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FULL-LENGTH RESEARCH ARTICLE

Carbon, Nitrogen and Phosphorus Mineralization as Influenced by Type of Organic Residues and Soil Contact Variation in Vertisol of Central India

Amrit Lal Meena[1] · Pramod Jha[2] · M. L. Dotaniya[2] · Bipin Kumar[3] · B. P. Meena[2] · R. L. Jat[4]

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Abstract Soil organic carbon (C) and nitrogen (N) play a critical role in plant nutrient dynamics and crop yield performance under different soils. In this study, the amount of carbon (C), N and phosphorus (P) mineralization as influenced by type of organic residue and soil contact variation was studied for an incubation period of 75 days under controlled laboratory conditions. Different types of residues, viz. subabul residue (*Leucaena leucocephala*) (T_1), biochar of subabul residue (T_2) and FYM (T_3), along with unamended soil with no residue constituted the three modes of application, viz. buried (t_1), incorporated (t_2) and surface applied (t_3) treatments. Among all the treatments, subabul residue applied treatments showed highest rate of C mineralization in which surface applied (SA) treatments varied from 19.54 at 24 h to 239.55 mg CO_2–C/100 g soil at 75 days followed by subabul incorporated (I) (17.04–237.8 mg CO_2–C/100 g soil) and subabul buried (B) (15.91–225.5 mg CO_2–C/100 g soil) treatments. Subabul incorporated (I) treatment showed lowest rate of mineralization of NO_3–N, which varies from 0.010 and 0.613 mg kg^{-1} at 7 days to 4.640 and 10.03 mg kg^{-1} at 75 days after incubation followed by subabul buried (B) and SA treatments. These results will be useful in the selection of agriculture crop inputs and their proper placement in soils with respect to nutrient mineralization.

Keywords Biochar · Nutrient dynamics · Subabul residue

Introduction

Soil organic matter (SOM), one of the crucial indicators for the soil fertility and crop productivity, has received global attention recently. An appropriate package and practices of SOM management not only improve crop productivity [18, 26], but also reduce the atmospheric concentration of carbon dioxide (CO_2) [20, 24, 51]. The use of more stable substances such as carbonized materials from incomplete combustion of organic materials such as black C, pyrogenic feedstocks and charcoal could provide a long-term stability for maintaining high levels of SOM and available nutrients in the soil [3, 45, 50]. Biochar as defined by Lehmann and Joseph [41] is a fine-grained, porous, stable C product remaining after plant biomass subjected to thermochemical conversion process at low temperature (350–600 °C) in a little or no oxygen environment known as pyrolysis [54, 60, 63, 64]. In recent years, it has gained attention as a potential amendment to boost soil fertility and crop productivity across the globe with multidimensional opportunity to reduce the C emission from soils by storing C in long-lived C pools [45, 48]. As a source of C-negative recalcitrant soil C pool, biochar serve as a sink of atmospheric CO_2 stored in highly recalcitrant soil C

✉ M. L. Dotaniya
mohan30682@gmail.com

[1] ICAR-Indian Institute of Farming Systems Research, Modipuram, Meerut 250 110, India

[2] ICAR-Indian Institute of Soil Science, Nabibagh, Bhopal 462 038, India

[3] ICAR-Indian Agricultural Research Institute, New Delhi 110 012, India

[4] ICAR-Indian Institute of Pulse Research, Kanpur 208 017, India

stocks [9, 59]. The biochar provides a pool of C that undergoes minimal microbial degradation in soil [28, 46]. Accumulation of this persistent pool of C within the soil can improve soil structure, water holding capacity, cation exchange capacity, surface sorption capacity, base saturation and nutrient cycling [19, 21, 22, 34]. In addition, the biochar can have a protective effect for other sources of C within the soil and has been reported to decrease the mineralization rate of both native SOC and fresh inputs of C, i.e., raw residues [7, 35, 43, 58]. Estimation of net C mineralized or converted into CO_2 from biochars decomposition was needed to improve understanding on the efficiency of biochars in enhancing soil quality, C sequestration and biochar stability in soils [55]. Biochar could offset 12% of the current anthropogenic CO_2–C equivalent emissions from soils [47]. Positive effects of biochar on the soil ecosystem and functions were proposed to derive either directly from the nutrients within biochar itself, or indirectly from its ability to sorb and retain nutrients [33, 42]. Besides sequestering C, biochar has been observed for potential improvement in N and P cycling in the soil– plant system [44, 58]. A major portion of N exists in a complex organic form that must be ammonified to NH_4^+–N and then nitrified to NO_3^-–N prior to plant uptake. Biochar improved N and phosphorus (P) mineralization kinetics in soils [13, 31]. It plays an important role in the biochemical cycling of N and P through its large surface area, pH and nutrient content [23], which varied among different types of biochar depending on their source of raw material [6, 31]. Recent studies have demonstrated that the addition of biochar to surface mineral soils may directly influence N transformations [67]. Gaskin et al. [29] reported that N from biochar could not be available to plants at the initial stage of application in the soil due to lower rate of decomposition. The addition of biochar to soils resulted slower mineralization rate compared uncharred material [40] and decreased net C and N mineralization in soil [11, 17]. Many researchers revealed that biochar application directly involved in the biochemical process related to mostly for N and P utilization [2], but reduced the activities of C cycle enzymes [42, 53, 57]. Asai et al. [1] showed that biochar application improves P availability and crop yield under low-P status soils. Although most soil properties could be improved through the application of crop residues or pyrolyzed material, there is limited research work on the effect of subabul biochar with respect to C, N and P dynamics under different soil contact variations. Keeping this in mind, the hypothesis of this study was to evaluate the effect of organic residues and soil contact variation on C, N and P mineralization in Vertisol of central India.

Materials and Methods

Experimental Design and Treatment Details

An incubation experiment was conducted at ICAR-Indian Institute of Soil Science, Bhopal. For this, the bulk surface soil (0–15 cm) sample was collected, air-dried and ground to pass through a 2-mm sieve. The physicochemical properties of the soil were analyzed as per the procedure described in Singh et al. [56] and are presented in Table 1. The different residues, i.e., subabul residue, biochar of subabul and FYM were collected locally and analyzed for total organic carbon content (TOC); properties are described in Table 2. Subabul (*Leucaena leucocephala*) stem and twigs were collected locally term as subabul residue and dried at 80 °C for 12 h. Further these materials were used for biochar preparation by the indigenous technique (drum method). The experiment was conducted for 75 days with treatments based on the type of organic residues and their method of placement. Three different types of residues, viz. subabul residue (T_1), biochar of subabul (T_2) and FYM (T_3), were formulated by mixing 100 g soil in 250-mL conical flasks. These residues were applied as buried

Table 1 Physicochemical properties of experimental soil

Soil properties	Value
pH (1:2.5 soil/water)	7.80
EC (dS/m at 25 °C)	0.17
Sand (%)	24.5
Silt (%)	23.0
Clay (%)	52.5
Organic carbon (g/kg soil)	4.50
Available N (mg/kg soil)	86.5
Available P (mg/kg soil)	5.7
Available K (mg/kg soil)	222.3

Table 2 Physicochemical properties of biochar

Parameters	Values
pH	9.3
EC (dS/m)	0.14
TOC (%)	74.0
Ash (%)	7.4
Biochar alkalinity (Cmol (p^+)/kg)	39.0
Ash alkalinity (Cmol (p^+)/kg)	580
Exchangeable Ca^{2+} (Cmol (p^+)/kg)	5.2
Exchangeable Mg^{2+} (Cmol (p^+)/kg)	3.3
Exchangeable K^+ (Cmol (p^+)/kg)	17.0
Exchangeable Na^+ (Cmol (p^+)/kg)	0.85

(B) (t_1), incorporated (I) (t_2) and surface applied (SA) (t_3) treatments. The highest TOC content was observed in biochar (74.0%) followed by subabul residue (47.1%) and FYM (24.2%).

Incubation Experiment for C Mineralization

For this, 100 g soil was taken into a 250-mL conical flask and was subjected to different treatments based on the C equivalent basis equal to biochar C content with three replications. Throughout the experiment, moisture content of each sample was maintained at the field capacity level. For C mineralization, vial containing 10 mL of 2 M NaOH was kept inside the conical flask in hanging position and airtight with parafilms. Simultaneously blank sample run to compute the exact C mineralization rate. Soil C mineralization was measured under controlled conditions in the laboratory at an interval of 1, 7, 15, 30, 45, 60 and 75 days after incubation. Produced CO_2–C was determined by titration of the NaOH solution with 0.5 M HCl in an excess of $BaCl_2$, using phenolphthalein as an indicator [70].

Incubation Experiment for Mineralization of N and P

The 100 g soil was transferred to moisture box, and all the treatments were formulated as in case of the above-mentioned soil incubation experiment. These moisture boxes were kept in an incubator at 25 °C temperature, and a destructive sampling was done at different time interval of 7, 15, 30, 45, 60 and 75 days after incubation. Ammoniacal nitrogen (NH_4^+–N) and nitrate nitrogen (NO_3^-–N) were determined by transferring 10 g soil to a 100-mL conical flask and 50 mL of 2 M KCl. NH_4^+–N and NO_3^-–N were measured calorimetrically using continuous-flow auto-analyzer (Sanþþ System, Skalar, the Netherlands). Phosphorus content in the samples at same interval was measured by Olsen et al. [52].

Statistical Analysis

The experiment was conducted in complete randomized design (CRD) with three replications as per the procedure described in Gomez and Gomez [30]. To assess the statistical differences among different treatments, C, N and P mineralization was analyzed via repeated measures ANOVA at the $p < 0.05$ significance level. All statistical analyses were performed using SPSS 16.0 software (SPSS Inc., Chicago, IL, USA).

Results and Discussion

Effect of Treatments on Carbon Mineralization

The cumulative amount of CO_2–C was higher in soils amended with different residues as compared to unamended soil during the incubation period. The measured ANOVA results showed that cumulative C mineralization rate was significantly affected by residue type and placement method over unamended. Surface applied treatment showed the highest rate of C mineralization as compared to other treatments followed by incorporated and buried treatments. On average, C mineralization rate in different treatments with their modes of application ranged between 14.77 and 225.5 mg CO_2–C/100 g soil after one day and 75 days of incubation, respectively, for buried; 9.20 and 237.8 mg CO_2–C/100 g soil, respectively, for incorporated; and 8.50 and 239.5 mg CO_2–C/100 g soil for surface applied treatments. Maximum C mineralization was observed under subabul residue applied treatments in which surface applied treatment varied from 19.54 at 24 h to 239.55 mg CO_2–C/100 g soil at 75 days followed by subabul incorporated (I) (17.04–237.8 mg CO_2–C/100 g soil) and subabul buried (B) (15.91–225.5 mg CO_2–C/100 g soil) treatment. On the contrary, all the biochar applied treatments remained on a par with control throughout the incubation period (Fig. 1).

Soil treated with FYM remained on a par with subabul residue applied treatments up to 15 days after incubation, but after 30 days subabul residue-treated soils showed the highest level of C mineralization over all other treatments. The response differences in total CO_2–C emission in the study were clear among all the applied treatments as compared to control soil, which is regulated by higher microbial activity and availability of C substrates [25, 44]. The cumulative C mineralization was higher for residues placed on the soil surface than for residues incorporated and buried into the soils [14]. Previous studies showed that the C mineralization rate was larger when the residues were subjected to surface applied treatment as compared to that of incorporated and buried treatments [44]. Differences in C mineralization between residues incorporated into soils and residues placed on the soil surface have been found to relate to moisture conditions and temperature [65], while moisture conditions and temperature were controlled in our incubation experiment. The incorporation of residues into soils reduced gas diffusivity and might further decrease residue decomposition rate [68]. The incorporation of organic residue in soil enhances the soil microbial population and diversity which affect the plant nutrient dynamics in soil. The C-to-N ratio of residues and microbial population affect the kinetics of organic residue.

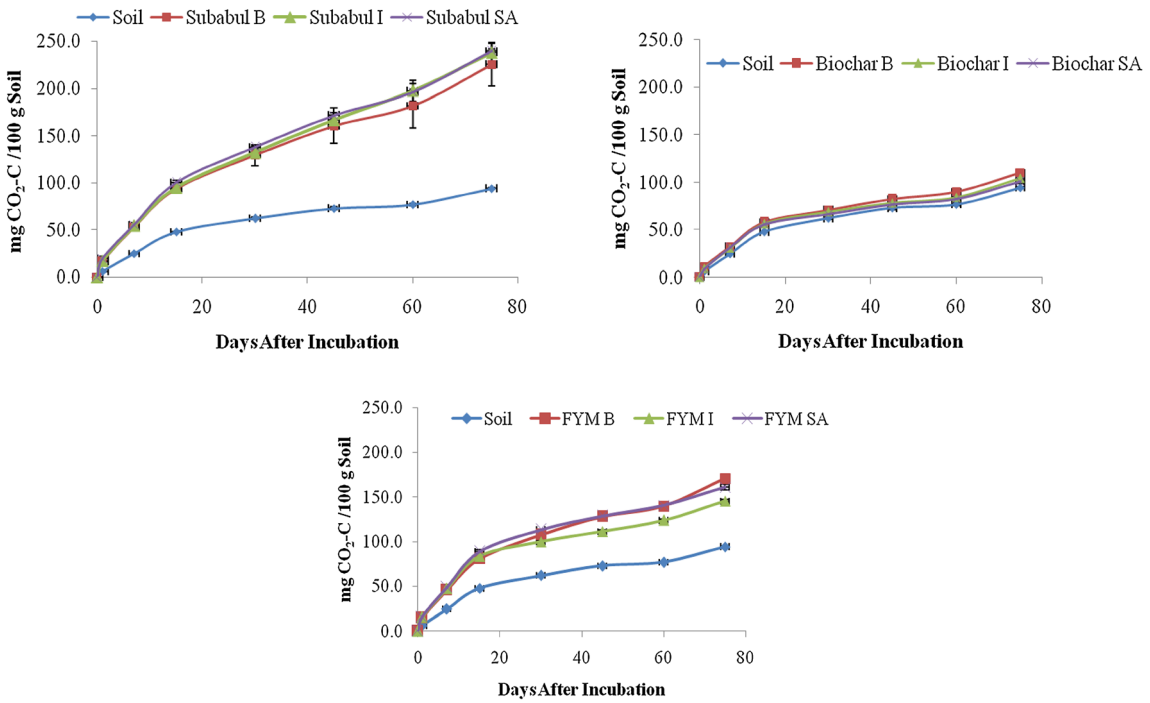

Fig. 1 Impact of organic residues and soil contact variation on carbon mineralization in soil (mg CO_2–C/100 g soil)

Although C-use efficiency could be enhanced under anaerobic conditions compared to aerobic, CO_2 emissions were higher for residues placed on the soil surface than for residues incorporated into the soils. These results indicated that the incorporation of residues into the soils inhibited residue decomposition, most likely by modifying the availability of oxygen to decomposer microorganisms [68]. Experimental results were in line with many studies conducted by other researchers [10, 44, 58], which revealed that uncharred subabul residue showed higher rates of C mineralization as compared to same material was converted to biochar. When uncharred feedstocks went through pyrolysis process, some of the elements in biochar were concentrated due to loss volatile materials, while some elements experienced concentration declined [49, 55]. Due to significant increase in C content, it increased the C/N ratio and reduced microbial activity in biochar applied treatments which ultimately affected C mineralization [8]. Conducted incubation experiments indicated that in case of biochar applied treatments only a small fraction usually less than 5% is degraded within the time fraction of laboratory incubations, depending both on the material from which it has been produced and on the processing conditions [69]. Jien et al. [39] observed that SOM stabilization by biochar application in the soil and found decreased C mineralization due to the formation of macro-aggregates which wrapped the biochar and compost and prevent from rapid decomposition by microbes [36, 38].

Effect of Treatments on NH_4^+–N and NO_3^-–N Mineralization rate

During the period of investigation, within the organic treatments no significant difference ($p = 0.05$) was found regarding net NH_4^+–N mineralization. However, different types of placement methods found significant differences between subabul and biochar and FYM (Table 3). Among the treatments, lower rate of ammoniacal nitrogen (NH_4^+–N) mineralization was observed at up to 45 days after incubation, while slight increment was observed after 60 and 75 days of incubation. Impacts of types of organic residues and their modes of application on NO_3^-–N mineralization are given in Table 4. Results revealed that subabul residue applied treatments showed the lowest rate of NO_3^-–N mineralization as compared to other treatments. Among all the treatments, subabul incorporated (I) treatment observed lowest rate of mineralization of nitrate nitrogen (NO_3^-–N) as compared to unamended soil, which varies from 0.010 mg kg^{-1} at 7 days to 4.640 mg kg^{-1} at 75 days after incubation, respectively, followed by subabul buried (B) and surface applied (SA) treatments (Table 4). Even at 30 days after incubation, no content of NO_3^-–N was observed in all the subabul residue applied treatments. NO_3^-–N mineralization rate was highest in FYM applied treatments followed by biochar and subabul residue applied treatments. Experimental results indicated that the application of subabul residue in the form of biochar mineralization was found on a par with

Table 3 Effect of the type of residue and placement application on ammoniacal nitrogen (NH_4^+–N) mineralization rate

Treatments	Days after incubation					
	7	15	30	45	60	75
Unamended soil	24.45^{b}	7.458^{bc}	13.08^{a}	4.093^{ab}	15.23^{a}	15.28^{bc}
Subabul B (T_1t_1)	24.44^{b}	5.415^{ab}	11.77^{a}	4.032^{ab}	13.43^{a}	16.06^{bcd}
Subabul I (T_1t_2)	22.11^{b}	4.748^{a}	12.38^{a}	3.671^{ab}	14.96^{a}	17.71^{d}
Subabul SA (T_1t_3)	7.707^{a}	4.24^{a}	18.87^{a}	3.288^{a}	13.82^{a}	17.08 ^{cd}
Biochar B (T_2t_1)	7.113^{a}	6.231^{abc}	12.09^{a}	3.396^{a}	14.94^{a}	16.21^{bcd}
Biochar I (T_2t_2)	7.067^{a}	4.746^{a}	10.95^{a}	3.854^{ab}	13.34^{a}	16.09^{bcd}
Biochar SA (T_2t_3)	6.237^{a}	5.553^{ab}	12.32^{a}	4.448^{ab}	14.11^{a}	16.06^{bcd}
FYM B (T_3t_1)	8.187^{a}	6.358^{abc}	12.63^{a}	4.931^{bc}	13.94^{a}	15.08^{bc}
FYM I (T_3t_2)	9.917^{a}	6.045^{ab}	12.36^{a}	5.490^{c}	13.12^{a}	14.57^{b}
FYM SA (T_3t_3)	9.317^{a}	8.077^{d}	13.12^{a}	5.937^{c}	13.43^{a}	12.85^{a}

Different letters showing significant difference at 5% level of significance

Table 4 Effect of the type of residue and placement application on nitrate nitrogen (NO_3^-–N) mineralization rate

Treatments	Days after incubation					
	7	15	30	45	60	75
Unamended soil	7.340^{b}	12.61^{b}	16.69^{b}	17.54^{b}	17.28^{b}	12.00^{b}
Subabul B (T_1t_1)	0.047^{a}	0.450^{a}	0.00^{a}	0.647^{a}	3.045^{a}	2.957^{a}
Subabul I (T_1t_2)	0.010^{a}	0.227^{a}	0.00^{a}	0.533^{a}	3.054^{a}	4.640^{a}
Subabul SA (T_1t_3)	1.350^{a}	0.540^{a}	0.00^{a}	0.558^{a}	3.124^{a}	20.87^{c}
Biochar B (T_2t_1)	10.07^{b}	10.87^{b}	17.88^{b}	22.49^{b}	32.65^{d}	31.84^{d}
Biochar I (T_2t_2)	9.47^{b}	8.253^{b}	10.96^{b}	16.04^{b}	29.44^{c}	27.85^{d}
Biochar SA (T_2t_3)	9.337^{b}	9.737^{b}	16.39^{b}	25.04^{b}	39.25^{e}	38.85^{e}
FYM B (T_3t_1)	58.69^{c}	55.99^{c}	68.71^{c}	99.99^{c}	106.3^{f}	107.8^{fg}
FYM I (T_3t_2)	56.94^{c}	58.06^{c}	67.58^{c}	102.6^{c}	112.6^{g}	111.5^{g}
FYM SA (T_3t_3)	59.74^{c}	61.50^{c}	67.87^{c}	101.1^{c}	106.5^{f}	104.8^{f}

Different letters showing significant difference at 5% level of significance

unamended soil up to 45 and increased two- to threefold up to 75 days after incubation as compared to unamended soil. Incubated results were in accordance with studies conducted by many researchers which revealed that when fresh biochar material is applied to soil because of its higher surface area and cation and anion exchange capacity, NH_4^+–N gets immobilized on the anion exchange sites of biochar at initial stages of application [8, 12, 13, 37, 66]. The define rate of NH_4^+–N was at 15 and 45 days was due to lower amount of labile N in the organic matter and promotes more microbial immobilization in soil. Shortage of labile N and more amount of recalcitrant organic N in soil lead to the imbalance in the C-to-N ratio and reduce the mineralization rate of organic residue. Such findings were also reported by Li et al. [44]. Results also indicated that biochar application stabilized SOM through sorption that rendered SOM inaccessible to microbial decomposition may have caused a reduction in NH_4^+–N mineralization [42]. Black cotton soil, relatively rich in clay, further enhanced with the application of biochar, which increased the surface acidic functional groups and adsorbed NH_4^+–N on surface [35, 43]. The primary and most consistent effects of biochar observed in this study were the increased NO_3^-–N concentration with days of incubation. Several studies have indeed demonstrated that the application of biochar in soil stimulates the nitrifying community in soil due to its porous structure, increased CEC and adsorption of NH_4^+–N and dissolved organic C on its surface [13, 42, 61]. Additionally biochar adsorbs the allelopathic molecules from soil solution that may otherwise inhibit nitrification onto the biochar surface and likewise influences nitrifying bacteria [27]. Studies conducted by DeLuca et al. [15, 16] shows that the application of biochar in soil enhanced adsorption of compound with a high C-to-N ratio onto the surface of biochar that otherwise would increase N immobilization upon decomposition is

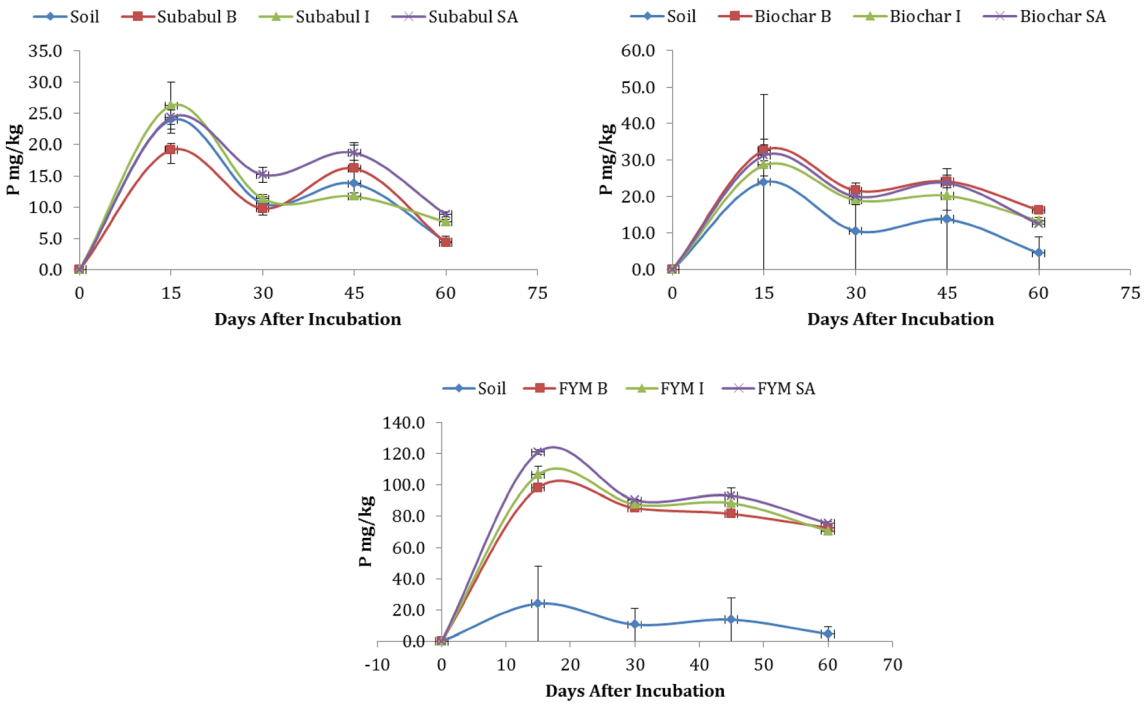

Fig. 2 Impact of organic residues and soil contact variation on phosphorus mineralization (mg kg^{-1})

likely to contribute to the stimulation of ammonification and nitrification [32]. Soil N availability may be important factor for residue decomposition [14]. The soils amended with subabul residue had a lower cumulative N mineralization than unamended soil. Furthermore, the addition of biochar resulted in a higher mineral N compared to unamended soil; the cumulative mineral N for residues placed on the soil surface was higher than for residues incorporated and buried into the soils.

Effect of Treatments on P Mineralization

Results revealed that the addition of biochar adversely mediated P mineralization rate in all the treatments along with unamended soil was observed after 60 days of incubation (Fig. 2). All the methods of subabul residue application (23.25 and 6.91 mg kg^{-1} at 15 and 60 days after incubation, respectively) were on a par with unamended soil (23.96 mg kg^{-1} and 4.49 mg kg^{-1} at 15 and 60 days after incubation, respectively) for P mineralization. Biochar-treated soil showed 1 to twofold increase in P mineralization rate, while the same was four- to eightfold in case of FYM-treated soil. Among the treatments, highest rate of P mineralization was observed when the residues were subjected to surface applied treatment followed by incorporated and buried treatments. The influence of different organic residues and their method of placement on P mineralization was generally of a smaller consistency across different treatments and days of incubation. The decrease in available P with days after incubation at the application of biochar could be a result of P sorption to biochar surfaces. A few research observations are available on P adsorption to biochar surfaces were conducted in forest soils and found small decrease in P availability after incubation [4]. Beaton et al. [5] demonstrated adsorption of P on biochar surfaces as a result of hydrogen bonding between orthophosphate and biochar surfaces. Takaya et al. [62] revealed that incomplete digestion and P adsorption by the residual biochar might have resulted in lower P mineralization. Phosphorus mineralization is much affected by the soil as well as organic residue properties. Lower concentration of P in residue slows down the mineralization process due to wider difference in C-to-P ratio in soil for proper mineralization. The soil properties much affected the P mineralization dynamics in soil and also decide the plant nutrient availability in soil solution. Phosphorus is an immobile nutrient in soil and its mineralization process is mediated by microbial biomass and their diversity in soil, which is directly or indirectly affected the phosphorus-use efficiency in soil–plant system.

Conclusions

Incubation results indicated that C mineralization was highest in subabul application than biochar and FYM application over control. Soil surface applied residues showed higher C mineralization as compared to

incorporated and buried treatments. Further, results showed that lower rate of NO_3^-–N mineralization in subabul residue applied treatments as compared to control, but when the same material was applied in the form of biochar it showed a higher rate of nitrate mineralization even 2 to 3 times higher after 45 days of incubation. Phosphorus mineralization showed a nonsignificant change with days after incubation among all the treatments. This type of study could be used in the rational use of organic residues for soil fertility management and sustainable crop production without compromising environmental health.

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