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Effect of Lime Particles Size and Application Rate on Soil Acidity and Base Cation Dynamics

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Abstract: Liming is a common practice for improving the quality of soils affected by acidity, due to climate change, acid rain, nitrate leaching, and oxidation of sulphide minerals. This study aimed not only to assess the efficiency of fine and coarse lime application at 3 t/ha and 6 t/ha doses in mitigating soil acidity, but also to determine which of the options has a stronger impact on improving subsoil characteristics. Over a period of two years, several key parameters were monitored: pH, exchangeable base cations (Ca_{exch}, Mg_{exch}, and K_{exch}), and Al_{exch}. In the topsoil, the highest increase in pH was observed 6 months after the ap-plication of 3 t/ha fine lime, while in the subsoil, only a modest increase was noted. The highest values of exchangeable base cations in the topsoil were: Ca_{exch} = 9.06 cmol/kg soil; Mg_{exch} = 1.15 cmol/kg soil; K_{exch} = 0.360 cmol/kg soil. These were recorded 24 months after applying 6 t/ha of fine aglime, and were higher than those obtained in the subsoil. While in topsoil Al_{exch} reached 0 at T3, in subsoil, the largest decrease was 38% at T3. The release rate of ca Ca and its mobility depend on the particle size and dose of aglime, playing an important role in alleviating aluminium toxicity.

Keywords: pH, exchangeable calcium, exchangeable magnesium, cation exchange capacity

1. Introduction

Soil acidification has a significant impact on reducing crop productivity and damaging terrestrial and aquatic ecosystems. Approximately 4 billion hectares of land worldwide are estimated to suffer from varying levels of acidity [1]. While soil acidity typically increases gradually in natural ecosystems, extensive studies have revealed that soil pH can shift rapidly from alkaline to acidic when annual precipitation surpasses annual potential evapotranspiration [2, 3]. Major factors driving soil acidification include nitrate leaching, caused by ammonium fertilizers and biological nitrogen fixation, removal of crop products, accumulation of organic matter, acid rain and oxidation of sulphide minerals [4].

Acidic soils are characterized by low pH, reduced cation exchange capacity (CEC), and decreased base saturation. Natural processes like the leaching of cations and decomposition of organic matter alter soil pH. The displacement of essential basic cations, which move deeper into the soil, is harmful as they are replaced by elements like aluminium, iron, manganese, and hydrogen [5]. This chemical shift reduces nutrient availability, thereby lowering crop yields [1].

The chemical properties of soil, including plant nutrient availability, are heavily influenced by pH. When soil pH falls below 5.5, deficiencies in nutrients such as nitrogen, phosphorus, potassium, sulphur, calcium, magnesium and molybdenum occur, making a pH increase necessary for optimal crop growth [6]. Soil fertility depends heavily on CEC, with lower CEC leading to faster pH changes and weaker buffering capacity [7].

Tian and Niu (2015) [8] conducted a meta-analysis of 106 studies to identify global patterns of soil acidification. Their findings revealed that nitrogen addition globally reduced soil pH by an average of 0.26, shifting soils into the Al^{3+} buffering phase. In Romania, the use of chemical fertilizers

per hectare increased by 63.33% from 60 kg a.s./ha in 2007 to 98 kg a.s./ha in 2020, with 60 kg a.s./ha being nitrogen primarily applied as ammonium nitrate and urea [9]. The annual application of nitrogen fertilizers contributes to soil acidification, primarily through nitrate leaching and the release of hydrogen ions during the conversion of ammonium-based fertilizers [10]. Over time, these processes lower soil pH, reduce cation exchange capacity, and decrease base saturation. Base cations such as Ca^{2+}, Mg^{2+}, and K^+ play a crucial role in buffering against nitrogen-induced soil acidification in its early stages.

Liming is a widely adopted method for improving the quality of acidic soils, providing both direct and indirect benefits. It influences soil acidity, nutrient mobility, heavy metal behavior, aggregate stability, and biological activity [11]. Studies show that lime reduces exchangeable and soluble aluminum, increases soil pH, and enhances the availability of nutrients like phosphorus, nitrogen, calcium, and magnesium [12]. Additionally, liming promotes microbial activity, boosts organic matter decomposition, facilitates nutrient absorption, and strengthens soil structure by forming stable aggregates. By balancing soil pH, lime decreases toxicity caused by hydrogen, aluminum, and manganese [13, 14]. It produces bicarbonate (HCO_3^-) and hydroxide (OH^-), which neutralize H^+ and Al^{3+} ions, raising soil pH through reactions with carbon dioxide and water. Research by Daba et al. [15] highlights the significant improvement in wheat and maize yields due to lime's role in elevating soil pH, increasing exchangeable base cations (e.g., Ca^{2+} and Mg^{2+}), and reducing aluminum toxicity, with pH adjustment being the most impactful. The required lime amount depends on factors such as soil acidity severity, soil properties, land use, and fertilizer application methods. However, the effectiveness of various lime types in improving soil chemical properties is not well-documented. Field efficiency of lime application is influenced by material fineness, even distribution, and thorough soil mixing; yet, due to limited effectiveness in field conditions, application rates are often doubled. Moreover, treating acidic subsoil presents a significant challenge, as surface lime application does not effectively address subsoil acidity [4].

Calcium carbonate, despite being a limited natural resource, remains the most widely used amendment for neutralizing acidic soils due to its cost-effectiveness and efficiency [3]. By neutralizing excess hydrogen ions, it enhances agricultural productivity [16]. Additionally, it improves the soil's chemical and physical properties while promoting nutrient uptake by plants [15]. Its long-term presence in the soil significantly influences its effectiveness, as it is essential for enhancing the productivity of acidic soils.

Given the limited data on the effectiveness of various lime types and the frequent need to double application rates, this study aimed to assess the effectiveness of fine and coarse agricultural lime applications at rates of 3 t/ha and 6 t/ha in mitigating soil acidity. Over a two-year period, it focused on evaluating key parameters, including soil pH changes, cation exchange capacity (CEC), levels of exchangeable base cations (Ca^{2+}, Mg^{2+}, and K^+), and exchangeable aluminium. Furthermore, the study examined whether the fineness of lime or higher application rates had a more substantial impact on enhancing subsoil characteristics compared to lower rates.

2. Materials and Methods

2.1. Experimental site

The experimental field GPS coordinates are: 45°25'31.8"N 21°23'51.8"E, located in Barzava Plain, in the West part of Romania.

From a climatic point of view, the area where the experimental field was located is characterized by an average annual temperature of 10.5°C and an annual average of precipitation between 600-700 mm, distributed unevenly throughout the year. The number of days with snow on the ground is an average of 25 days, a fact determined by the geographic position.

The type of soil on which the research was carried out is a Gleyic Fluvisols with the main characteristics presented in Table 1.

Table 1. Main characteristics of Gleyic Fluvisol

Characteristic	
Texture	Loamy sandy
Structure	Granular
Total porosity	54% in the first horizon
Humus content	2.8%
pH	$5.03 \div 5.12$
CEC (cation exchange capacity)	$9.27 \div 9.42$ cmol/kg
Ca_{exch} (exchangeable Ca)	$4.63 \div 4.71$ cmol/kg
Mg_{exch} (exchangeable Mg)	$0.74 \div 0.85$ cmol/kg
K_{exch} (exchangeable K)	$0.140 \div 0.151$ cmol/kg
Al_{exch} (exchangeable Al)	$2.780 \div 3.300$ cmol/kg

The study took place between September 2022 and March 2024, utilizing randomized experimental plots measuring 50m x 30m under maize cultivation. The design included four amendment variants arranged in three replications. Agricultural limestone (aglime) with 98% $CaCO_3$ was applied in two distinct doses—3 t/ha and 6 t/ha—and two granulometries: coarse (1–2 mm) and fine (0.1 mm). pH, base cations and exchangeable aluminum were monitored at two soil depths, topsoil 0–20 cm (d1) and subsoil 20–40 cm (d2). Figure 1 provides a graphical representation of the experimental setup.
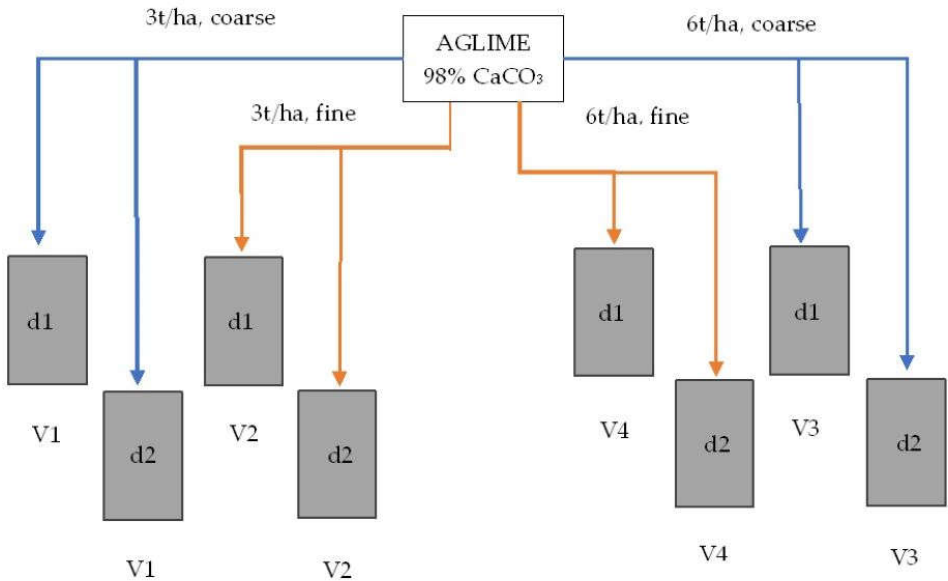

Figure 1. Field experiences description.

2.2. Soil Sampling

To determine the influence of aglime on pH, CEC, and the exchangeable forms of Ca, Mg, K, and Al, four soil sampling periods were established: T_0 – September 2022 (before aglime application), T_1 – March 2023 (6 months after application), T_2 – September 2023 (12 months after application), T_3 – March 2024 (18 months after application).

Soil samples were collected from each replication of each aglime variant at two depths during the four sampling periods, resulting in a total of 96 soil samples. After collection, soil samples from the two specified depths were placed in labelled polyethylene bags. Prior to laboratory analysis, the

samples were air-dried and passed through a 2 mm soil sieve to separate the fine soil fraction (<2 mm) from the coarse fraction [17, 18, 19].

2.2.1. pH Determination

Soil pH was measured according the FAO, [20, 21, 22], which recommends a 1:2.5 soil-to-water ratio. For the measurements a Mettler Toledo digital pH-meter was used, bearing a combine glass electrode, which had been previously calibrated with buffer solutions of pH=4, pH=7, pH=10, using reagents of analytical grade (Merck).

2.2.2. Exchangeable Ca (Ca_{exch}), Mg (Mg_{exch}), and K (K_{exch}) Determination

The exchangeable forms of Ca_{exch}, Mg_{exch}, and K_{exch} were determined from the 0.1M $BaCl_2 \cdot 2H_2O$ soil extract using atomic absorption spectrometry. A 2 g soil sample (<2 mm sieved) was subjected to repeated extraction with 20 ml of 0.1M $BaCl_2 \cdot 2H_2O$ to obtain a final volume of approximately 100 ml. Merck-grade reagents were used [23]. After filtration and adjusting the extract to a final volume of 100 ml, the concentrations of Ca_{exch}, Mg_{exch}, and K_{exch} were determined using a Varian 240 FS (USA) atomic absorption spectrophotometer. The working conditions were as follows: air:acetylene ratio = 13.50:2, Nebulizer uptake rate = 5 ml/min, Wavelengths: λ_{Ca} = 422.7 nm, λ_{Mg} = 282.5 nm, λ_K = 766.5 nm. For calibration were used standard solutions with the concentration ranging from 0.3 to 3 mg/l, prepared from multielement solution ICP Standard solution 1000 mg/L (Merck).

2.2.3. Exchangeable Al (Al_{exch}) Determination

Al_{exch} was determined using ICP-MS (Inductively Coupled Plasma Mass Spectrometry) on a PlasmaQuant Elite – Analytik Jena instrument with an AIM3300 autosampler (Germany), from a 1M KCl extract, using a soil-to-solution ratio of 1:10. The instrument's operating conditions were as follows: plasma flow: 9 L/min, auxiliary flow: 1.50 L/min, nebulizer flow: 1.05 L/min, sampling depth: 5 mm, RF power: 1.40 kW. For calibration, standards with concentrations ranging from 0.5 to 100 µg/L were prepared from a multi-element ICP standard solution (1000 mg/L, Merck). A 10 µg/L Sc internal standard (Analytik Jena) was used.

2.2.4 CEC Determination

CEC was calculated based on the exchangeable forms of the base cations: Ca, Mg, K, and Al, using the following equation [24]:

$$CEC \left(\frac{cmol}{kg\ soil}\right) = \left[\frac{Ca_{exch}}{20}\right] + \left[\frac{Mg_{exch}}{12}\right] + \left[\frac{K_{exch}}{39}\right] + \left[\frac{Al_{exch}}{9}\right] \quad (1)$$

2.3 Data Analysis

Statistical analysis was performed using Microsoft Excel, SPSS 12.0, and OriginPro 2024b. Statistical significance level was determined at 0.05 (p value). The results for pH, Ca_{exch}, Mg_{exch}, Al_{exch} and K_{exch} were expressed as mean ± standard deviation (SD) (n=3). Correlation analysis was carried out to determine the association between soil parameters at different time intervals after the application of the two aglime doses with two different granulometries. Analysis of variance (ANOVA) and mean separation (Tuckey test) were carried out to identify the statistical significance of the effect of aglime rate and granulation on some soil parameters. For dimensionality reduction, principal component analysis (PCA) with varimax rotation was performed to provide a concise and optimal description of the data.

3. Results

The values obtained for the investigated parameters, expressed as the mean of three distinct determinations ± SD, are presented in Tables 2 and 3. To determine whether there were significant differences between the experimental variants at the same time after lime application, the means were compared using ANOVA and the Tukey test at a statistical significance level of $\alpha = 0.05$.

Table 2. pH, CEC and Al_{exch} values after lime application.

	T0	T1	T2	T3
		V1, d_1=0-20 cm		
pH	5.08±0.10	5.62±0.06ab	6.47±0.05a	6.39±0.04b
CEC (cmol/kg)	9.4±0.18	9.71±0.05c	10.21±0.10d	10.88±0.14b
Al_{exch}(cmol/kg)	3.10±0.310	1.98±0.050	0.43±0.080b	0.08±0.060b
		V1, d_2=20-40 cm		
pH	5.15±0.04	5.39±0.02cde	5.43±0.08d	5.64±0.05d
CEC (cmol/kg)	9.31±0.12	9.62±0.04c	10.12±0.24d	10.05±0.16c
Al_{exch}(cmol/kg)	2.79±0.470	2.43±0.460	1.98±0.250a	1.71±0.530a
		V2, d_1=0-20 cm		
pH	5.12±0.02	5.80±0.06a	6.55±0.03a	6.33±0.05b
CEC (cmol/kg)	9.27±0.03	9.92±0.08c	10.79±0.27c	10.37±0.08c
Al_{exch} (cmol/kg)	2.78±0.250	1.74±0.290	0.31±0.030b	0.05±0.010b
		V2, d_2=20-40 cm		
pH	5.24±0.05	5.6±0.12abc	5.72±0.04c	5.85±0.06c
CEC (cmol/kg)	9.24±0.16	9.93±0.03c	11.1±0.15abc	10.19±0.19c
Al_{exch} (cmol/kg)	2.590±0.140	2.29±0.440	1.84±0.230a	1.61±0.260a
		V3, d_1=0-20 cm		
pH	5.03±0.05	5.49±0.04abc	6.28±0.04b	6.6±0.05a
CEC (cmol/kg)	9.42±0.11	10.61±0.06a	11.04±0.15a	12.43±0.17a
Al_{exch}(cmol/kg)	3.300±0.590	2.24±0.540	0.44±0.210b	-
		V3, d_2=20-40 cm		
pH	5.13±0.03	5.21±0.1cde	5.39±0.06d	5.36±0.05a
CEC (cmol/kg)	9.3±0.25	10.47±0.24a	10.75±0.08abc	12.21±0.19a
Al_{exch}(cmol/kg)	2.330±0.540	2.23±0.190	1.94±0.300a	1.7±0.400a
		V4 d_1=0-20 cm		
pH	5.06±0.12	5.62±0.08ab	6.57±0.03a	6.63±0.08a
CEC (cmol/kg)	9.41±0.19	11.22±0.20b	11.55±0.13bc	12.09±0.19a
Al_{exch} (cmol/kg)	3.290±0.360	2.19±0.480	0.35±0.130b	-
		V4, d_2=20-40 cm		
pH	5.17±0.03	5.31±0.08de	5.45±0.07d	5.6±0.14d
CEC (cmol/kg)	9.26±0.06	11.05±0.16b	11.44±0.17	12.13±0.15a
Al_{exch} (cmol/kg)	2.790±0.630	2.61±0.370	2.1±0.210a	1.86±0.460a

Means (calculated at the same time) that do not share a letter are significantly different at the 0.05 level.

Table 3. Exchangeable Ca, Mg and K values after lime application

	T0	T1	T2	T3
		V1, d_1=0-20 cm		
Ca_{exch} (cmol/kg)	4.70±0.32	5.82±0.79ab	7.25±0.56ab	8.10±0.30ab
Mg_{exch} (cmol/kg)	0.85±0.085	0.97±0.174	1.03±0.069	1.12±0.188
K_{exch} (cmol/kg)	0.151±0.022	0.212±0.017	0.264±0.038	0.286±0.082abc
		V1, d_2=20-40 cm		
Ca_{exch} (cmol/kg)	4.65±0.39	5.69±0.44ab	6.18±0.71b	7.03±0.30bc
Mg_{exch} (cmol/kg)	0.740±0.123	0.77±0.147	0.82±0.182	0.91±0.058

K_{exch} (cmol/kg)	0.122±0.013	0.159±0.024	0.191±0.017	0.209±0.023c
		V2, d_1=0-20 cm		
Ca_{exch} (cmol/kg)	4.63±0.50	6.45±0.56ab	7.66±0.54ab	7.46±0.47bc
Mg_{exch} (cmol/kg)	0.740±0.028	0.82±0.060	1.04±0.06	1.06±0.064
K_{exch} (cmol/kg)	0.140±0.035	0.225±0.05	0.267±0.027	0.297±0.029abc
		V2, d_2=20-40 cm		
Ca_{exch} (cmol/kg)	4.62±0.39	5.45±0.46b	6.54±0.28b	6.82±0.20c
Mg_{exch} (cmol/kg)	0.830±0.046	0.89±0.102	0.93±0.115	1.03±0.047
K_{exch} (cmol/kg)	0.110±0.019	0.172±0.021	0.213±0.014	0.215±0.016bc
		V3, d_1=0-20 cm		
Ca_{exch} (cmol/kg)	4.71±0.31	6.47±0.56ab	7.50±0.49ab	8.20±0.75ab
Mg_{exch} (cmol/kg)	0.750±0.051	0.85±0.016	1.1±0.101	1.15±0.068
K_{exch} (cmol/kg)	0.150±0.023	0.231±0.042	0.283±0.054	0.334±0.044ab
		V3, d_2=20-40 cm		
Ca_{exch} (cmol/kg)	4.65±0.37	5.28±0.51b	6.20±0.80b	7.65±0.43bc
Mg_{exch} (cmol/kg)	0.840±0.062	0.92±0.198	0.99±0.091	1.02±0.127
K_{exch} (cmol/kg)	0.129±0.024	0.18±0.095	0.22±0.065	0.254±0.04abc
		V4 d_1=0-20 cm		
Ca_{exch} (cmol/kg)	4.70±0.30	6.95±0.26a	8.20±0.38a	9.06±0.26a
Mg_{exch} (cmol/kg)	0.75±0.073	0.85±0.051	1.03±0.126	1.1±0.202
K_{exch} (cmol/kg)	0.142±0.013	0.246±0.048	0.289±0.063	0.36±0.041a
		V4, d_2=20-40 cm		
Ca_{exch} (cmol/kg)	4.65±0.37	5.96±0.31ab	7.00±0.52b	7.85±0.43bc
Mg_{exch} (cmol/kg)	0.840±0.113	0.9±0.150	0.88±0.122	1.01±0.016
K_{exch} (cmol/kg)	0.120±0.011	0.21±0.069	0.243±0.046	0.274±0.028abc

Means (calculated at the same time) that do not share a letter are significantly different at the 0.05 level.

3.1. Modification in Soil pH Values

In variant V1 (Table 2), soil pH increased from 5.08 to 6.47 in the topsoil (d_1) and from 5.15 to 5.85 in the subsoil (d_2). The highest pH value for V1 was recorded at T2 in the topsoil, while in the subsoil it peaked at T3. In variant V2, the maximum pH values, 6.55 for d_1 and 5.85 for d_2, were both reached at T2. For V3, the highest pH in the topsoil (6.60) was observed at T3, whereas the subsoil's maximum pH (5.70) occurred at T2.

At T2, statistical analysis (α = 0.05) showed significant differences in the highest pH values between V2 (d_1) and V3 (d_2), as well as between V1 (d_1) and V2 (d_2). Similarly, at T3, significant differences were noted between V4 (d_2) and V3 (d_1), as well as within V4 (d_1 and d_2). In variant V4, pH values rose from 5.06 to 6.60 in the topsoil and from 5.17 to 5.60 in the subsoil, with the highest pH levels for both depths recorded at T3.

During T1, V2 exhibited the most significant pH increase, rising by 12% in the topsoil and 6% in the subsoil (Table S1). At T2, V4 showed the largest pH increase in the topsoil, with a 23% rise compared to T0, while in the subsoil, V2 recorded the greatest increase, an 8% rise relative to T0. Fine lime applied to the topsoil (d_1) resulted in the highest pH increase at T2, regardless of the quantity applied, whereas coarse lime was most effective at T3. In the subsoil (d_2), the greatest pH increase was observed at T3 following the application of 3 t/ha fine lime (V2).

3.2. CEC Values After Lime Application

The cation exchange capacity (CEC) rises as pH values increase, because of the deprotonation of pH-dependent charges. These charges originate from either the carboxylic or phenolic groups in organic matter, or from aluminium or iron hydroxides. The greatest increases in CEC values were observed with the application of 6 tons per hectare of fine lime (V4), regardless of the time elapsed since the amendment was applied (Table 2). In this variant, the increases compared to T0 are 16% at T1, 18% at T2, and 24% at T3. When the same amount of amendment is applied for the same duration,

fine lime results in greater increases in CEC compared to coarse lime. The highest CEC value recorded was 12.43 cmol/kg with the application of 6 tons per hectare of fine lime at T3, at d_1. At depth d_2, the maximum CEC value was 12.21 cmol/kg in V3 at T3. Between the two mentioned values, there are no significant differences at a statistical significance level of $\alpha = 0.05$. However, the increase in CEC values compared to T0 is roughly the same for both V3 and V4, at 23.9% and 23.3%, respectively (Table S1).

3.3. Modification of Exchangeable Ca, Mg and K Values

The studied soil exhibits a basification of the colloidal complex due to both pH-dependent and permanent charges of mineral colloids, resulting in aluminium ions (Al^{3+}, $Al(OH)^{2+}$, $Al(OH)_2^+$) replacing basic cations in the cation exchange capacity. Consequently, the Ca_{exch} content is reduced, ranging from 4.62 to 4.71 cmol/kg at T0 (Table 3). The application of aglime increased this content, with a maximum value of 9.06 cmol/kg observed in V4 T3 at d_1, which is 48% higher than in V3 at T0. Over time, the calcium content retained by the colloidal complex increases following aglime application. This gradual effect is due to the slow reactivity of lime in releasing Ca^{2+} ions, resulting in a longer-lasting impact. Similar values were found in V1 at T3 and V3 at T3, with 8.1 cmol/kg (41.9% higher than T0) and 8.2 cmol/kg (42.5% higher than T0) (Table S1), respectively. Statistically, there are no significant differences between these maximum values at a significance level of $\alpha = 0.05$. Ca_{exch} at depth d_2 are significantly lower compared to d_1, with the highest value recorded in the V3 at T3 variant at 7.85 cmol/kg.

The application of lime resulted in an increase in exchangeable magnesium (Mg_{exch}) content, which rose from 0.74 cmol/kg to 1.15 cmol/kg at d_1 in variant V3 at T3 (Table 3). Lower increases in exchangeable Mg were observed at d_2, with the highest value being 1.03 cmol/kg in variant V2 at T3. Regardless of the lime amount, the content of exchangeable Mg increases with time after its application. At d_1, the most significant increases in Mg_{exch} compared to T0 were at doses of 6 t/ha, with coarse lime showing an increase of 34.7% and fine lime an increase of 31.8% (Table S1). At d_2, the largest increases compared to T0 were at doses of 3 t/ha, with coarse lime showing an increase of 18.6% and fine lime 19.4%. The mean values determined for Mg_{exch}, regardless of the experimental variant and depth, do not differ significantly at a statistical level of assurance $\alpha=0.05$, at any of the time intervals: T1(P=0.666), T2(P=0.116), T3 (P=0.318).

The treatments with 3 t/ha F (fine lime) and 6 t/ha F show a rapid increase in the Ca/Mg ratio, reaching a peak around T1/T2 before slightly declining by T3 (Figure 2). This indicates that fine lime dissolves quickly, releasing calcium (Ca) into the soil at an accelerated rate and temporarily boosting the Ca/Mg ratio. The 6 t/ha F treatment exhibits a more pronounced effect than 3 t/ha F, achieving the highest peak. In contrast, the 3 t/ha C and 6 t/ha C treatments display a steadier rise in the Ca/Mg ratio without a sharp peak. The 6 t/ha C treatment has the lowest R^2 value (0.4425), reflecting higher variability, likely due to its slower soil breakdown. The 3 t/ha C treatment follows a consistent linear trend ($R^2 = 0.952$), resulting in a gradual, predictable increase in Ca/Mg over time.
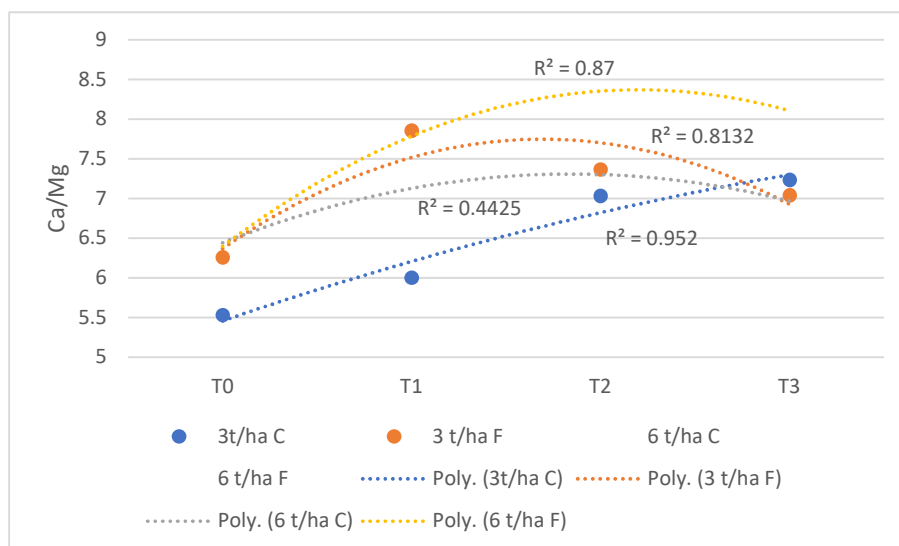
Figure 2. Influence of lime application on Ca/Mg ratio.

Exchangeable potassium (K_{exch}) content also increased with depth, lime additions, and time since application. The values ranged from 0.110 cmol /kg to 0.360 cmol /kg (Table 3). Although lower compared to Mg_{exch} values, the increases relative to T0 ranged from 23.2% in variant V5T1 to 60.5% in variant V4T3. Like Ca_{exch} and Mg_{exch}, K_{exch} content at a d_1 was higher than at d_2, regardless of the fertilization variant. The application of fine lime, at both doses and depths, resulted in higher increases in K_{exch} content, particularly at T1 and T2. At T3, the highest content at both depths was achieved with the application of 6 t/ha fine lime (an increase of 60.5% at T1 and 56.2% at T2) and coarse lime (an increase of 55.1% at T1 and 49.2% at T2) (Table S1). It was observed that there were no significant differences between the mean values determined in the 4 experimental variants, at the 2 depths (d_1 and d_2) at α=0.05, both at the time interval T1 (P=0.434) and T2 (P=0.137).

3.4. Exchangeable Aluminium

Al_{exch} is present in significant quantities in acidic soils with pH levels below 5.5. Analysis of the samples indicates that (Table 2) the Al_{exch} content decreases as soil acidity diminishes, with values ranging from 3.3 cmol/ kg soil in V3T0 (d_1) to 0 cmol/ kg soil in V3T3 (d_1) and V4T3 (d_1). At the highest amount of aglime applied, Al_{exch} content declines further over time following amendment application. At T1, Al_{exch} content decreases in d_1 by 32.12% in V3 and 37.14% in V2, and in d_2 between 4.29% in V3 and 12.90% in V1. It was observed that there were no significant differences between the mean values determined in the 4 experimental variants, at the 2 depths at α=0.05, at the time interval T1 (P=0.2665). By T2, regardless of depth, the reduction in Al^{3+} content in the colloidal complex doubles, reaching 89.3% in V4 at d_1 and 29.03% in V3 at d_2 (Table S1). Although the findings reveal that fine lime is more effective than coarse lime in reducing Al_{exch} content, in subsoil this phenomenon is reduced.

3.5 Pearson Correlations

Examining the Pearson correlation matrix across three-time intervals (T1, T2, and T3) within the four experimental variants (V1–V4) and two soil depths (0–20 cm and 20–40 cm), several trends are observed.

At **T1**, there are strong positive correlations between CEC and Ca_{exch} (R=0.87), and between Mg_{exch} and K_{exch} (R=0.89) (Figure 3). A negative correlation exists between pH and Al_{exch} (R=-0.75). Lime application typically increases soil pH, reducing acidity, which aligns with the observed negative correlation between pH and exchangeable aluminium.
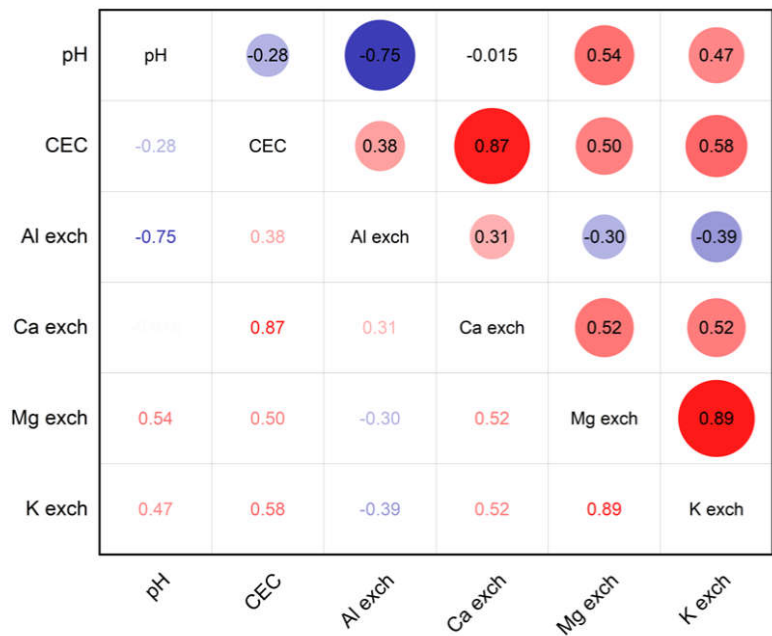
Figure 3. Pearson correlation matrix at T1.

T2 reveals strong positive correlations between pH and Ca_{exch} (R=0.87), pH and Mg_{exch} (R=0.78), and pH and K_{exch} (R=0.85) (Figure 4). Additional positive correlations are noted between Ca_{exch} and K_{exch} (R=0.94), Mg_{exch} and K_{exch} (R=0.83), and Ca_{exch} and Mg_{exch} (R=0.67). Strong negative correlations are present between Al_{exch} and pH (R=-0.98), Al_{exch} and Ca_{exch} (R=-0.86), Al_{exch} and Mg_{exch} (R=-0.84), and Al_{exch} and K_{exch} (R=-0.87). These relationships highlight lime's role in increasing soil pH, reducing aluminium toxicity, and enhancing nutrient availability.
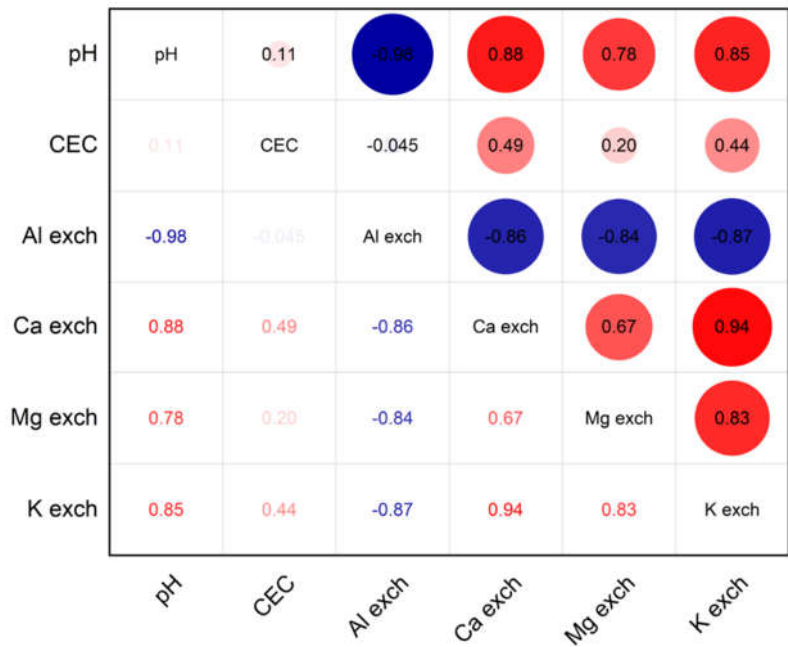

Figure 4. Pearson correlation matrix at T2.

At T3, strong positive correlations between pH and Ca_{exch} (R=0.63), pH and Mg_{exch} (R=0.80), and pH and K_{exch} (R=0.79) persist, alongside correlations between Ca_{exch} and K_{exch} (R=0.91) and Mg_{exch} and K_{exch} (R=0.79) (Figure 5). Positive correlations between CEC and Ca exch (R=0.77), CEC and K_{exch} (R=0.66), and CEC and Mg_{exch} (R=0.43) are intensified. Strong negative correlations between Al_{exch} and pH (R=-0.95), Al_{exch} and Ca_{exch} (R=-0.63), Al_{exch} and Mg_{exch} (R=-0.82), and Al_{exch} and K_{exch} (R=-0.87) are maintained.
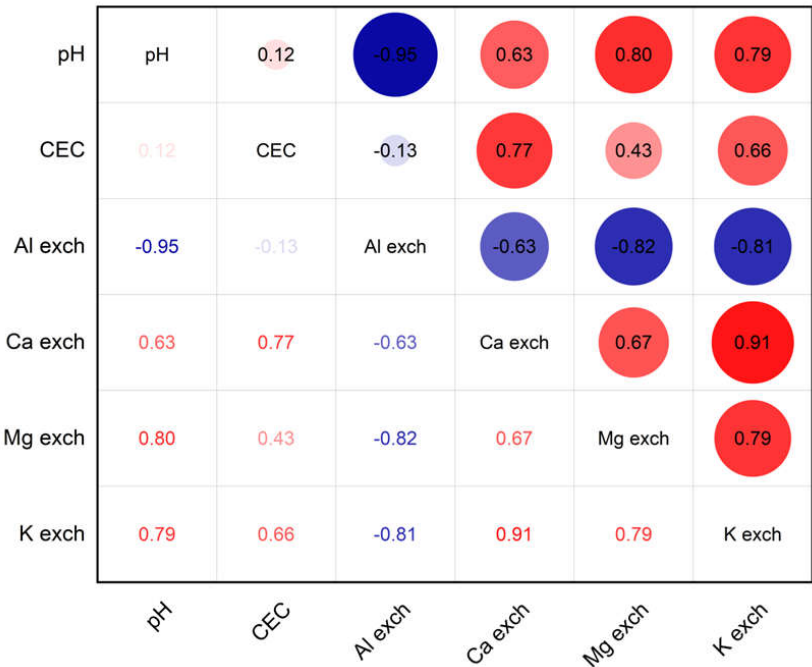

Figure 5. Pearson correlation matrix at T3.

Our findings underscore aglime's effectiveness in enhancing soil health by neutralizing acidity, reducing exchangeable aluminium, and improving nutrient availability (calcium, magnesium, and potassium). Sixteen months after application, the lasting improvements emphasize the long-term benefits of lime amendments for soil fertility and plant growth.

3.6 Principal Component Analysis

Principal component analysis (PCA), Figure 6 – applied to data obtained at intervals T1, T2 and T3, at depth d_1 extracted 2 components with Eigenvalue>1 that provide in total 94.21% of the total variation. PC1 contributing with 81.65% of the total variance is strongly correlated with pH and the exchangeable content of Ca, Mg and K. This indicates that both pH and Ca_{exch}, Mg_{exch} and K_{exch} are strongly and directly influenced by the addition of aglime in the soil. PC2 accounts for the next 20.58% and its correlated with CEC and Al_{exch}. Analysing Figure 6 it is observed that aglime, at a dose of 6t/ha (V3 and V4), regardless of granulation, at interval T3 determines the highest values for Ca_{exch}, respectively K_{exch}. The Al_{exch} content, on the other hand, is strongly influenced by the same V3 and V4 variants, at the T1 time interval.
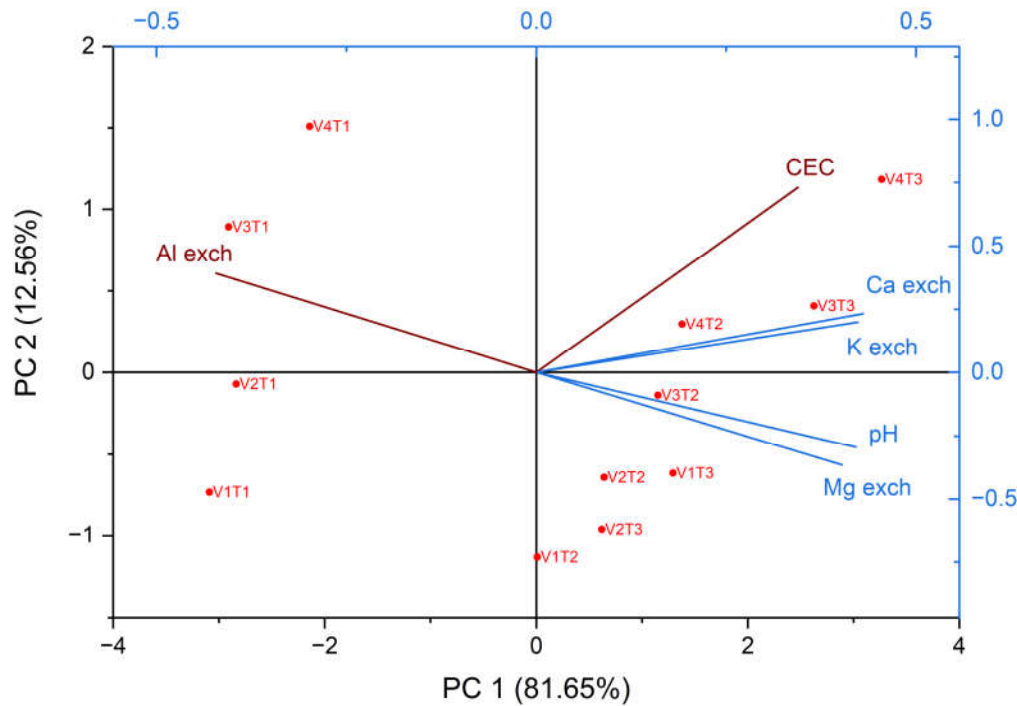
Figure 6. The influence of aglime dose on soil parameters.

4. Discussion

The effectiveness of calcium carbonate in raising pH levels depends on factors such as soil texture, organic matter, and the size of lime particles [25]. While the greatest pH increases in T1 occurred with 3 t/ha of fine lime, the most notable differences compared to T0 at T2 and T3 were observed with 6 t/ha of fine lime. Enesi et al. [26] demonstrated that higher lime application rates yielded greater pH increases and improved yields, while Oliver et al. [27] emphasized liming rate as the most influential factor for pH and yield changes in acidic soils. The soil's buffering capacity, or its resistance to pH alterations, dictates the lime quantity required for significant changes. Although higher lime amounts can more effectively overcome buffering capacity, the resulting pH increase can vary with management practices [28]. Greater lime application neutralizes more acidity, producing a higher final pH. Our findings align with [29], who found that changes in soil pH depended on lime rates, time post-application, and soil depth.

Finer lime particles have a greater surface area in contact with the soil, enabling a faster and more efficient reaction with soil acids, which accelerates the pH increase. Their smaller size allows them to dissolve more quickly, ensuring a prompt adjustment in soil pH. In contrast, coarser particles dissolve at a slower rate, leading to a more gradual pH change. Additionally, finer particles distribute more evenly throughout the soil, promoting a consistent pH adjustment, while coarser particles may lead to uneven changes. Overall, finer lime particles contribute to a quicker and more uniform rise in soil pH, whereas coarser particles offer a slower yet sustained effect. This conclusion aligns with findings from [30], which highlighted that powdered lime was the most effective, achieving the highest pH increase in both severely acidic (+19%) and moderately acidic (+14%) environments. In a study examining lime particle sizes ranging from 3 mm to 0.005 mm, Scott et al. [31] observed that finer particles consistently caused larger pH increases, with no minimum particle size for maximum efficacy identified.

Improving subsoil acidity is notably challenging, as liming often proves ineffective due to the slow downward movement of lime within soil profiles. Our study revealed only a modest increase

in soil pH at a depth of 20-40 cm following liming. Lime predominantly impacts the topsoil, moving through the profile at a very gradual pace. Over time, it can incrementally enhance subsoil pH by neutralizing acidity, provided it is applied in adequate amounts and properly incorporated. Du Toit et al. [32] demonstrated that liming raised soil pH above the target level of 5.5 at depths of 5–10 cm (pH 6.83) and 10–15 cm (pH 5.73), but showed no significant effect at depths of 15–40 cm. Research made in New Zeeland showed that lime application at 8 t/ha increased soil pH by about 0.5 pH units in the 7.5-15 and 15-30 cm soil horizons [33].

The mineralogical study conducted by [34], utilizing X-ray diffraction of the soil, revealed that the upper horizons contain illitic (30%), chloritic (50%), and kaolinitic minerals (<15%). Due to the soil's acidic nature, the cation exchange capacity (CEC) is low because of the presence of H^+ and Al^{3+} ions, which occupy exchange sites and reduce the overall CEC. At low pH values (pH < 5.5), only the permanent charges of 2:1 type clay and a small portion of the pH-dependent charges of organic colloids and 1:1 type clay holds exchangeable ions.

After lime application, as the pH increases, the negative charges of 1:1 type clay (such as kaolinite), humus, and even Fe and Al oxides increase, thereby enhancing the cation exchange capacity [35]. The effect of calcium carbonate application in this study on cation exchange capacity aligns with previously reported results [36]. These effects mainly involve a decrease in the Al level in the soil and, consequently, a decrease in the effective saturation of Al in the exchange complex, as well as an increase in the proportion of Ca in the exchange complex for both studied soils [37-39]. Fine lime particles react more quickly, increasing soil pH relatively fast. As the pH increases, the negative charge on clay minerals and organic matter may slightly increase, enhancing the soil's CEC. Coarse lime particles dissolve more slowly, having minimal immediate effect on pH and CEC. Lime application promotes microbial activity and organic matter breakdown, leading to the formation of new functional groups (such as carboxyl and phenol groups), which can contribute additional cation exchange sites, further increasing CEC.

Applying lime to the soil generally increases the calcium content in the topsoil, and this increase is directly proportional to the amount of lime applied. Similar results were observed in [40], which showed that the highest exchangeable levels of Ca, Mg, and K were recorded in plots that received the highest dose of lime. Lime primarily affects the soil layers where it is incorporated [41]. In our study, lime was applied and mixed into the topsoil, resulting in the most significant immediate increase in calcium content there. Over time, some calcium from the lime leached into the subsoil, particularly with substantial autumn rainfall. Fine lime particles provided relatively faster calcium release and movement even in the subsoil, as their smaller size allows easier transport by water moving through the soil profile, enabling deeper layer penetration. Coarser lime particles dissolve and release calcium more slowly. However, due to their limited movement through the soil, calcium release is more gradual and sustained over a longer period. Because of the sandy loam texture of the studied soil, calcium may leach faster compared to clay-rich soils, leading to an increase in calcium content in the subsoil.

Aluminium toxicity poses a significant challenge in acidic soils, as pH levels below 5.5 cause aluminium to be solubilized into toxic ionic forms like Al^{3+}, which hinder plant root growth and nutrient absorption [42, 43]. At pH levels between 5.0 and 5.25, $Al(OH)_2^+$ species dominate, while between 5.25 and 6.50, $Al(OH)_2^+$ becomes predominant. According to Sparks et al. [4], 6–28% of the total aluminium in soil solution occurs as free Al^{3+}. Post-liming, as pH rises, the concentration of monomeric Al^{3+} ions decrease, giving way to Al-hydroxy species. In the topsoil layer (0-20 cm), as pH exceeds 5.5 beginning at T2, Al-hydroxy ions likely polymerize, forming large, positively charged structures tightly bound to the negatively charged colloidal sites, rendering them non-exchangeable. By T3, the increasing concentration of Ca^{2+} ions raise the pH to values between 6.3 and 6.63. Calcium ions from lime exhibit a higher affinity for exchange sites on soil colloids compared to Al^{3+}, displacing Al ions, which subsequently precipitate as $Al(OH)_3$. This displacement frees the negative colloidal sites for further cation exchange.

A study by Moir and Moot [33] demonstrated that liming reduced Al exch levels in the surface soil horizon (0-7 cm) from 0.9 to 0.1–0.2 meq/100 g. However, the effects were less pronounced in deeper horizons, showing only modest reductions in Al exch. This increase in pH resulted in a decline of exchangeable aluminium, from around 1.0 meq/100 g at an 8 t/ha lime rate to approximately 0.45 and 1.1 meq Al/100 g in the 7.5–15 cm and 15–30 cm soil horizons, respectively. Literature indicates that aluminium toxicity remains a concern in soils with pH below 5.5, while Whitley et al. [44] reported that soils with pH (H_2O) up to 5.9 may still exhibit aluminium concentrations toxic to plants.

By analysing PCA we can see that V3 and V4 generally show stronger effects on improving soil properties like pH, calcium (Ca_{exch}), magnesium (Mg_{exch}), and potassium (K_{exch}), as indicated by their alignment with vectors for these parameters in the PCA biplot. The higher lime dose likely enhances nutrient availability and neutralizes acidity more effectively. V1 (3 t/ha coarse lime) and V2 (3 t/ha fine lime) show a milder impact, with less pronounced changes in soil properties. However, fine lime (V2) may exhibit quicker reactivity compared to coarse lime (V1), due to its finer granulometry. At d_1 (0–20 cm), lime application tends to have a greater influence, as reflected by stronger positive correlations with properties like pH, Ca_{exch}, and CEC. This aligns with the common observation that lime primarily affects the topsoil layer. At d_2 (20–40 cm), the effects are less pronounced but still visible, suggesting residual or gradual lime influence over time. The initial changes 6 months after lime application (T1) are evident but less stable, as lime begins to dissolve and interact with the soil. Twelve months after application (T2) the strongest improvements are observed, with significant increases in pH and nutrient availability (Ca exch, Mg exch, K exch) and a marked reduction in exchangeable aluminium (Al exch). Eighteen months after lime application (T3) effects remain, but there is slight attenuation compared to T2, reflecting potential leaching or stabilization over time.

5. Conclusions

The capacity of calcium carbonate to correct soil acidity is determined by several factors, including soil texture, organic matter content, lime particle size, and application rate. Fine lime particles demonstrated superior efficiency over coarser particles, leading to quicker and more uniform pH increases due to their greater surface area and faster solubility. The most pronounced pH elevation occurred with applications of highest dose of fine lime, particularly over extended timeframes, corroborating previous findings that associate higher lime rates with improved pH and crop productivity.

Lime application also enhanced cation exchange capacity (CEC), largely through reductions in exchangeable aluminium (Al^{3+}) and increases in calcium (Ca^{2+}) availability, especially in the topsoil. Although lime's downward movement within the soil profile is generally slow, fine particles contributed to slight improvements in subsoil pH, particularly in sandy loam soils where leaching is more evident. As soil pH increased following liming, so did the negative charge of soil colloids, boosting CEC—further supported by enhanced microbial activity and organic matter decomposition.

Incorporation of lime primarily raises calcium concentrations in the surface layers, with the magnitude of increase proportional to the applied dose. Fine lime particles facilitated faster calcium release and deeper mobility, while coarser particles provided a slower, more prolonged supply. Over time, leaching—especially in sandy loam soils—allowed some calcium to reach lower soil horizons.

Additionally, liming plays a crucial role in alleviating aluminium toxicity by elevating pH levels. This transformation reduces harmful Al^{3+} ions into less toxic Al-hydroxy species. As pH rises, aluminium is displaced from exchange sites and precipitated as $Al(OH)_3$, enabling calcium to occupy those sites. While these effects are most pronounced in the topsoil, aluminium toxicity may persist at depth or in soils with pH below 5.5—and, in some cases, even up to pH 5.9.

Soil parameters such as pH, CEC, and exchangeable cations (Ca^{2+}, Mg^{2+}, K^+) showed strong positive correlations with lime application, especially at higher rates. Conversely, exchangeable aluminium (Al^{3+}) displayed a negative correlation with both pH and nutrient cations, reflecting lime's efficacy in neutralizing soil acidity and reducing aluminium toxicity. Principal component analysis further highlighted the effectiveness of higher lime doses in improving soil health. Fine lime

produced rapid responses, while coarse lime offered a slower but sustained impact. Notable improvements in the topsoil and consistent patterns across later sampling periods underscore lime's role in enhancing nutrient availability and ameliorating acid soil conditions over time.

Supplementary Materials: The following supporting information can be downloaded at: www.mdpi.com/xxx/s1, Table S1: Percentage variation of monitored parameters.

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Data Availability Statement: The original contributions presented in the study are included in the article. Further inquires can be directed to the corresponding author/s.

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Abbreviations

The following abbreviations are used in this manuscript:

Aglime   Agricultural lime (CaCO$_3$)
Kg a.s./ha   Kg active substance/ha

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