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SYNERGISM OF BIOCHAR AND LIME ON CHEMICAL PROPERTIES OF TROPICAL ACID SOILS: A MIXED-EFFECTS MODELLING APPROACH

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ABSTRACT

In tropical regions, soil acidification is one of the greatest limiting factors to agricultural productivity and has been observed in more than 50 per cent of arable soils across the world. This study compared the synergistic effect of elephant dung biochar and lime on the chemical characteristics of acidic soils in three different agro-ecological units (AEU) of Kerala, India using advanced statistical approach. Soil samples were collected from Kole land (AEU 6), North central laterite (AEU 10), and Onattukara sandy plain (AEU 3). A factorial CRD incubation experiment was conducted with treatment combinations including four biochar levels (0, 1.1, 2.2, 4.4 g kg^{-1} of soil) and three lime levels (0, 25% of lime requirement, 50% of lime requirement), and applied in 1 kg soil samples of each AEU. The temporal dynamics of pH, electrical conductivity, active acidity, exchangeable acidity, potential acidity, pH-dependent acidity and exchangeable cations were analysed at 30, 60, 90, 120 and 150 days after incubation (DAI). Mixed effects modelling indicated synergistic interaction between biochar and lime ($p<0.001$) when the amendments are co-applied, in pH, active acidity, exchangeable acidity, and exchangeable aluminium. Increase in pH of 2.80 units in soil samples taken from Kole lands, and significant relative improvements (2.06 units) in soil samples from North central laterites were resulted from the application of biochar at the rate of 4.4 g kg^{-1} of soil along with lime applied for 50% of lime requirement. In the soil samples from Onattukara sandy plains, after 60 DAI, 78% of overall pH improvement has been attained by the application of biochar at the rate of 2.2 g kg^{-1} of soil along with 50% of lime requirement. This research provides insights for the long-term field level research studies for precision soil management strategies in tropical acid soils using the combination of biochar and lime.
***Keywords*:** Soil acidity; Mixed-effects modelling; Tropical soils; Onattukara sandy plain; Kole lands

Introduction

Affecting approximately half of arable soils worldwide, soil acidification poses a threat to agricultural sustainability in a wide range of diverse ecosystems (von Uexkull & Mutert, 1995; Bolan *et al.*, 2023). It is more evident in tropical areas where severe weathering events and excessive rainfall cause acidification to surpass the buffering capacity (Guo *et al.*, 2010). The end result of accumulating exchangeable aluminium and hydrogen ions is toxicity to plant roots and interference with nutrient-cycling processes (Kochian *et al.*, 2004). The state of Kerala in India is endowed with heterogeneous pedologic conditions, which includes Kole lands, which are acid sulphate soils with extreme levels of acidity (pH<4.0); North-central laterites, soils with moderate levels of acidity and high iron-aluminium oxide levels; and Onattukara sandy plains, which are soils with low buffer capacity (Sujatha *et al.*, 2014; Ritsema *et al.*, 2000). More than 90 per cent of soils in Kerala are acidic in reaction. As much as 54 per cent of soils are extremely acidic to strongly acidic (pH 3.5 to 5.5), hindering the productivity of most crops. The widespread application of lime leads to its supply shortages and high costs, as well as the unsustainable use of finite mineral resources (Shi *et al.*, 2019). The need to identify alternative means for soil acidity amelioration has become crucially important for sustainable agricultural development and food production.

Biochar has been shown to be very beneficial in highly weathered tropical soils, soils with low pH, or soils with low cation exchange capacity and favours carbon sequestration (Lehmann and Joseph, 2015; Dai *et al*., 2017). As biochar application leads to significant changes in soil properties *viz.,* soil pH, oxides and hydroxides of Fe and Al and concentration of nutrients in soil solution, these may influence the adsorption-desorption behaviour of nutrients in the soil and subsequently its availability to the crops. Certain biowastes are highly suitable for pyrolysis and biochar production due to their high lignocellulosic content. The biochar produced from elephant-dung found to be have promising total alkalinity (2.50 meq g^{-1}) to ameliorate the soil acidity. This study evaluates the synergistic effects of biochar-lime co-application through mixed-effects modelling, quantifies the dynamic effects over time, and provides evidence-based solutions on precision soil management in acidic soils of the tropics.

Materials and Methods

Study Sites and Soil Characterization

Soil samples were collected from three representative agro-ecological units of Kerala, India. The soil samples collected from Kole land (AEU 6) are located in Thrissur district of Kerala, characterized by acid sulphate soils classified as *Typic Sulfihemists/Sulfic Epiaquepts*. Sampling locations of North central laterites (AEU 10) are situated in Malappuram district, representing highly weathered lateritic soils classified as *Ustic Kanhaplohumults*. The location of soil sampling from Onattukara sandy plains (AEU 3) are in Alappuzha district, characterized by sandy coastal soils classified as *Aquic Ustipsamments.*

Surface soil samples (0-15 cm depth) were collected during the post-monsoon period (October 2024) using stratified random sampling. Five random locations within each site were selected using GPS coordinates. Samples were air-dried under shade for 72 hours, crushed, and passed through a 2-mm stainless steel sieve. Initial soil characterization was conducted using standard analytical procedures following international protocols (Table 1).

Biochar Production and Characterization

Fresh elephant dung was collected and air-dried under shade for 10-12 days until moisture content reached below 10%. The dried material was ground using a hammer mill and passed through a 2 mm sieve. Biochar production was conducted using a laboratory-scale tubular muffle furnace under controlled pyrolysis conditions. The feedstock was loaded into stainless steel crucibles with tight-fitting lids. The furnace was programmed to rise the temperature from ambient temperature to 450°C at 10°C min^{-1}, maintained for 4 hours and then cooled slowly over 12 hours. Biochar yield was approximately 32.7% of original feedstock mass. Comprehensive characterization was conducted using standard analytical methods (Table 2).

Experimental Design and Treatments

The incubation experiment employed a factorial completely randomized design with two factors. First factor represented biochar application rates: B0 (control), B1 (1.105 g kg^{-1}), B2 (2.21 g kg^{-1}), and B3 (4.42 g kg^{-1}). Second factor represented lime application rates adjusted for each soil type based on initial pH and buffering capacity. For Kole lands the levels were L0 (control), L1- lime applied for 25% of lime requirement (0.55 g CaO kg^{-1}), and L2 - lime applied for 50% of lime requirement (1.1 g kg^{-1}). In the case of North Central laterites, the levels were L0 (control), L1- lime applied for 25% of lime requirement (0.0225 g kg^{-1}), and L2 lime applied for 50% of lime requirement (0.045 g kg^{-1}). In the case of Onattukara sandy plains, the amounts were L0 (control), L1- lime applied for 25% of lime requirement (0.05 g kg^{-1}), and L2 - lime applied for 50% of lime requirement (0.1 g kg^{-1}). The sampling done at 30, 60, 90, 120, and 150 days after incubation. Individual experimental units consisted of 1 kg portions of air-dried soil placed in high-density polyethylene containers without lids in aerobic condition. Biochar and lime amendments were thoroughly mixed using a stainless-steel spatula. Soil moisture content was adjusted to 60% of field capacity using distilled water. The incubation was conducted in a controlled environment chamber maintained at 25 ± 2°C with 65 ± 5% relative humidity. Soil samples were collected at predetermined intervals following standardized sampling protocols to ensure consistency and reproducibility. Approximately 50 g of soil was collected from each container, air-dried for 48 hours at room temperature, and subsequently ground to pass through a 2-mm sieve for chemical analysis using standard analytical procedures.

Statistical Analysis

Linear mixed-effects models were employed to analyse the hierarchical data structure. The models incorporated fixed effects for biochar rate, lime rate, soil type, incubation time, and their interactions, while treating individual containers as random effects. Temporal correlation was modelled using autoregressive correlation structure AR(1). Treatment stability was quantified using stability index calculated as the inverse of coefficient of variation for each response variable during the final 60 days. All analyses were conducted using R software version 4.3.0 with NLME (Nonlinear Mixed-Effects Models) package.

Results and Discussion

Mixed-Effects Model Analysis

Mixed-effects model revealed highly significant main effects and complex interaction patterns among all range of measured parameters. Biochar had a main effect ($p< 0.001$) on all the response variables, with F - statistic values in the range of 67.3 (available iron) to 203.6 (exchangeable acidity). Correspondingly, the significant effect of lime was obtained ($p<0.001$) in all parameters, with F-statistics ranging between 45.9 and 178.9, thus demonstrating the effectiveness of the two amendments (Table 3). The interaction between biochar and lime was found to be synergistic ($p<0.001$) for the soil parameters *viz.*, pH, active acidity, exchangeable acidity, and exchangeable aluminium, when the amendments are applied together (Figure 1). This finding is in line with recent theoretical frameworks that postulate that biochar acts through a number of parallel processes for soil acidity reduction (Shi *et al.*, 2019). The alkaline functional groups, like carbonates, hydroxides and organic bases, of biochar are involved in the pH buffering process immediately through proton consumption. The carbonate alkalinity (1.38 meq g^{-1}) reacts rapidly with soil acidity according to release water and CO_2. In addition to it, the dissolution of lime releases additional Ca^{2+} ions into the soil solution. These Ca^{2+} ions occupy the exchange sites by displacing the toxic Al^{3+} and H^+ ions into solution where they can be neutralized by biochar alkalinity.

The high specific surface area of biochar increases the cation-exchange capacity of the soil, which consequently raises the capacity of the soil to absorb the Ca^{2+} and Mg^{2+} ions released via the dissolution of lime. Its porous structure also provides it with microsites which dissolve lime thus providing lime particles with a greater reactive surface area and accelerating neutralization reactions. Biochar functional groups have the potential to complex with dissolved $Al^{3+,}$ and this reduces its action and phytotoxicity at even very low concentrations of the Al^{3+}. This mechanism of complexation justifies much of the tendency to find greater improvements in plant available pH than would be calculated by acid-neutralisation considerations. Conditional R 2 = ranged between 0.78 (available iron) and 0.94 (exchangeable acidity) so that subtle patterns in responding to soil-chemicals were well represented by the model.

Soil Type-Specific Responses

The observed differences in the reactivity between the three agro-ecological units can be explained by some basic difference in the soil chemistry and buffering mechanism (Table 4). The most evident absolute improvements were observed in the Kole lands, which could be explained by high initial acidity and by the high organic matter content. A significant pH change was observed from 4.61 to 7.42 which is equivalent to 2.80 pH units with treatment combination B3L2 (application of biochar at the rate of 4.4 g kg^{-1} along with 1.1 g kg^{-1} of lime) (Figure 2). Reduction of exchangeable acidity by 95.7% means that the primary limitation of acid-sulphate systems, that is, toxic Al^{3+} and H^+, are neutralised (Ritsema *et al.*, 2000). The North Central lateritic soils have shown highest relative gains with B_3L_2 (application of biochar at the rate of 4.4 g kg^{-1} along with 0.045 g kg^{-1} of lime), and the most consistent responses to all measured parameters. With the B_3L_2 treatment, the pH increased to 7.32, which represents an increase of 2.06 pH units. In addition, the available iron increased by 67.3% which is of special interest as lateritic soils often have problems with iron-deficiency despite their elevated total iron level (George *et al.*, 2019). The Onattukara sandy plain soils exhibited rapid early reactions, though with moderate absolute improvements. The optimal treatment was B2L2 (application of biochar at the rate of 2.2 g kg^{-1} along with 0.1 g kg^{-1} of lime), rather than highest application rates, which highlights the importance of soil-specific optimisation. The sandy soils, after 60 days, attained, 78 % of the overall pH improvement, which is characteristic of a low buffering factor and a strong reaction kinetics.

Temporal Dynamics

Temporal analysis showed that soil chemical evolution has three separate phases (Table 5). Phase-1 (0-60 days post incubation, DAI) was characterized by fast early responses with 65-78 per cent of all of the overall gains achieved. This step is a sign of immediate responses of the alkaline amendments and acidity of the soil. Phase II (60-120 D AI) was marked by a slow stabilising process system with another 20-25% of the overall improvement coming through slower processes. The system equilibrated in Phase 3 (120-150 DAI) and thereafter had only slight changes.

A detailed characterisation of the biochar generated of the elephant dung brings out the attributes that give it high efficiency. The total alkalinity of 2.50 meq g^{-1} is divided into carbonate alkalinity (1.38 meq g^{-1}), structural and organic alkalinity (0.62 meq g^{-1}), and miscellaneous inorganic alkalinity (0.45 meq g^{-1}), which provides numerous ways of neutralising acids. This profile of fractionated alkalinity is what explains the short-term bufferings of pH and the long-term bufferings of pH as seen in the time series. XRD patterns revealed calcite as the principal ability crystalline phase with other peaks attributable to dolomite and magnesite hence highlighting the carbonate cause of the alkalinity. The Fourier-transform infrared spectroscopy revealed the key functional groups such as O-H stretching (3200 -3600 cm^{-1}), carbonate CO_3^{2-} (1400-1500 cm^{-1}), and metal-oxygen bonds (600-800 cm^{-1}), as the supporting evidence of several sources of alkalinity.

Comparative Analysis

The improvements in pH (1.39-2.80 units) found in this study are substantiating the high alkalinity of biochar prepared using elephant dung and the strong initial acidity of test soils. These results support earlier studies that suggested biochar efficacy was optimized in soils of high acidity and low initial buffering capacity (Liu *et al*., 2024). These results align with the studies demonstrating biochar alkalinity as the key factor of effectiveness and provides the practical information on biochar selection and quality consideration. Also, the findings align with a recent study that showed soil pH and cation exchange capacity are always critical predictors of soil amelioration effects (Dai *et al*., 2017). High stability indices attributed to treatments demonstrate that biochar incorporation introduces long lasting changes to soil buffering capacity and this aspect validates modern research of biochar long term benefits compared to the conventional lime amendments. Regarding practical application, specific recommendations may be developed concerning each type of soil. The B_3L_2 (application of biochar at the rate of 4.4 g kg^{-1} along with 1.1 g kg^{-1} of lime treatment) will be most effective in soil samples from Kole lands with extreme acidity. In the case of north-central lateritic soils and Onattukara sandy soils, B_2L_2 (application of biochar at the rate of 2.2 g kg^{-1} along with 50% of lime requirement) was found to provides optimal benefits. The rapid response stage of the first instance suggests scheduling the applications 60-90 days before planting, and the triphasic response pattern suggests that the monitoring process should focus on 60-day intervals.

Conclusions

The synergistic effect of biochar-lime combination in ameliorating soil acidity across diverse tropical soil types has been revealed in this study. Mixed-effects modelling revealed significant synergistic interactions ($p < 0.001$) with conditional R^2 values exceeding 0.86. The treatment showed marked effects on Kole soils, 2.80 units improvement in pH and 95.7% decrease in exchangeable acidity. The lateritic soils also showed similar improvements, whereas the sandy soils found to be rapid in attaining equilibrium. Application of biochar at the rate of 4.4 g kg^{-1} of soil along with 50% of lime requirement resulted in maximum pH increase of 2.80 units in soil samples taken from Kole lands, and significant relative improvements (2.06 units) in soil samples from North central laterites. In the soil samples from Onattukara sandy soils, after 60 days of incubation, 78% of the overall pH improvement has been attained by the application of biochar at the rate of 2.2 g kg^{-1} of soil along with 50% of lime requirement. The temporal response in three-phase pattern provides practical guidance for application timing, with high treatment stability indices (0.82-0.94) indicating sustainable benefits. This research provides insights for the long-term field level research studies for precision soil management strategies in tropical acidic soils using the combination of biochar and lime.

Table 1 : Initial physicochemical properties of experimental soils

Parameter	Kole lands (AEU 6)	North central laterites (AEU 10)	Onattukara sandy plain (AEU 3)
pH	4.62 ± 0.08	5.26 ± 0.12	5.56 ± 0.09
EC (dS m^{-1})	0.05 ± 0.01	0.12 ± 0.02	0.09 ± 0.01
Organic carbon (%)	2.83 ± 0.15	0.86 ± 0.08	0.52 ± 0.06
Clay content (%)	54.2 ± 3.1	38.7 ± 2.8	8.9 ± 1.2
Active acidity (meq $100g^{-1}$)	4.40 ± 0.22	0.55 ± 0.08	1.16 ± 0.14
Exchangeable acidity (meq $100g^{-1}$)	2.82 ± 0.18	1.10 ± 0.12	3.38 ± 0.28
pH dependent acidity (meq $100g^{-1}$)	32.19 ± 1.85	18.20 ± 1.42	32.10 ± 2.15
Potential acidity (meq $100g^{-1}$)	35.01 ± 2.08	19.30 ± 1.56	35.48 ± 2.48
Exchangeable Al^{3+} (meq $100g^{-1}$)	0.32 ± 0.05	0.78 ± 0.09	1.79 ± 0.16

Exchangeable Fe^{2+} (meq $100g^{-1}$)	1.89 ± 0.12	0.16 ± 0.03	1.16 ± 0.08
Exchangeable H^+ (meq $100g^{-1}$)	0.61 ± 0.08	0.32 ± 0.04	1.59 ± 0.12
Available Fe (mg kg^{-1})	45.6 ± 3.2	9.54 ± 1.18	28.7 ± 2.4

Table 2 : Physicochemical properties of elephant dung biochar

Parameter	Value
Chemical Properties	
pH (1:10 H_2O)	10.22 ± 0.15
Electrical conductivity (dS m^{-1})	0.13 ± 0.02
Calcium carbonate equivalence (%)	10.50 ± 0.85
Alkalinity Fractions	
Total alkalinity (meq g^{-1})	2.50 ± 0.18
Structural and organic alkalinity (meq g^{-1})	0.62
Carbonate alkalinity (meq g^{-1})	1.38
Other inorganic alkalinity (meq g^{-1})	0.45
Proximate Analysis	
Ash content (%)	16.6 ± 1.2
Volatile matter (%)	33.9
Fixed carbon (%)	46.8
Ultimate Analysis	
Carbon content (%)	32.14
Nitrogen content (%)	2.20
C/N ratio	14.62
Elemental Composition	
Phosphorus (mg kg^{-1})	22.71 ± 1.85
Potassium (mg kg^{-1})	67.85 ± 4.22
Calcium (mg kg^{-1})	32.88 ± 2.95
Heavy Metal Content	
Nickel (mg kg^{-1})	9.32
Chromium (mg kg^{-1})	14.75
Lead (mg kg^{-1})	0.20

Table 3 : Mixed-effects model results for key soil chemical parameters

Response Variable	Biochar Effect	Lime Effect	B×L Interaction	R^2 (conditional)
pH	$F_{3,1347}$ = 156.7***	$F_{2,1347}$ = 134.2***	$F_{6,1347}$ = 12.8***	0.89
Active acidity	$F_{3,1347}$ = 189.4***	$F_{2,1347}$ = 167.8***	$F_{6,1347}$ = 15.2***	0.92
Exchangeable acidity	$F_{3,1347}$ = 203.6***	$F_{2,1347}$ = 178.9***	$F_{6,1347}$ = 18.4***	0.94
Exchangeable Al^{3+}	$F_{3,1347}$ = 145.8***	$F_{2,1347}$ = 123.4***	$F_{6,1347}$= 11.7***	0.86
Available Fe	$F_{3,1347}$ = 67.3***	$F_{2,1347}$ = 45.9***	$F_{6,1347}$ = 5.8**	0.78

***$p < 0.001$, **$p < 0.01$

Table 5 : Temporal analysis of pH improvement patterns across soil types

Soil Type	Treatment	Phase I (0-60 DAI)	Phase II (60-120 DAI)	Phase III (120-150 DAI)	Rate Constant (k)
Kole lands	B3L2	2.24 ± 0.18 (68%)	0.78 ± 0.13 (24%)	0.26 ± 0.09 (8%)	0.031
North central laterites	B3L2	1.92 ± 0.16 (73%)	0.56 ± 0.10 (21%)	0.16 ± 0.06 (6%)	0.039
Onattukara sandy plains	B2L2	1.35 ± 0.11 (76%)	0.35 ± 0.07 (20%)	0.07 ± 0.04 (4%)	0.045

Table 4 : Treatment responses across soil types at 150 days after incubation

Soil Type	Parameter	Initial	B0L0	Optimal Treatment	Improvement	% change	Stability Index
Kole lands	pH	4.62 ± 0.08	4.31 ± 0.12	7.42 ± 0.18 (B_3L_2)	+2.80	+60.6	0.91
	Exchangeable acidity (meq $100g^{-1}$)	2.82 ± 0.18	3.05 ± 0.24	0.12 ± 0.05	-2.70	-95.7	0.93
	Exchangeable Al^{3+} (meq $100g^{-1}$)	0.32 ± 0.05	0.35 ± 0.07	0.05 ± 0.02	-0.27	-84.4	0.89

North central laterites	pH	5.26 ± 0.12	5.16 ± 0.15	7.32 ± 0.14 (B_3L_2)	+2.06	+39.2	0.94
	Available Fe (mg kg^{-1})	9.54 ± 1.18	9.12 ± 1.34	15.96 ± 1.67	+6.42	+67.3	0.85
	Exchangeable Al^{3+} (meq $100g^{-1}$)	0.78 ± 0.09	0.85 ± 0.12	0.24 ± 0.06	-0.54	-69.2	0.88
Onattukara Sandy plains	pH	5.56 ± 0.09	5.52 ± 0.11	6.95 ± 0.13 (B_2L_2)	+1.39	+25.0	0.87
	Exchangeable acidity (meq $100g^{-1}$)	3.38 ± 0.28	3.50 ± 0.32	1.96 ± 0.18	-1.42	-42.0	0.85
	Available Fe (mg kg^{-1})	28.7 ± 2.4	27.9 ± 2.8	38.2 ± 3.1	+9.5	+33.1	0.82

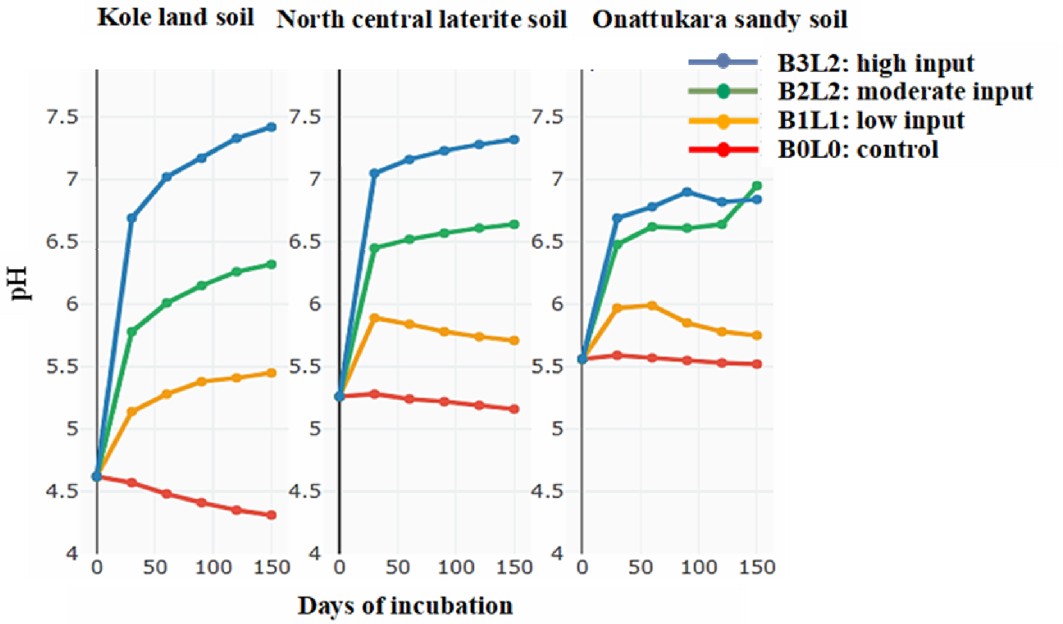

Fig. 1 : Temporal pH changes across soil types and treatment combinations. Multi-panel line plot showing pH evolution over 150 days for each soil type (Kole lands, North central laterites, Onattukara sandy plains) with different treatment combinations (B_0L_0: control, B_1L_1: low input, B_2L_2: moderate input, B_3L_2: high input)

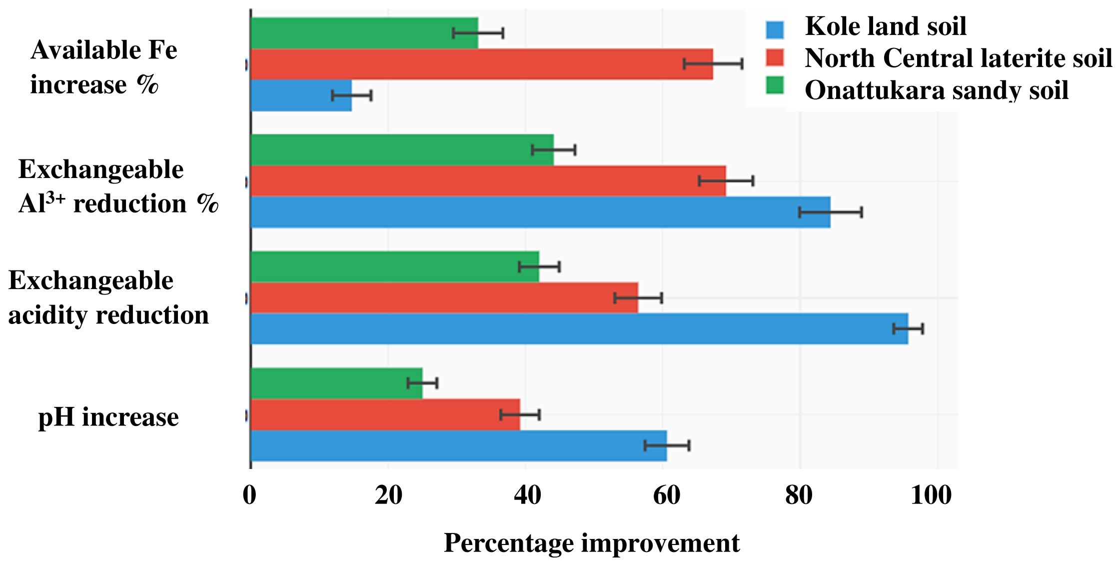

Fig. 2 : Soil type-specific treatment responses at 150 days. Bar chart showing percentage improvements for key parameters (pH increase %, exchangeable acidity reduction %, exchangeable Al^{3+} reduction %, available Fe increase %) across the three soil types for optimal treatment combinations.

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