluminum (Al), and Bray's phosphorus (P) were measured periodically. The mean increase in soil pH was 0.65, 1.35, and 2.0 unit at 2%, 4%, and 6% (w/w) of biochar incorporation, respectively. Application of biochar significantly ($P < 0.01$) reduced ammonium (NH₄⁺-N) content of soil, whereas NO₃-N concentration increased by threefold and fivefold by application of 2% and 4% (w/w) of biochar, respectively. Exchangeable soil potassium (K) and Ca + Mg concentrations increased with the application of biochar. Application of biochar above 2% (w/w) reduced exchangeable Al concentration to nondetectable limit. The study suggests Leucaena biochar may serve as an amendment for N transformation in highly acidic soil.

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Introduction

Soil acidity affects nearly 50% of the world's potentially arable land, particularly in humid tropics (Von Uexkull and Mutert 1995). In India, approximately one-third (49 M ha) of the cultivated land is affected by soil acidity (Mandal 1997). In acidic soils, crop productivity is mostly constrained by aluminum (Al) and iron (Fe) toxicity, phosphorus (P) deficiency, and other acidity-related soil fertility and plant nutritional problems (Manoj et al. 2012; Patiram 1991). In most situations, poor crop growth in acidic soils can be correlated directly with aluminum (Al) saturation (Abruna-Rodriguez et al. 1982; Sartain and Kamprath 1977). Although liming of acidic soils can ameliorate soil acidity, this is neither an economic option for poor farmers nor an effective strategy for alleviating subsoil acidity. Recent studies have shown that direct application and incorporation of green manures, animal wastes, and crop residues can ameliorate soil acidity (Berek, Radjagukguk, and Maas 1995; Hue 1992; Xu, Tang, and Chen 2006). However, the reclamation effects of direct incorporation of organic residues in soil do not last long due to rapid mineralization of added residues.

The conversion of organic waste to produce biochar using the pyrolysis process is one viable option that can enhance natural rates of carbon sequestration in the soil, reduce farm waste, and improve the soil quality (McHenry 2009). Biochar application resulted in higher grain yields at sites with low P availability and improved the response to N and NP chemical fertilizer treatments (Asai et al. 2009); moreover, the amendments can significantly improve soil quality, including increase in pH, organic carbon, and exchangeable cations as well as reduction in

tensile strength when applied at higher rates (Chan et al. 2007; Jha et al. 2010). Increase in soil pH and concomitant reduction in exchangeable Al with application of biochar in acidic soils has been reported (Steiner et al. 2007; Van Zwieten et al. 2010).

The effect of biochar on pH, exchangeable acidity, basic cations, and Al content of acidic soils has been reported mostly from temperate agro-ecosystems (Chan et al. 2007; Nelissen et al. 2014; Rajkovich et al. 2012). Information on biochar or biochar as liming material is completely lacking from Indian subcontinent. Subabul (*Leucaena leucocephala*) also known as white lead trees, a member of fabaceae family, is a popular farm forestry tree species widely distributed in tropical and subtropical countries. It is one of the fast growing hardy evergreen species grown in several parts of India as a source of forage and pulp production. The fast growth nature of *Leucaena* and its adaptability under diverse edaphic conditions offer good opportunity for making biochar as an amendment to acidic soils. In the present study, we quantified the liming effect of *Leucaena* biochar samples by an incubation experiment in an acidic Alfisol. The study was conducted with the hypothesis that leguminous biochar with high alkalinity could be the substitute for lime in correction of soil acidity. The present work was carried out with the objective to determine the effect of different rates of *Leucaena* biochar application on soil pH, electrical conductivity (EC), exchangeable NH_4^+ and NO_3^- -N, exchangeable bases ($\text{Ca}^{2+} + \text{Mg}^{2+}$, K^+ , Na^+), Al concentrations, and available P in acidic Alfisol of Palampur, India.

Materials and methods

Soil sampling and analysis

An acidic Alfisol (U.S. Soil Taxonomy) used in this study was from the long-term fertilizer experiment plot of Palampur, Himachal Pradesh, India (32°N, 76°E). The sample was taken from the topsoil (0–15 cm), air-dried, and ground to pass through a 2 mm sieve. Texture of soil was determined by hydrometer method. Soil pH and EC were determined in 1:2.5 soil to water suspension. Soil total organic carbon was determined by dry combustion technique using a TOC analyzer (Shimadzu 5000 VA, Kyoto, Japan). Soil-exchangeable hydrogen and aluminum (H^+ and Al^{3+}) were determined by extracting 5 g of soil with 25 ml of 1 M potassium chloride (KCl), shaking gently, and allowing the sample to rest for 30 min. Samples were then filtered and extraction flasks washed four times with 25 ml of 1 N KCl. Phenolphthalein was added to the extracts, and then the extracts were titrated with 0.01 N sodium hydroxide (NaOH) (Thomas 1982). The concentrations of ammonium (NH_4^+ -N) and nitrate (NO_3^- -N) in soil were determined by shaking 5 g soil with 50 ml of 2 M KCl for 1 h, followed by centrifugation and filtration through a filter paper. NH_4^+ and NO_3^- -N in filtered extracts were determined colorimetrically using flow injection analyzer (FOSS, Hillerod, Denmark). Exchangeable base cations were extracted with neutral 1 M ammonium acetate. Ca^{2+} and Mg^{2+} were then determined by versenate titration method and K^+ and Na^+ by flame photometry. Exchangeable Al was extracted by shaking 5 g soil with 50 ml of 1 M KCl for 30 min, followed by centrifugation and filtration, and Al concentration in filtered extracts was then determined by inductively coupled plasma atomic emission spectrometer (ICP-AES, Perkin Elmer, Waltham, Massachusetts, USA). Soil-available P was determined by Bray's method (Bray and Kurtz 1945). Basic physical and chemical properties of soil are given in Table 1.

Biochar preparation and analysis

Subabul (*Leucaena leucocephala*) stem and twigs were collected locally and dried at 80°C for 12 h. Biochar was produced by indigenous technique (drum method). The stems were chopped to 8–10 cm in length and were then pyrolyzed under oxygen-limited conditions for 4 h then allowed to cool overnight. Subsequently, the biochar was crushed manually and ground to pass through a 2 mm sieve. The chemical properties of original biochar are presented in Table 2.

Table 1. General characteristics of the soil used in incubation study.

Parameters	Value
Sand (%)	30
Silt (%)	46
Clay (%)	24
Soil pH (H ₂ O)	4.5
EC (dS m ⁻¹)	0.16
Total C (g kg ⁻¹)	13.5
Total N (g kg ⁻¹)	1.4
Exchangeable acidity (cmol (p+) kg ⁻¹)	3.5
Exchangeable Ca ²⁺ (cmol (p+) kg ⁻¹)	1.8
Exchangeable Mg ²⁺ (cmol (p+) kg ⁻¹)	0.9
Exchangeable K ⁺ (cmol (p+) kg ⁻¹)	0.25
Exchangeable Na ⁺ (cmol (p+) kg ⁻¹)	0.12
Exchangeable Al ³⁺ (cmol (p+) kg ⁻¹)	3.4
Effective cation exchange capacity (cmol (p+) kg ⁻¹)	6.5

Table 2. General characteristics of biochar used in incubation study.

Parameters	Value
pH (H ₂ O)	9.3
EC (dS m ⁻¹)	0.14
Total C (g kg ⁻¹)	79.9
Ash (%)	7.4
Biochar alkalinity (cmol (p+) kg ⁻¹)	39
Ash alkalinity (cmol (p+) kg ⁻¹)	580
Exchangeable Ca ²⁺ (cmol (p+) kg ⁻¹)	5.2
Exchangeable Mg ²⁺ (cmol (p+) kg ⁻¹)	3.3
Exchangeable K ⁺ (cmol (p+) kg ⁻¹)	17
Exchangeable Na ⁺ (cmol (p+) kg ⁻¹)	0.85

Biochar pH and EC were determined in 1:5 biochar to deionized water extraction (Gaskin et al. 2008). Total C in original biomass and biochar sample was determined by dry combustion method using Shimadzu TOC analyzer (solid module). The ash alkalinity of biochar was determined by the method outlined by Pansu and Gautheyrou (2006). Briefly, the ash of biochar was obtained by heating the sample at 500°C for 4 h in muffle furnace and then 0.5 g of biochar ash was dissolved in 25 ml of 1 M hydrochloric acid (HCl). Subsequently, 5 ml of aliquot was titrated against 0.05 M NaOH. Basic cation concentration (Ca²⁺, Mg²⁺, K⁺, and Na⁺) in aliquot was determined using the method as mentioned in case of soil.

Biochar sample alkalinity was determined using the back titration method. In brief, 0.2 g of biochar sample was weighed into a beaker in triplicate followed by addition of 40 ml of 0.03 M HCl solution. The samples were shaken for 2 h on horizontal shaker at 25°C. After shaking, samples were left undisturbed for 24 h. Residual HCl was titrated to pH 7.0 with 0.05 M NaOH. Biochar alkalinity was computed by determining the amount of HCl consumed.

Soil incubation

Air-dried soil samples of 250 g were placed in glass beakers and were amended with biochar at 0, 20, 40, and 60 g kg⁻¹ corresponding to field application rates of approximately 0, 13, 26, and 39 t ha⁻¹ (considering soil depth of 0–5 cm and bulk density of 1.3 Mg m⁻³). All glass beakers were adjusted to 60% of water-holding capacity with deionized water and covered with aluminum foil with a small hole to allow air diffusion. The beakers were weighed every alternate day to maintain constant moisture content throughout the incubation period. The soils amended with and without biochar were incubated at 25°C and subsampled at 1, 6, 20, 41, 68, and 90 days. There were three replicates for each treatment. At each sampling date, soil samples were taken from each beaker using a spatula

and the disturbed soils were leveled out. Soil pH and exchangeable bases (K^+ , Ca^{2+} , Mg^{2+} , Na^+), exchangeable Al, and available P were determined on each sampling date. Soil moisture content was determined gravimetrically at each sampling stage for expression of results on dry weight basis. Biochar was analyzed similar to the soil.

After incubation, NH_4^+ and NO_3^- -N content and EC were determined in all the samples. Effective cation exchange capacity (ECEC) was calculated from the sum of the exchangeable base cations and Al.

Statistical analysis

SPSS Windows version was used for statistical analysis. One-way analysis of variance (ANOVA) was performed for each time interval of the incubation for comparisons of means. Significant effects of various treatments were measured using the *t*-test.

Results and discussion

Soil pH and electrical conductivity

Soil pH change in biochar-amended soil during incubation is presented in Figure 1. The incorporation of *Leucaena* biochar increased soil pH compared with controls ($P < 0.01$). The initial pH of the unamended soil (control) was 4.56. Addition of biochar significantly increased soil pH at all the rates of biochar application. The mean increase in soil pH compared with control was 0.65, 1.35, and 2.0 units at 2%, 4%, and 6% (w/w) of biochar incorporation, respectively. As expected, the magnitude of soil pH increase was significant ($P < 0.01$) as the biochar addition rates increased from 20 to 40 and 60 g kg^{-1} . The maximum rise in soil pH was observed after 1 week of incubation, thereafter a slight decrease in soil pH was recorded till the termination of incubation study (Figure 1). The decline of soil pH was ascribed to the nitrification of NH_4^+ in soil. Soil pH fluctuations showed a similar trend irrespective of the biochar addition rates. Beyond 300°C, the C started to become ashed and alkali salts began to separate from the organic matrix, increasing the pH of biochar. Higher pH of biochar indicates their potential as amendments to neutralize soil acidity (Chintala et al. 2014). Generally, legume materials have higher ash alkalinity due to the unbalanced uptake of cations and anions, and thus have greater amelioration effects on soil acidity than non-legume materials (Wang, Li, and Xu 2009). The input of ash alkalinity and the mineralization of organic N are two main factors contributing to increased soil pH early in the incubation study, while nitrification of NH_4^+ -N would contribute to decreased soil pH later in the incubation; the balance of these reactions determined the final soil pH (Yuan and Xu 2011).

Change in soil EC with the application of biochar was measured after the completion of incubation experiment (Figure 2). Application of biochar significantly increased the EC of soil at all rates of biochar application ($P < 0.05$). The EC of initial soil was 0.16 $dS m^{-1}$. Application of 2%, 4%, and 6% (w/w) of biochar increased soil EC from 0.16 to 0.43, 0.72 and 0.75 $dS m^{-1}$, respectively. There was no significant difference in EC between 4% and 6% (w/w) of biochar incorporation ($P > 0.05$). Similar result was reported by Novak et al. (2009). They also did not observe any significant difference in leachate EC with increasing biochar addition. Soils amended with 6% (w/w) of *Leucaena* biochar had the highest EC (0.75 $dS m^{-1}$), followed by 4% and 2% of biochar application rates. The unamended soil had the lowest EC of 0.13 $dS m^{-1}$, reflecting the extractable cation concentrations measured in the biochar.

Ammoniacal and nitrate-N content

The changes in nitrogen dynamics with application of biochar are not fully understood (Clough et al. 2010; Lehmann 2007; Singh et al. 2010). Effects of different levels of biochar application on changes

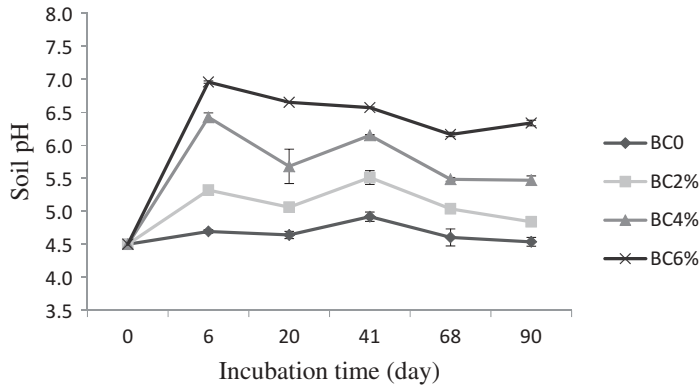


Figure 1. Soil pH during the incubation study amended with *Leucaena* biochar.

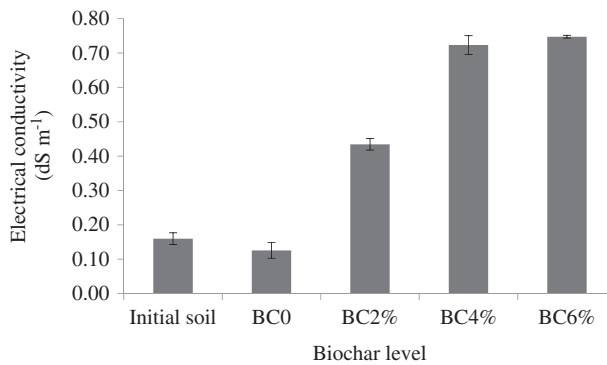


Figure 2. Soil EC during the incubation study amended with *Leucaena* biochar.

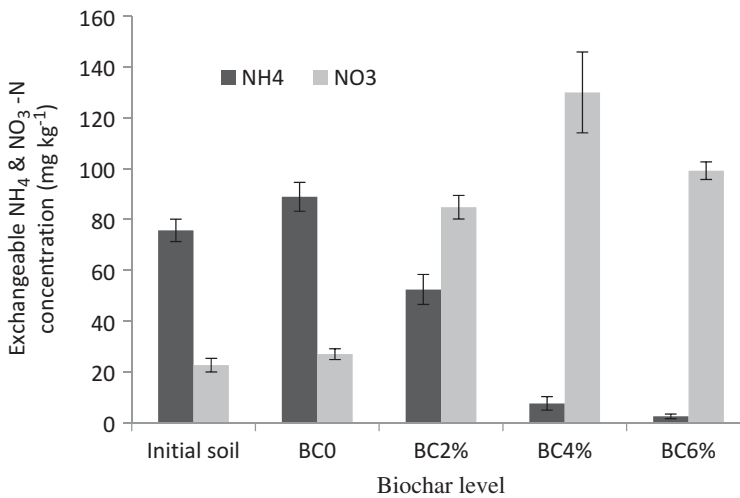


Figure 3. Effect of biochar addition on soil mineral N content.

of $\text{NH}_4^+ - \text{N}$ and $\text{NO}_3^- - \text{N}$ concentration at the end of incubation are shown in [Figure 3](#). Application of biochar significantly reduced the concentration of $\text{NH}_4^+ - \text{N}$ in soil ($P < 0.01$). At the same time, it

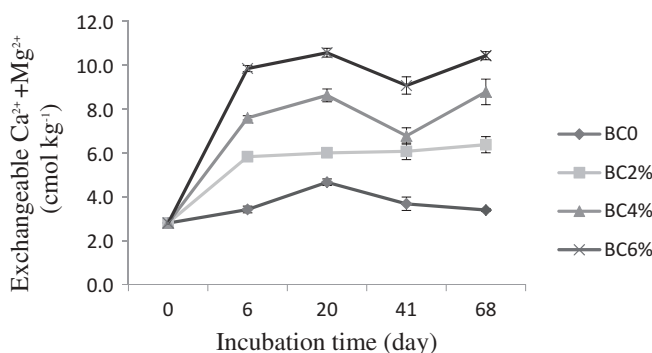


Figure 4. Exchangeable Ca + Mg concentration as affected by biochar application.

significantly increased the $\text{NO}_3^- - \text{N}$ concentration in soil ($P < 0.01$). The results clearly demonstrate that application of biochar in acidic soil enhances the process of nitrification. Biochar is more important as a soil conditioner and driver of nutrient transformations and less so as a primary source of nutrients (Glaser, Lehmann, and Zech 2002; Lehmann et al. 2003). In present study, acidic (Alfisol) soil of Palampur contains majority of mineral N in ammoniacal form. Approximately, 77% of mineral N present in soil was in $\text{NH}_4^+ - \text{N}$ form. Ammonification is a biotic process driven primarily by heterotrophic bacteria and a variety of fungi (Stevenson and Cole 1999) and $\text{NH}_4^+ - \text{N}$ is the predominant form of N in extremely acidic soil. With the application of 2% (w/w) of biochar, the $\text{NH}_4^+ - \text{N}$ content decreased to the extent of 38%. Biochar application at 4% and 6% (w/w) reduced the $\text{NH}_4^+ - \text{N}$ concentration from 89 (control) to 7.6 and 2.6 mg kg^{-1} , respectively.

Application of biochar significantly favors the process of nitrification in acidic soil. In control soil (no biochar), $\text{NO}_3^- - \text{N}$ content at the end of incubation study was 27 mg kg^{-1} , which was increased to 84 and 130 mg kg^{-1} on the application of 2% and 4% of biochar, respectively. Biochar application beyond 4% (w/w) could not increase the $\text{NO}_3^- - \text{N}$ concentration in soil. In fact, there was significant decline in $\text{NO}_3^- - \text{N}$ content in soil with the application of 6% of biochar. pH is the major driver of the nitrification response to biochar additions to soil. Nitrification is essentially a biotic process that is most commonly mediated by autotrophic organisms, including bacteria and archaea, in agricultural and forest soils (Grenon, Bradley, and Titus 2004; Stevenson and Cole 1999). The transformation of N from $\text{NH}_4^+ - \text{N}$ to $\text{NO}_3^- - \text{N}$ during the incubation was due to the input of ash alkalinity and subsequent increase in soil pH. Biochar has been found to increase net nitrification rates in temperate and boreal forest soils that otherwise demonstrate no net nitrification (Berglund, DeLuca, and Zackrisson 2004). It is well understood that autotrophic nitrifying bacteria are favored by less acidic soil conditions (Stevenson and Cole 1999).

The rapid response of the nitrifier community to biochar additions in soils with low nitrification activity and the lack of a stimulatory effect on actively nitrifying communities suggest that biochar may be adsorbing inhibitory compounds in the soil environment (Zackrisson, Nilsson, and Wardle 1996), which then allows nitrification to proceed.

Exchangeable base cations

The incorporation of biochar at all levels increased exchangeable base cations of the soil (Figures 4–6) ($P < 0.05$) because the legume biochar had higher alkalinity and thus neutralized more exchangeable acidity of the soil. For all biochar application rates, the concentration of Ca + Mg was significantly higher than the control. Biochar amendments elevated exchangeable Ca + Mg level in the Alfisol from 3.58 to 8.54 $\text{cmol (p}^+) \text{ kg}^{-1}$. Residue contains base cations (Wang, Li, and Xu

2009), and after pyrolysis, these base cations in original biomass were retained in the biochar samples (Yuan and Xu 2011). During incubation period, base cations released from the biochar samples exchanged with Al^{3+} and H^+ and enriched soil with these exchangeable base cations. When the biochar was incorporated into the soil, these base cations released into the soil and occupied soil exchange sites. Therefore, the incorporation of biochar not only decreased soil acidity but also improved soil fertility. The data suggest that the basic cations (K, Ca + Mg, Na) in the biochar samples released quickly leading to the quick rise in soil pH during the incubation period. As the dose of biochar incorporation increased from 2% to 4% and 6%, there was a significant increase in Ca + Mg concentration at each sampling stage. The neutralization of soil-exchangeable acidity by biochar application is also evident from the decrease in exchangeable Al content of soil. The decrease in soil-exchangeable Al further reduced the potential toxicity of Al in acidic soils. Addition of biochar at 2%, 4%, and 6% (w/w) increased mean soil-exchangeable Ca + Mg concentration by 50%, 92%, and 138%, respectively, at all sampling stages of incubation study.

Similar trend was observed for exchangeable K and Na (Figures 5 and 6). The addition of biochar led to greater increase in soil-exchangeable K. The acidic soil used in the incubation study was poor in terms of exchangeable potassium (110 mg kg^{-1}). Application of biochar significantly ($P < 0.01$) increased soil-exchangeable K at all rates of application. Soil-exchangeable K of unamended soil was $0.28 \text{ cmol (p}^+) \text{ kg}^{-1}$. Exchangeable K of soil increased to 5.8, 9.2, and 13.8 times with addition of 2%, 4%, and 6% of biochar, respectively. Similar results for K were reported by Lehmann et al. (2003) when biochar was added to Oxisol of Brazilian Amazon. Generally, acidic soils of India are poor in

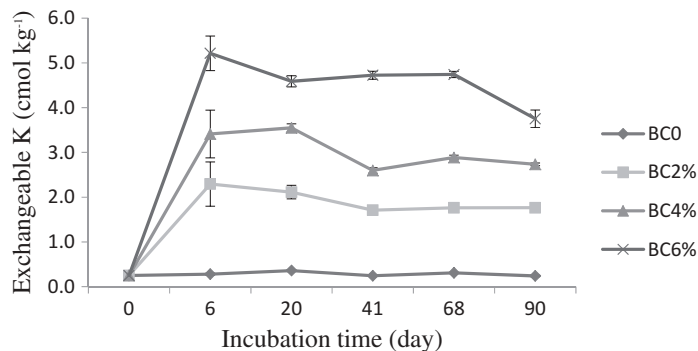


Figure 5. Exchangeable K concentration as affected by biochar application.

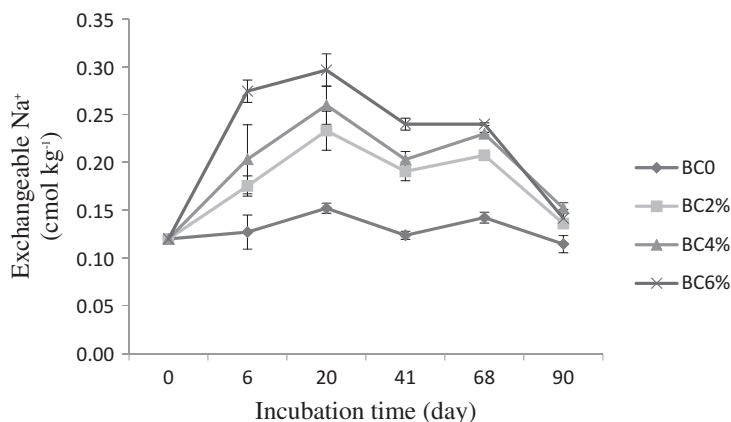


Figure 6. Exchangeable Na concentration as affected by biochar application.

terms of exchangeable K concentration, as the soil clay is mostly dominated by 1:1 type (kaolinitic) clay minerals. Under these circumstances, legume biochar could be the potential amendment not only for soil pH correction but also for substitution of potassic fertilizer. Exchangeable K was lower than the exchangeable Ca + Mg of the soil; however, the biochar sample contains higher K content than the Ca + Mg. Therefore, biochar incorporation could provide alternate source of potassium in acidic soils (Yuan and Xu 2011). The exchangeable Na content of soil increased in the beginning of the study (up to 20 days), thereafter there was a gradual decline in exchangeable Na content till termination of the study. The increase in exchangeable Na content was to the extent of 2, 2.5, and 3 times with the application of 2%, 4%, and 6% (w/w) of *Leucaena* biochar (Figure 6).

Exchangeable Al content

The impact of the addition of biochar at different rates in soil-exchangeable Al concentration during the incubation period is shown in Table 3. When the soil pH drops below 5, Al^{3+} is solubilized into the soil solution and this is the most important rhizotoxic Al species. The cause of soil acidity is mainly due to exchangeable Al, while exchangeable H contributes less to exchange acidity. In present study, exchangeable Al occupies 94.7% of exchange acidity for the control soil, and remaining 5.3% of exchange acidity is contributed by exchangeable H. It signifies that exchangeable Al is the major contributor of exchangeable acidity of Alfisol (Yuan and Xu 2011). Application of biochar significantly ($P < 0.01$) reduced the concentration of exchangeable Al during the incubation period. It is consistent with the findings of other workers (Steiner et al. 2007; Novak et al. 2009; Yuan and Xu 2011). Van Zwieten et al. (2010) also reported that an increase in soil pH from 4.2 to between 5.4 and 5.9 with the application of 1% (w/w) papermill biochar in a ferrosol resulted in a concomitant reduction in exchangeable Al from 2 to <0.1 cmol (p) kg^{-1} . Application of biochar at 2% (w/w) reduced Al concentration of incubated soil from 3.55 to 0.5 cmol (p^+) kg^{-1} and the effect lasted till the termination of the incubation study. Application of biochar above 2% (w/w) reduced exchangeable Al concentration to nondetectable limit in all the sampling days. It indicates that Al toxicity decreases due to its complexation with high-molecular-weight organic compounds (Alleoni et al. 2010) present in the biochar. The results clearly depict that the use of *Leucaena* biochar offers an alternative to liming material for ameliorating strongly acidic soils and may be effective in increasing soil pH.

Available phosphorus

Application of biochar significantly ($P < 0.05$) affected the P availability in high P soils of Palampur (Figure 7). Acidic soils are generally deficient in available P but the soil used in present investigation was high in available P due to long-term application of NPK fertilizer (since 1972), which led to the build-up of available P even under the acidic soil environment. The initial P status of incubated soil was 54 mg kg^{-1} . Application of biochar significantly reduced the availability of P at all levels of biochar application. It was observed that 2–6% (w/w) application of biochar to soil reduced P availability by 35–48% by the end of incubation study. The decrease in P availability with application of biochar and time could be due to increase in concentration of exchangeable bases (Ca + Mg), which subsequently reacted with P and formed unavailable tricalcium phosphate.

Table 3. Effect of different levels of biochar application on exchangeable Al^{3+} concentration.

Al (cmol kg^{-1})					
Treatments	0	6	20	41	68
BC 0	3.41	3.55 (0.11)a	3.59(0.23)a	4.47(0.07)a	3.71(0.10)a
BC 2%	3.41	0.30 (0.11)b	0.34 (0.12)b	0.34 (0.03)b	0.30 (0.05)b
BC 4%	3.41	ND	ND	ND	ND
BC 6%	341	ND	ND	ND	ND

ND, not detectable; means followed by same letter are not significant; values in parentheses indicate standard error of mean.

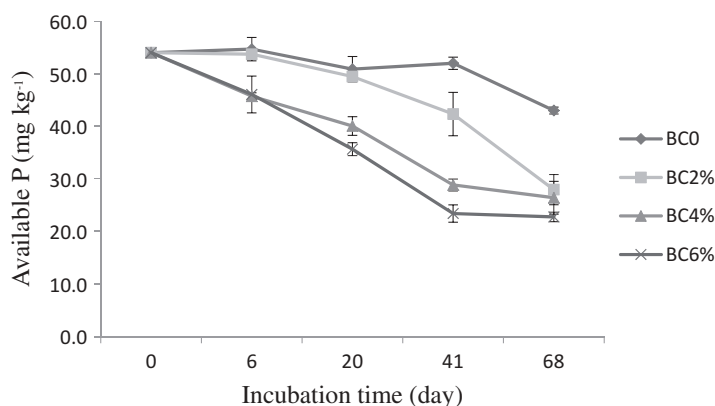


Figure 7. Available P concentration as affected by biochar application.

Conclusions

There is a need to develop alternate liming material for reclamation of acidic soil. The conversion of *Leucaena* woods and twigs to biochar offers industry an attractive option for developing amendment for reclamation of acidic soil. Biochar characterization and short-term soil incubations can provide some insight into the short-term effects of applying biochar that could be used as an amendment for reclamation of acidic soil. The biochar from *Leucaena* biomass was shown to provide benefits to Alfisol and improvement in both nutrient transformation and ameliorative performance. The present work provides useful information on *Leucaena* biochar and its impact on nutrient transformation and alleviation of soil acidity. Soil-exchangeable base cations and pH increased significantly by addition of biochar in highly acidic Alfisol of Palampur. The process of nitrification increased significantly by application of biochar. Soil-exchangeable Al content decreased to nondetectable limit by application of biochar. The effect of *Leucaena* biochar addition on acidic soil reclamation and nutrient transformations needs to be further assessed in field before the large-scale applications to agricultural fields.

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