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Short-Term Effects of Wood Ash-Based Fertilizers on the Chemical Properties of Oxisol After Cowpea Harvest

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Abstract

The response of fertilizer sources containing wood ash to liming is still poorly understood in tropical soils. The objective was to evaluate the effects of wood ash and organomineral fertilizers whether or not combined with liming on the chemical properties of an Oxisol following the harvest of cowpea (*Vigna unguiculata* (L.) Walp.) cv. BRS Guariba. The field experiment was conducted in randomized blocks with four replicates, using a 5 × 2 factorial design: no fertilizer (NF), mineral fertilizer (MF), wood ash (WA), simple-mixture organomineral fertilizer (SMOF), and pelletized organomineral fertilizer (POF), with (WL) or without liming (NL). Soil samples were collected after harvest. WA and SMOF reduced acidity and increased P, K, Ca, and Mg content without liming, while POF exhibited the highest P, K, and Ca contents when combined with limestone. Under liming conditions, POF reached 93.85 mg kg^{-1} of P, 286.14 mg kg^{-1} of K, 2.56 cmolc kg^{-1} of Ca, and 64.32% base saturation. The results demonstrate that the response of ash-containing sources depends on liming management: WA and SMOF favored soil chemical properties under natural acidity, while POF performed better when combined with limestone.

Keywords: agroindustrial waste; alternative soil amendment; acidity management; Oxisol; tropical soil; base saturation

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1. Introduction

Highly weathered tropical soils exhibit high acidity, low base saturation, and high exchangeable aluminum activity conditions that restrict nutrient availability, limit agricultural productivity, and make it difficult to maintain fertility in cultivated systems [1–3]. In these soils, the predominance of Fe and Al oxides and of mineral surfaces with pH-dependent charges favors phosphorus adsorption and reduces the retention of basic cations, increasing the reliance on soil amendments and soluble mineral fertilizers [4–6]. The instability of the input market underscores the need to utilize agroindustrial waste as soil conditioners and sources of nutrients [7,8].

The cowpea (*Vigna unguiculata* (L.) Walp.) is of dietary and socioeconomic importance in tropical regions due to its nutritional value and its role in agricultural systems [9,10]. Although adapted to high temperatures, variable water availability, and low-fertility soils, its cultivation may be limited by soil acidity and low nutrient availability in weathered soils [11]. In this study, its cultivation provided an agronomic context for evaluating changes in soil chemical properties after one growing cycle.

Organomineral fertilizers are produced by combining mineral sources and organic materials and can act to supply nutrients and improve soil condition [12]. In tropical soils, their use can alter soil acidity, nutrient availability, and exchangeable cation levels [13]. Wood ash is a byproduct with potential for agricultural use due to the presence of oxides, carbonates, hydroxides, and silicates of calcium, magnesium, and potassium, which contribute to acidity correction and the supply of nutrients to the soil [14,15].

The application of biomass ash can raise soil pH, reduce acidity, and increase the availability of Ca, Mg, K, and P in acidic soils through the addition of basic cations and its alkaline nature [16]. In different classes of acidic soils, the incorporation of biomass ash reduces exchangeable acidity and promotes the conversion of aluminum into less reactive forms, thereby improving soil chemical conditions [17]. The agricultural use of this residue also recycles nutrients and reduces environmental liabilities associated with industrial disposal [8]. However, its low bulk density and the volume required for application may limit its practical use in agricultural systems [18].

The incorporation of wood ash into organomineral fertilizers combines the nutrient supply and alkaline nature of this residue with complementary mineral sources. In Oxisols, these formulations can raise the pH and increase nutrient availability, with responses distinct from those observed for ash alone and conventional mineral fertilizers [19].

Previous studies have separately evaluated the application of wood ash, organomineral fertilizers, and limestone in acidic soils, but there is still little information on the response of ash-containing sources to liming management when compared to conditions without fertilizer and with conventional mineral fertilizer in tropical Oxisols. In this study, three ash-containing sources were evaluated alongside these two reference treatments, under conditions with and without liming. It was expected that the effects of the sources on soil acidity and nutrients would vary depending on liming management. The objective was to evaluate, after one growing cycle of cowpea (*Vigna unguiculata* (L.) Walp.) cv. BRS Guariba, the short-term effects of these combinations on the chemical properties of an Oxisol.

2. Materials and Methods

2.1. Experimental Area

The experiment was conducted under field conditions at the experimental site of the Federal University of Rondonópolis (UFR), in the municipality of Rondonópolis, Mato Grosso, Brazil, at the geographic coordinates 16°27′49′′ S and 54°34′46′′ W, at an elevation of 290 m, from September 2024 to January 2025.

The region's climate is classified as Aw according to the Köppen–Geiger classification, characterized by a rainy summer and a dry winter. The meteorological data were recorded by an automatic weather station belonging to the Graduate Program in Agricultural Engineering at the Universidade Federal de Rondonópolis, located approximately 260 m from the experimental area. During the experimental period, cumulative precipitation totaled 907.8 mm, and temperatures ranged from 21.1 °C to 41.0 °C.

2.2. Soil Characterization

The soil in the experimental area was classified as a dystrophic Red Latosol according to the Brazilian Soil Classification System [20], corresponding to an Oxisol according to

Soil Taxonomy [21]. The area had no history of agricultural cultivation or fertilization, having been subjected only to sporadic clearing and occupied by spontaneous Cerrado vegetation. This condition reduced the potential influence of residual fertilizer effects on the responses evaluated.

Prior to setting up the experiment, samples were collected from the 0.00–0.20 m layer for chemical and granulometric characterization. The analyses were performed according to the methodologies described in the Manual of Soil Analysis Methods [22]. The chemical and granulometric characterization of the soil is presented in Table 1.

Table 1. Chemical and granulometric characterization of the soil in the experimental area in the 0–0.20 m layer prior to the setup of the experiment.

pH ($CaCl_2$)	P	K	Ca	Mg	Al	H+Al	BS	CEC	O.M.	V	m
	----mg kg^{-1}----		----------		cmol$_c$ kg^{-1}	----------			g kg^{-1}	------%------	
4.0	2.90	67.0	0.30	0.25	1.07	6.07	0.72	6.79	24.10	10.60	59.78
S	B	Cu	Fe	Mn	Zn				Sand	Silt	Clay
----------	mg kg^{-1}	----------							---------	g kg^{-1}	---------
11.50	0.10	0.70	133.0	24.40	0.60				407.0	129.0	464.0

P, K, Zn, Cu, Fe, and Mn were extracted using the Mehlich-1 method; Ca, Mg, and Al, using a 1 mol L^{-1} KCl solution; H+Al, using a calcium acetate solution at pH 7.0; S, using a calcium phosphate solution; and B, using hot water. Organic matter was determined by oxidation with potassium dichromate followed by colorimetric analysis. Particle size analysis was performed using the densimeter method, with NaOH as the dispersant.

2.3. Characterization of Sources Containing Wood Ash

The wood ash used in the experiment was derived from the combustion of *Eucalyptus* sp. biomass, collected from the bottom of a furnace at an agro-industrial facility located in Rondonópolis, Mato Grosso, Brazil. The chemical composition of the original ash and the experimental batches of the organomineral fertilizers in simple mixture (SMOF) and pelletized (POF) forms was determined in a certified laboratory, in accordance with the official methods of MAPA [23]. The results of these analyses are summarized in Table 2. The values for SMOF and POF correspond to the analytical results of the batches used in the experiment and do not represent commercial guarantees. The neutralizing capacity and relative total neutralizing capacity were determined only in the original ash.

Table 2. Chemical characterization of wood ash (WA) and of the experimental batches of organomineral fertilizers in simple mixture (SMOF) and pelletized (POF) used in the experiment.

Attributes	Unit	WA	SMOF	POF
pH (H_2O)	—	9.34	9.67	9.58
pH ($CaCl_2$)	—	8.20	9.21	9.02
Organic Carbon	g kg^{-1}	60.70	122.5	183.8
N	g kg^{-1}	2.30	0.5	2.1
P_2O_5	g kg^{-1}	71.60	26.5	25.8
K_2O	g kg^{-1}	26.70	34.7	34.7
Ca	g kg^{-1}	51.70	112.2	94.0
Mg	g kg^{-1}	43.20	66.3	54.3
S	g kg^{-1}	1.60	—	—
B	g kg^{-1}	0.10	0.32	3.74
Cu	g kg^{-1}	0.04	0.06	0.06
Fe	g kg^{-1}	10.23	8.90	8.34
Mn	g kg^{-1}	0.55	0.99	0.79
Zn	g kg^{-1}	0.21	0.44	0.39
Si	g kg^{-1}	266.10	148.7	148.9

Table 2. *Cont*.

Attributes	Unit	WA	SMOF	POF
CaO	g kg^{-1}	81.45	—	—
MgO	g kg^{-1}	69.80	—	—
Neutralization power (PN)	%	35.99	—	—
Relative total neutralization power (PRNT)	%	34.05	—	—
Cd	mg kg^{-1}	<0.050	—	—
Pb	mg kg^{-1}	<0.060	—	—
Cr	mg kg^{-1}	<0.030	—	—
As	mg kg^{-1}	<0.060	—	—
Hg	mg kg^{-1}	<0.020	—	—

WA: wood ash; SMOF: organomineral fertilizer in a simple mixture; POF: pelletized organomineral fertilizer; P_2O_5: phosphorus expressed as phosphorus pentoxide; K_2O: potassium expressed as potassium oxide; CaO: calcium oxide; MgO: magnesium oxide. The symbol "—" means the parameter was not determined. The symbol "<" means the result is below the respective quantification limit.

In wood ash, the main raw material in organomineral formulations, the concentrations of Cd, Pb, Cr, As, and Hg were below their respective limits of quantification. Based on these results and the experimental scope of the study, it was assumed that the risk of these elements being contributed by the ash used was low; for this reason, they were not determined in the final batches of SMOF and POF. This assumption, however, does not allow us to demonstrate the absence of these elements or the compliance of the final organomineral fertilizers with the limits established by SDA Normative Instruction No. 27/2006, as amended by SDA Normative Instruction No. 7/2016 [24,25] since the added mineral sources may also contribute to their concentrations. The absence of these determinations constitutes a limitation of the study.

2.4. Experimental Design and Treatments

The experiment was conducted using a randomized block design, consisting of four blocks (four replicates), in a 5 × 2 factorial layout. The treatments consisted of five fertilizer sources [no fertilizer (NF), mineral fertilizer (MF), wood ash (WA), simple-blend organomineral fertilizer (SMOF), and pelletized organomineral fertilizer (POF)] and two liming treatments [with liming (WL) and without liming (NL)], totaling ten treatment combinations and 40 experimental plots. Each combination was represented once in each block, and each 9 m^2 (3 × 3 m) plot constituted an experimental unit.

The NF treatment served as the control and received no fertilizers. In the MF treatment, fertilization was carried out with 120 kg ha^{-1} of P_2O_5, using simple superphosphate (18% P_2O_5) as the source; 100 kg ha^{-1} of K_2O, using potassium chloride (60% K_2O) as the source; and 50 kg ha^{-1} of micronutrients, using Fritted Trace Elements (FTE) BR-12 as the source [B (1.8%), Cu (0.85%), S (3.9%), Fe (3.0%), Mn (2.0%), Mo (0.1%), and Zn (9.0%)], in accordance with recommendations for Cerrado soils [4,26]. WA, an agroindustrial residue from the combustion of *Eucalyptus* sp. biomass, was applied at a rate of 24 t ha^{-1}, determined based on results obtained with species of the genus *Vigna* in Cerrado Oxisol [27]. SMOF and POF were experimental organomineral formulations under development, not registered as commercial products, and were applied at a rate of 12 t ha^{-1}. SMOF was prepared by directly mixing wood ash enriched with phosphate and potash mineral fertilizers and micronutrients. To obtain POF, the ash underwent pretreatment, followed by the addition of mineral sources and pellet formation. This process is the subject of Patent Application No. BR 10 2025 014836-6, filed with the Instituto Nacional da Propriedade Industrial, Brazil. The

individual proportions of the formulation's components are not presented as they are part of the patent application. The treatments were not established based on the equivalence of nutrient loads or neutralization capacity and, therefore, should be interpreted as complete sources. Since SMOF and POF also differed in terms of processing, their comparison does not represent an isolated test of the effect of pelletization.

The second factor evaluated was liming management, established under two conditions prior to fertilization: with liming (WL), aimed at correcting acidity and increasing base saturation; and without lime (NL), in which the soil was maintained under natural acidity.

The product rates and nutrient loads applied per hectare are presented in Supplementary Table S2. A rate of 12 t ha^{-1} of SMOF and POF was set to compare their effects on soil chemical properties with those of 24 t ha^{-1} of WA and MF. The ash mass fraction in the final SMOF and POF plots was not determined directly; therefore, the total product rate does not represent the actual mass of ash applied.

2.5. Establishment and Management

The experimental area underwent conventional soil preparation by harrowing to turn the soil, remove stones, and level the experimental area. In the liming treatment (WL), commercial dolomitic limestone (PRNT = 86%, CaO = 31.0%, and MgO = 21.0%) was applied to raise the base saturation to 60% [4]. The liming requirement was calculated using the base saturation method, according to the equation $NC = [(V2 - V1) \times CEC]/PRNT$, using the initial values for base saturation and CEC presented in Table 1, where V2 = 60% and PRNT = 86%. The calculated dose was 3.90 t ha^{-1}, corresponding to 3.51 kg of limestone per 9 m^2 plot. The liming agent was distributed manually and incorporated shallowly into the 0.00–0.10 m layer 50 days before sowing. In the first few days after lime application, supplemental sprinkler irrigation was performed to ensure adequate moisture for the initial soil reaction, a period followed by the onset of regular rainfall. In the no-liming treatment (NL), the soil was maintained in its natural acidic condition.

Fertilizers were applied at the time of seeding, according to the rates defined for each treatment. MF and POF were applied locally along the planting row at depths of 0.00–0.08 m and 0.00–0.10 m, respectively. WA and SMOF were broadcast and superficially incorporated into the 0.00–0.10 m layer using hoes. Since the application location differed among the sources, the effects of composition, processing, and application method were not evaluated independently.

The cowpea (*Vigna unguiculata* (L.) Walp.), cultivar BRS Guariba, was sown manually in six rows per plot, with a row spacing of 0.50 m and a density of 10 plants per linear meter. The cowpea crop was used as an agronomic context to evaluate, after harvest, the short-term effects of the treatments on soil chemical properties. Cultural management consisted of manual weeding to control weeds.

2.6. Soil Sampling and Chemical Analyses

The cowpea harvest was conducted 82 days after sowing, and soil samples were collected the following day from the 0.00–0.20 m layer. In each experimental unit, three subsamples were collected from the usable area of the plot, between the four central rows and excluding the edge rows. The subsamples were obtained using a Dutch auger, with one collected along the crop row and the others approximately 7 and 10 cm from the row. Subsequently, the subsamples were homogenized to obtain a composite sample per plot. The same sampling strategy was used for all treatments. Since the fertilizers differed in terms of application location, the composite samples may partially reflect differences in the spatial distribution of the inputs within the plots.

The following soil chemical properties were evaluated: pH in $CaCl_2$, exchangeable aluminum (Al^{3+}), potential acidity (H+Al), base sum (BS), cation exchange capacity at pH 7.0 (CEC), base saturation (V%), aluminum saturation (m%), phosphorus (P), potassium (K), calcium (Ca), magnesium (Mg), and sulfur (S).

Chemical analyses were performed according to the methodologies described in the Manual of Soil Analysis Methods [22]. Soil pH was determined in a 0.01 mol L^{-1} $CaCl_2$ solution at a soil-to-solution ratio of 1:2.5. P and K contents were determined using the Mehlich-1 method. The Ca, Mg, and Al contents were obtained by extraction with a 1 mol L^{-1} KCl solution; potential acidity (H+Al) was estimated using the SMP method; and S was extracted with a calcium phosphate solution. The base sum was calculated as BS = Ca + Mg + K; the cation exchange capacity at pH 7.0 as CEC = BS + H+Al; base saturation as V = 100 × BS/CEC; and aluminum saturation as m = 100 × Al/(BS + Al).

2.7. Statistical Analysis

The normality of the residuals and the homogeneity of variances were assessed using the Shapiro–Wilk and Bartlett tests, respectively (Supplementary Table S1). pH, P, and m% were analyzed after log(x + 1) transformation to meet the assumptions of analysis of variance; the remaining variables were analyzed on their original scale. Means are presented on their original scale.

For each attribute, the model $Y_{ijk} = \mu + B_i + F_j + L_k + (F \times L)_{jk} + \varepsilon_{ijk}$, was fitted, where B_i represents the block, F_j the fertilizer source, L_k the liming treatment, $(F \times L)_{jk}$ the interaction, and εijk the experimental error.

Effects were evaluated using the F-test of ANOVA ($\alpha = 0.05$). When the interaction was significant, it was broken down by analyzing the fertilizer sources within each liming treatment and the treatments within each source. Simple effects were initially assessed using the F-test, and when significant, the means were grouped using the Scott–Knott test at the 5% level, using the ExpDes package (version 1.2.2) [28]. In the absence of a significant interaction, the main effects were interpreted, with grouping performed using the Scott–Knott test when significant. The analyses were conducted in R 4.5.0 [29].

Pearson's correlation ($p < 0.05$) and PCA utilized the 40 experimental observations. The correlation analysis included the 12 chemical attributes evaluated (pH, Al, H+Al, BS, CEC, V%, m%, P, K, Ca, Mg, and S) and was calculated using the Hmisc package (version 5.2-3) [30]. PCA was performed using the prcomp() function from the stats package after centering and standardizing pH, Al, H+Al, P, K, Ca, Mg, and S. BS, CEC, V%, and m% were excluded because they are mathematically derived from other attributes.

Model diagnostics were supplemented by visual inspection of the normal probability plots of the residuals (Supplementary Figures S1 and S2) and the plots of residuals versus fitted values (Supplementary Figures S3 and S4).

3. Results

3.1. Soil Acidity and Chemical Fertility

The interaction between fertilizer sources and liming management was significant for Al, H+Al, BS, CEC, V%, and m%, while pH showed only main effects. The complete ANOVA results and model diagnostics are presented in Supplementary Table S1. Soil pH was influenced by fertilizer sources and liming management, with no interaction between these factors (Figure 1). WA, SMOF, and POF had higher values than NF and MF, without differing from one another (Figure 1a), and liming increased the pH compared to the treatment without limestone (Figure 1b).

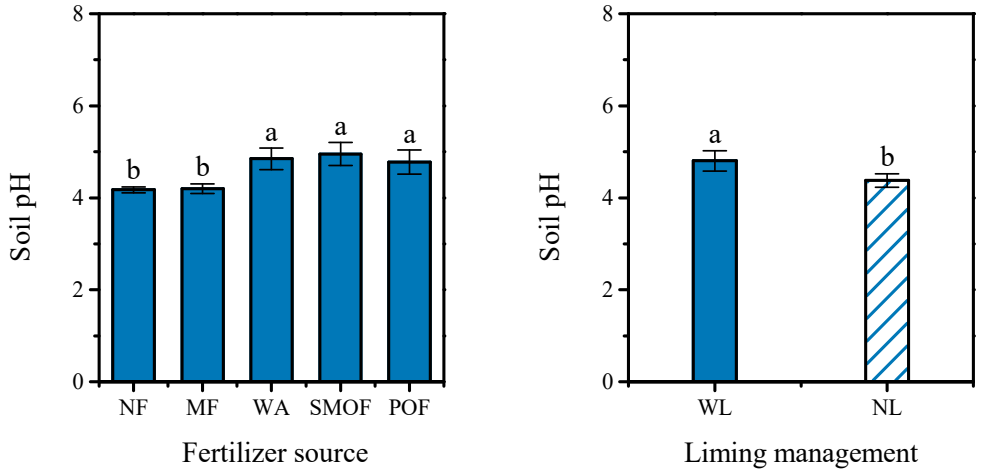


Figure 1. Main effects of fertilizer sources (**a**) and liming management (**b**) on soil pH in $CaCl_2$ after the harvest of cowpea (*Vigna unguiculata* (L.) Walp.) cv. BRS Guariba in an Oxisol. NF: no fertilizer; MF: mineral fertilizer; WA: wood ash; SMOF: simple-mixture organomineral fertilizer; POF: pelletized organomineral fertilizer; WL: with liming; NL: without liming. Means followed by different letters differ according to the Scott–Knott test ($p < 0.05$), both for the fertilizer sources in panel (**a**) and for the liming treatments in panel (**b**). Means and standard errors are presented on the original scale; comparisons were made after log(x + 1) transformation. Vertical bars represent the standard error of the mean.

WA and SMOF reduced Al, H+Al, and m% even without liming, while NF, MF, and POF-NL exhibited higher acidity (Figure 2a,b,f). In combination with liming, POF reduced Al from 1.17 to 0.29 cmolc kg^{-1}, corresponding to a 75.2% reduction, and increased BS, CEC, and V%, reaching 64.32% base saturation (Figure 2c–e).

3.2. Soil Macronutrients

The interaction between fertilizer sources and liming treatment was significant for P, K, Ca, and Mg, but not for S. The complete ANOVA results and model diagnostics are presented in Supplementary Table S1. The effects of fertilizer sources on P and K depended on the liming treatment (Figure 3a,b). Without liming, WA and SMOF had the highest P contents, at 68.76 and 73.41 mg kg^{-1}, respectively, while under liming, POF reached 93.85 mg kg^{-1}. Liming reduced P contents in WA and SMOF and increased this content more than fivefold in POF (Figure 3a). For K, the highest content under natural acidity was observed in SMOF (241.75 mg kg^{-1}), followed by WA and POF. When combined with liming, POF exhibited the highest K content, reaching 286.14 mg kg^{-1}, while SMOF showed a reduction in this attribute (Figure 3b).

A similar pattern was observed for the basic cations Ca and Mg (Figure 3c,d). Without liming, WA and SMOF had the highest Ca contents, at 2.04 and 2.08 cmolc kg^{-1}, respectively. Under liming, however, POF exhibited the highest Ca content (2.56 cmolc kg^{-1}), a value 4.6 times higher than that observed without limestone (Figure 3c). For Mg, POF, SMOF, and WA exhibited higher contents than NF and MF under liming, with no significant differences among them. Among the ash-containing sources, liming increased the Mg content only in POF, from 0.37 to 1.95 cmolc kg^{-1} (Figure 3d).

No statistically significant differences in S concentrations were detected among the fertilizer sources, liming treatments, or their interaction (Supplementary Table S1).

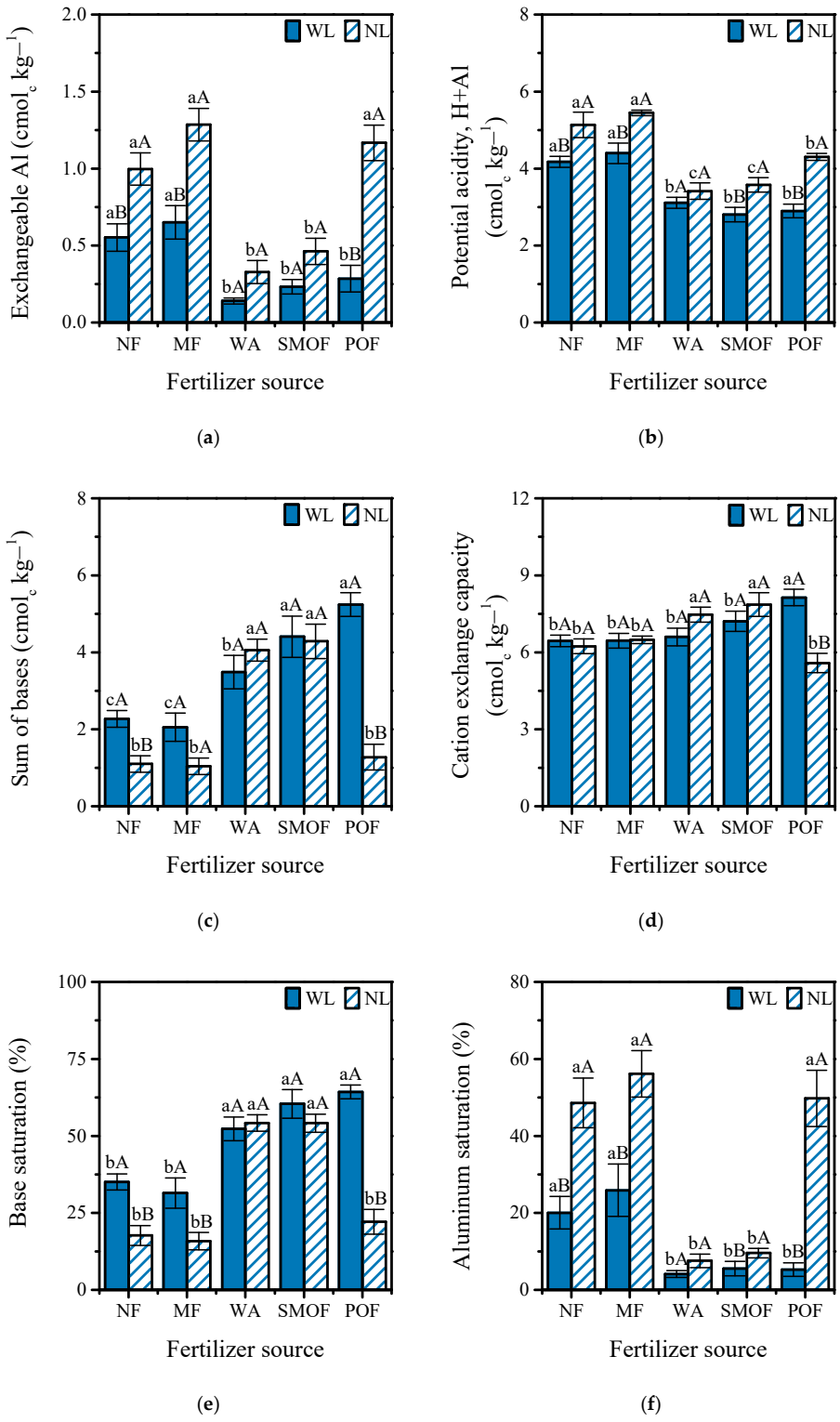


Figure 2. Interaction between fertilizer sources and liming management on exchangeable aluminum (Al^{3+}) (**a**), potential acidity (H+Al) (**b**), base sum (BS) (**c**), cation exchange capacity (CEC) (**d**), base saturation (V%) (**e**), and aluminum saturation (m%) (**f**) in soil collected after the harvest of cowpea (*Vigna unguiculata* (L.) Walp.) cv. BRS Guariba in an Oxisol. NF: no fertilizer; MF: mineral fertilizer; WA: wood ash; SMOF and POF: simple-mixture and pelletized organomineral fertilizers, respectively; WL: with liming; NL: without liming. Lowercase letters compare fertilizer sources within each liming treatment, and uppercase letters compare liming treatments within each fertilizer source, using the Scott–Knott test ($p < 0.05$). Vertical bars represent the standard error of the mean.

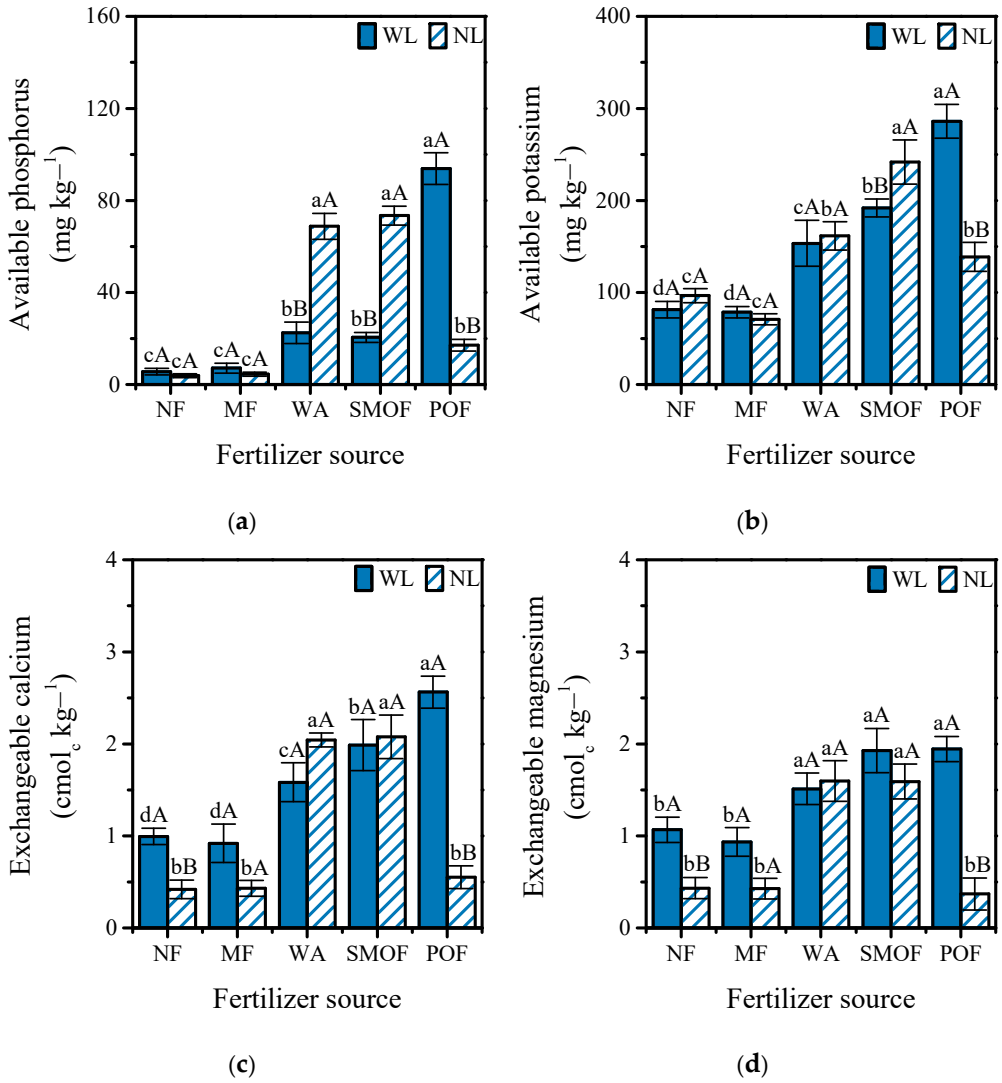


Figure 3. Interaction between fertilizer sources and liming management on the levels of available phosphorus (**a**), available potassium (**b**), exchangeable calcium (**c**), and exchangeable magnesium (**d**) in the soil after the harvest of cowpea (*Vigna unguiculata* (L.) Walp.) cv. BRS Guariba on Oxisol. NF: no fertilizer; MF: mineral fertilizer; WA: wood ash; SMOF: simple-mixture organomineral fertilizer; POF: pelletized organomineral fertilizer; WL: with liming; NL: without liming. Lowercase letters compare fertilizer sources within each liming treatment, and uppercase letters compare liming treatments within each fertilizer source, using the Scott–Knott test ($p < 0.05$). Means and standard errors are presented on the original scale; for P, comparisons were performed after log(x + 1) transformation. Vertical bars represent the standard error of the mean.

3.3. Correlation and Principal Component Analysis (PCA)

Pearson correlation analysis revealed positive associations between pH, BS, V%, CEC, Ca, Mg, K, and P, and negative associations between these attributes and Al, H+Al, and m% (Figure 4). S did not correlate with the other attributes. Some of the correlations involving BS, CEC, V%, and m% result from mathematical dependence, as these indicators are calculated based on other attributes.

The first two PCA components explained 82.38% of the variance: 69.60% in PC1 and 12.78% in PC2 (Figure 5; Supplementary Table S3). PC1 exhibited positive loadings for pH, P, K, Ca, and Mg, and negative loadings for Al and H+Al, separating higher pH values and macronutrients from higher acidity. The negative loading of S defined PC2. NF and MF were concentrated on the negative side of PC1, while WA and SMOF were on the positive

side, with and without liming. POF showed the greatest contrast among the treatments: positive with liming and negative without liming.

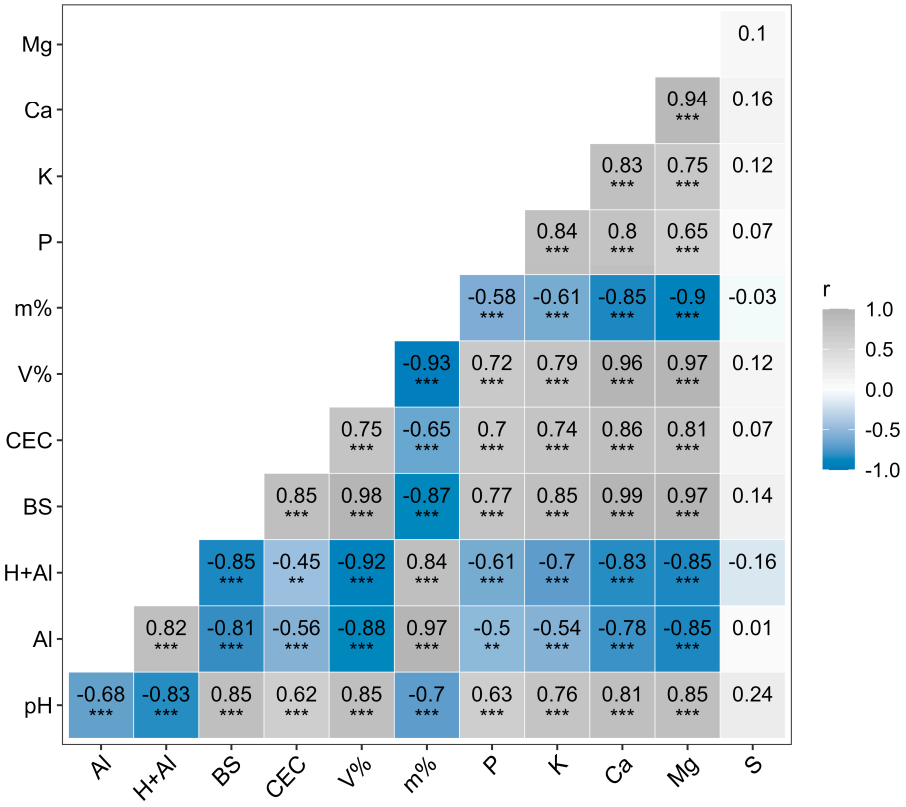


Figure 4. Pearson correlations among the chemical attributes of the Oxisol following the harvest of cowpea (*Vigna unguiculata* (L.) Walp.) cv. BRS Guariba. BS: base sum; CEC: cation exchange capacity at pH 7.0; V%: base saturation; m%: aluminum saturation. ** $p < 0.01$; *** $p < 0.001$.

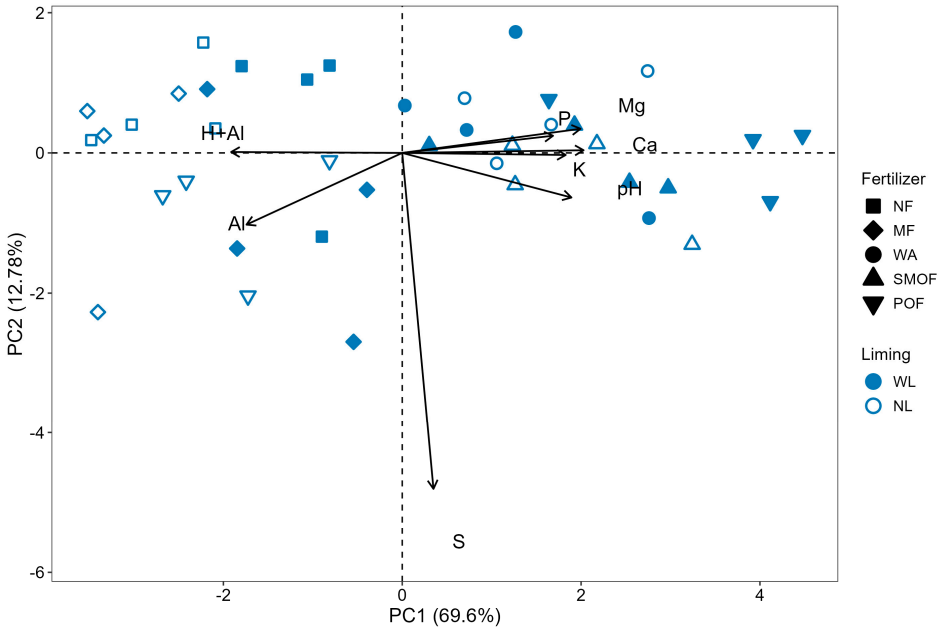


Figure 5. PCA biplot of pH, Al, H+Al, P, K, Ca, Mg, and S in Oxisol after the harvest of cowpea (*Vigna unguiculata* (L.) Walp.) cv. BRS Guariba. Al: exchangeable aluminum; H+Al: potential acidity; NF: no fertilizer; MF: mineral fertilizer; WA: wood ash; SMOF: simple-mixture organomineral fertilizer; POF: pelletized organomineral fertilizer; WL: with liming; NL: without liming. The dashed lines indicate the zero-reference lines for PC1 and PC2.

4. Discussion

4.1. Soil Acidity and Chemical Fertility

The increase in pH in WA, SMOF, and POF, compared to NF and MF, and the reductions in Al, H+Al, and m% observed mainly in WA and SMOF without liming and in POF with liming show that these treatments altered soil acidity under the conditions evaluated (Figures 1 and 2). Wood ash is alkaline in nature and may contain oxides, carbonates, and hydroxides of Ca and Mg [14,31], whose reactions contribute to the neutralization of acidity and the reduction in Al activity in the soil solution [18,32]. The formation of less soluble Al compounds may have contributed to the lower values of Al, H+Al, and m%, but these mechanisms were not directly evaluated.

WA and SMOF reduced Al, H+Al, and m% even without liming, consistent with responses reported for other acidic soils treated with biomass ash and organomineral fertilizers. In incubation experiments, the application of ash raised the pH from 4.22 to 6.60 and reduced exchangeable Al from 2.85 to less than 0.05 $cmol_c\ kg^{-1}$ [17]. In Oxisol cultivated with cowpeas, an organomineral fertilizer formulated with ash raised the pH to 6.2 without liming [19]. These studies show responses similar to those observed in this experiment, but do not allow for separate quantification of the ash's contribution or the neutralization capacity of the evaluated formulations.

WA and SMOF exhibited lower levels of Al, H+Al, and m% under natural acidity, while the highest values for these attributes occurred in NF and MF. WA and SMOF were broadcast and incorporated into the topsoil layer, while POF was applied in the seed row. In Oxisol, the incorporation of wood ash increased pH, P, and K compared to surface application [33], indicating that contact between the material and the soil influences chemical transformations after application. Since composition, particle size distribution, and fertilization location varied simultaneously, it is not possible to separate the contribution of these factors to the responses of WA, SMOF, and POF.

MF and NF showed similar values for attributes related to acidity. The supply of P, K, and micronutrients by the mineral fertilizer did not reduce Al, H+Al, or m% under the evaluated conditions. Conventional mineral fertilizers are formulated to supply nutrients and do not contain components intended to correct soil acidity [34,35]. Under natural acidity, the mineral fertilizer did not alter these attributes compared to the unfertilized control.

Increases in BS, V%, and CEC accompanied the increase in Ca, Mg, and K and the reduction in acidity in WA, SMOF, and POF. In Oxisols, an increase in pH can enhance the dissociation of pH-dependent charges and the number of cation exchange sites, thereby increasing nutrient retention in the exchange complex [1,2]. In acidic soil treated with rice husk ash, base saturation increased from 67.9 to 89.2%, and effective CEC increased from 5.20 to 7.86 $cmolc\ kg^{-1}$ after 60 days of incubation [36]. In Oxisol, the application of wood ash increased base saturation from 12.79 to 54.79% and CEC from 7.26 to 8.74 $cmol_c\ dm^{-3}$, accompanied by the removal of exchangeable Al at the highest doses evaluated [27]. In this study, the reduction in acidity occurred alongside an increase in base cations and indicators of soil chemical fertility.

4.2. Soil Macronutrients and Exchangeable Bases

Post-harvest levels of P, K, Ca, and Mg differed among WA, SMOF, POF, and MF in association with changes in acidity and the amounts of nutrients supplied. However, residual contents were not proportional to the applied rates (Supplementary Table S2). WA supplied the highest amounts of P_2O_5, K_2O, and Mg but did not result in the highest residual contents under all conditions. POF exhibited the highest P, Ca, and Mg contents under liming without supplying the corresponding highest amounts. Therefore, the applied rate alone does not explain the soil's chemical properties after harvest.

In WA and SMOF, the highest P and K levels occurred without liming. The reduction in acidity observed in these treatments coincided with higher concentrations of these nutrients in the soil. In Oxisol, the application of wood ash raised available P levels from approximately 1.5 mg dm^{-3} to values exceeding 15 mg dm^{-3} and substantially increased K levels after incorporation into the soil [33]. In fertilizers formulated with biomass ash, the increase in pH from 4.30 to values close to 6.20 was accompanied by an increase in nutrient availability under naturally acidic conditions [19]. The rise in pH can reduce phosphate adsorption on the surfaces of Fe and Al oxides and increase the extractable fraction of this nutrient [37]. This process may have contributed to the results for WA and SMOF, but its contribution was not directly measured in this study.

Among the treatments evaluated, POF exhibited the highest P contents under liming. This result coincided with a decrease in H+Al and an increase in base saturation. In systems fertilized with granular organomineral fertilizers, higher residual P concentrations have been associated with lower nutrient retention in the soil and its release from the organomineral fertilizer [7]. An increase in OH^- concentration may also alter the competition between phosphate and soil adsorption sites [6]. In the present study, however, no measurements of retention, dissolution, or release over time were performed. Thus, the results demonstrate greater residual P availability in the POF treatment combined with liming, without identifying the mechanism responsible for this response.

K contents did not proportionally follow the amounts applied by the sources. In Oxisol, the application of wood ash increased K contents from 79.91 to 372.18 mg dm^{-3} at the highest doses evaluated [27], while increases in more than 200% were recorded after ash incorporation under field conditions [33]. The solubility of potassium salts present in the ashes may contribute to these responses. However, WA supplied more K_2O than SMOF and POF (Supplementary Table S2), without exhibiting the highest residual levels under all conditions, indicating that other soil processes also influenced the availability of this nutrient after harvest.

Ca and Mg contents followed the reduction in acidity and the increases in BS, V%, and CEC. In Oxisol, the application of wood ash increased Ca contents from 0.48 to 2.79 cmolc dm^{-3} and Mg contents from 0.24 to 1.07 cmolc dm^{-3} [27]. In acidic soil treated with rice husk ash, Ca concentrations increased from 1.85 to 2.93 cmolc kg^{-1} and Mg concentrations from 0.95 to 2.11 cmolc kg^{-1} after 60 days of incubation [36]. In this study, WA and SMOF exhibited higher levels of these nutrients without liming, while POF showed the highest values with limestone. The residual Ca and Mg levels did not necessarily correspond to the highest applied rates (Supplementary Table S2), indicating the combined influence of nutrient supply and soil chemical changes.

No differences in S concentrations were detected among the fertilizer sources, liming treatments, or their interaction. In Oxisol cultivated under controlled conditions, the application of wood ash increased S concentrations from 11.08 to 76.20 mg dm^{-3}, while organomineral fertilizers increased concentrations to approximately 40.70 mg dm^{-3} [38]. This behavior differs from that observed in this study. The accumulated precipitation of 907.8 mm and the mobility of sulfate in the soil profile may have influenced the concentrations determined after harvest. Since the soil was evaluated at a single depth and at a single point in time, it was not possible to characterize the dynamics of S throughout the cycle.

In summary, WA and SMOF maintained higher availability of P, K, Ca, and Mg under natural acidity. Under liming, POF exhibited the highest P, K, and Ca contents; for Mg, it did not differ from WA and SMOF. Since the highest residual contents did not necessarily coincide with the highest amounts of nutrients supplied by the treatments, nutrient supply alone does not explain the post-harvest patterns.

4.3. Pearson Correlation and Principal Component Analysis

The associations observed among soil chemical properties indicated a gradient linking higher values of pH, P, K, Ca, and Mg to lower values of Al and H+Al. In highly weathered Oxisols, an increase in pH can reduce Al activity and alter nutrient retention, favoring the accumulation of exchangeable bases and available nutrients [1,6,15]. Some of these associations should be interpreted with caution because certain chemical indicators are mathematically derived from other attributes. The absence of a significant correlation between S and the other attributes merely indicates that its variation did not follow this gradient linearly; processes associated with sulfate mobility constitute a possible explanation, which was not directly evaluated in this study.

PCA summarized the covariation among the measured attributes and showed that most of the variability among the observations was associated with a gradient contrasting acidity and nutrient availability. This pattern is consistent with the chemical differences observed among the treatments, but it does not identify independent processes nor does it allow for attributing causality to mechanisms of nutrient supply, retention, or release. Since the analysis used directly measured attributes and excluded BS, CEC, V%, and m% due to their mathematical dependence, its interpretation was restricted to the multivariate description of soil chemical conditions after harvest.

4.4. Temporal Persistence of Effects and Environmental Considerations

Soil chemical attributes were assessed once, after harvest, and describe soil conditions 82 days after sowing. The concentrations of P, Ca, and Mg in POF under liming do not allow for inferring gradual release, residual effects, or the maintenance of these attributes in subsequent crops. Successive sampling is necessary to assess the evolution of nutrients in the soil profile and the duration of the treatments' effects.

The agricultural use of ash-derived materials also requires attention regarding the accumulation of potentially toxic trace elements in the soil. In different types of biomass ash, the chemical composition can vary depending on the plant species used, combustion conditions, and the origin of the raw material [39]. In the ash used in this study, Cd, Pb, Cr, As, and Hg had concentrations below their respective analytical limits of quantification (Table 2); these results do not indicate the absence of these elements in the analyzed sample.

The available data do not allow for an assessment of the environmental safety of the final organomineral fertilizers. Cd, Pb, Cr, As, and Hg were not determined in the final batches of SMOF and POF; therefore, it is not possible to infer their presence, concentration, or compliance with the limits established by Brazilian legislation. The study also did not evaluate the accumulation of these elements in the soil, their leaching, or their presence in plant tissues. Future studies should characterize the final materials and monitor the soil and plants after successive applications, as well as the distribution of these elements within the soil profile.

5. Conclusions

Wood ash and organomineral fertilizer in a simple mixture reduced acidity and increased the P, K, Ca, and Mg contents and the base saturation of the Oxisol even without liming. When combined with limestone, the pelletized organomineral fertilizer achieved the highest levels of P, K, Ca, and V%. Since the treatments differed in composition, dose, processing, and application method, the results characterize the combinations evaluated but do not isolate the effect of pelletization nor demonstrate agronomic equivalence among the sources. Inferences are limited to one cowpea growing cycle and a single post-harvest sampling; successive evaluations, including yield, biomass, nutrient uptake, and trace element

monitoring, are necessary to determine the persistence and agronomic and environmental relevance of the responses.

6. Patents

The formulation of the wood ash-based pelletized organomineral fertilizer, as well as its production process and the resulting product, are the subject of Patent Application No. BR 10 2025 014836-6, filed with the Instituto Nacional da Propriedade Industrial (INPI), Brazil.

Supplementary Materials: The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/soilsystems10110137/s1, Table S1 presents the analysis of variance results expressed as F-statistics with corresponding *p*-values, coefficients of variation, overall means, and residual normality and homoscedasticity tests for soil chemical attributes under different fertilizer sources and liming management. Table S2 presents the product doses and applied nutrient loads, and Table S3 presents the loadings of the variables in PC1 and PC2. Figures S1–S4 present the residual diagnostic plots for the fitted models.

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Conflicts of Interest: Edna Maria Bonfim-Silva, Tonny José Araújo da Silva, Niclene Ponce Rodrigues de Oliveira, Luana Aparecida Menegaz Meneghetti and Alessana Franciele Schlichting are listed as inventors on Patent Application No. BR 10 2025 014836-6, filed with the Instituto Nacional da Propriedade Industrial (INPI), Brazil, with the applicant being the Universidade Federal de Rondonópolis (UFR). The application relates to the formulation of the wood ash-based pelletized organomineral fertilizer evaluated in this study, its production process, and the resulting product. The other authors declare no conflicts of interest. The patent application did not influence the study design, data collection and analysis, interpretation of results, or manuscript preparation. The patented material was used solely as a fertilizer in this study.

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