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Limestone Dissolution Under Varying Rates and Soil Textures

Tiago Barreto Garcez[a], Denison Pogorzelski[b], Wedisson Oliveira Santos[c],
Patrícia Cardoso Matias[b], Fabiane Ballotin [b], and Reinaldo Bertola Cantarutti[b]

[a]Soil Department, Universidade Federal de Sergipe, Vicosa, Brazil; [b]Departamento de Solos, Universidade Federal de Viçosa, Viçosa, Brazil; [c]Laboratory of Soil and Plant Analysis, Institute of Agricultural Sciences, Universidade Federal de Uberlândia, Vicosa, Brazil

ABSTRACT

The agricultural frontier in Brazil advances mainly in areas of sandy soils, where the management of soil fertility is still a challenge, including the practice of liming. Limestone dissolution was investigated under varying doses (0–40 t ha^{-1}) in soil samples with a wide range of textural gradients (5%–74% clay). In view of textural variations and limestone rates, the objective was to generate information regarding pH, the residual liming effect, and exchangeable calcium (Ca) and magnesium (Mg) levels, including under overliming conditions. Throughout the textural gradient, the pH was between 4.2 and 8.5. On more sandy soils (5%–18% clay), overheating (pH > 7) was obtained with ~ 2 t ha^{-1} of limestone. On clayey soils (37%–74% clay), a similar effect was obtained with ~ 10 t ha^{-1} of limestone, indicating the marked difference in the acid buffering capacity of these soils. In addition, the equilibrium pH in the context of overliming in sandy soil was higher (8.2) compared with clayey soils (7.8). The residual liming effect was greater in sandy soils due to the lower potential acidity. Regardless of the soil texture, the Ca/Mg ratio increased in the context of overliming. Liming must be carefully applied and requires proper recommendations, especially on sandy soils, avoiding pH ≥ 6.5, a condition that is not suitable for most crops.

Introduction

Soil acidity is one of the most limiting factor that affects crop production worldwide, mainly in highly weathered tropical soils (Gurmessa 2020). Calcium (Ca) and magnesium (Mg) deficiencies plus aluminum (Al) toxicity are the most prominent chemical constraints in acidic soils. Liming is a key practice to overcome these chemical limitations and, consequently, to promote compensatory crop productions in these conditions.

Most limestone is composed mainly by calcite ($CaCO_3$), dolomite ($CaMg(CO_3)_2$), and magnesite ($MgCO_3$). During the dissolution of limestone applied to soils, Ca^{2+} and Mg^{2+} ions released into the soil solution displace adsorbed cations on mineral or organic sites. The release of acidic cations into the soil solution (mainly Al^{3+}, hydronium [H_3O^+], iron [$Fe^{2+/3+}$], and manganese [Mn^{2+}]), followed by a decrease in the soil pH, results in changes in the chemical forms of these cations, including ionic and precipitate species (Lindsay 1979). The buffering capacity of a soil includes hydrolysis of these acid cations; protonation/deprotonation of functional groups of organic matter (OM), soil minerals, and kaolinite and Fe-Al hydro(oxides); dissolution and precipitation of carbonates; and the complexation/decomplexation of Al with OM (Xu et al. 2012).

CONTACT Fabiane Ballotin ✉ fabiane.ballotin@ufv.br 🖃 Departamento de Solos, Universidade Federal de Viçosa, Avenida P H Rolfs, Viçosa, Brazil

There are many methods to calculate the lime requirement for acidic soils. The most common methods are based on approaches that estimate the limestone rate as a function of soil properties, including the clay content, the remaining phosphorus content (P_{rem}), the cation exchange capacity (CEC), potential acidity, exchangeable acidity, and buffer pH (Teixeira, Alvarez, and Neves 2020). In fact, according to these approaches, when acidic soils present different buffering capacity, the lime requirement should follow the same trend. However, extensive areas of sandy soils have been incorporated into agricultural production in Brazil, and limestone rates considered to be usual for clayey soils have also been used in these new areas; this approach may result in overliming. The effects of overliming of these soil properties and lime dissolution is still not well known. Intriguingly, more than 200 areas of sandy soils (97–101 g kg^{-1} clay) subjected to overliming (5.4–11.9 t ha^{-1}, relative total neutralizing power [RTNP] = 75%) did not present pH in water > 7.0. In this context, the study goal was to investigate the soil properties and lime dissolution under different rates and soils texture gradients.

Materials and methods

Experimental setup

Neossolo Quartzarênico (Entisol), from Barreiras, Bahia state, and Latossolo Vermelho Amarelo (Oxisol), from Viçosa, Minas Gerais state (Table 1), were used because they have contrasting textures (5% and 74% clay from the top layer [0–20 cm], respectively), but similar mineralogy. In fact, the mineral composition of the soils associated with the clay fraction was based on kaolinite (Kt), goethite (Gb) and gibbsite (Gt). The primary mineral quartz (Qz), as expected, gained prominence in the entisol (Figure 1) due to the abundance of the sand fraction in this soil (Figure 1).

They were mixed in different weight proportions to obtain a textural gradient. After mixing, the soil samples showed a clay content of 5%, 9%, 18%, 37%, 55%, and 74%. To facilitate communication, the group of soils with 37%, 55%, and 74% clay was called as "clayey soils" and the group with 5%, 9%, and 18% clay as "sandy soils."

The limestone dissolution trial was performed in a completely randomized block design, using a 6 × 8 factorial scheme with four replications: six clay contents (5%, 9%, 18%, 37%, 55%, and 74%) and eight limestone rates (0, 1.5, 3, 6, 10, 20, 30, and 40 t ha^{-1}). Magnesium limestone was used with the following characteristics: 381 g kg^{-1} Ca (53.4% CaO), 45 g kg^{-1} Mg (7.5% MgO), and 104.2% neutralizing power and 99.5% reactivity, resulting in 103.6% RTNP. The dissolution trial was carried out in plastic bags (1 L) containing 250 g of soil. The soil samples were moistened with deionized water until reaching about 80%–90% of the soil's maximum water retention capacity, which was previously determined and was kept at rest for 15 days. During this period, the humidity was kept constant and, at the end, the soil samples were dried and passed through a 2 mm sieve.

The limestone was added according to the treatments, again keeping the humidity constant (80%–90% of the soil's maximum water retention capacity). The soil samples were homogenized and kept at rest for 90 days at 25 ± 2°C, which is the time required for the complete dissolution of the finest limestone fractions (which characterizes its relative efficiency). The plastic bags were kept partially closed to avoid water loss by evaporation without limiting gas exchange. During this period, the

Table 1. Chemical and physical characteristics of neossolo Quartzarênico_NQ (Entisol_en), from Barreiras, Bahia state, and Latossolo Vermelho Amarelo_LVA (Oxisol_ox), from Viçosa, Minas Gerais state.

Soil	pH	P	K	Ca	Mg	Al	H+Al	EB	T	P_{rem}	OM	Clay	Silt	Sand
		mg dm^{-3}		————cmol$_c$ dm^{-3}————						mg L^{-1}	————mg L^{-1} ————			
LVA	4.8	0.8	20	0.08	0.00	0.2	6.9	0.13	7.03	7.0	29.6	740	40	220
NQ	5.2	2.2	13	0.15	0.01	0.1	1.3	0.19	1.49	44.8	2.60	50	20	930

pH in water (1:2.5, v:v); P and K, Mehlich-1; P_{rem}, remaining phosphorus (Alvarez et al. 2000); Ca^{2+}, Mg^{2+}, and Al^{3+}, 1 mol L^{-1} KCl; H +Al, 0.5 mol L^{-1} Ca(OAc) at pH 7.0; OM, organic matter, modified Walkley – Black method (Nelson and Sommers, 1982); EB, exchangeable bases; T, cation exchange capacity at pH 7.
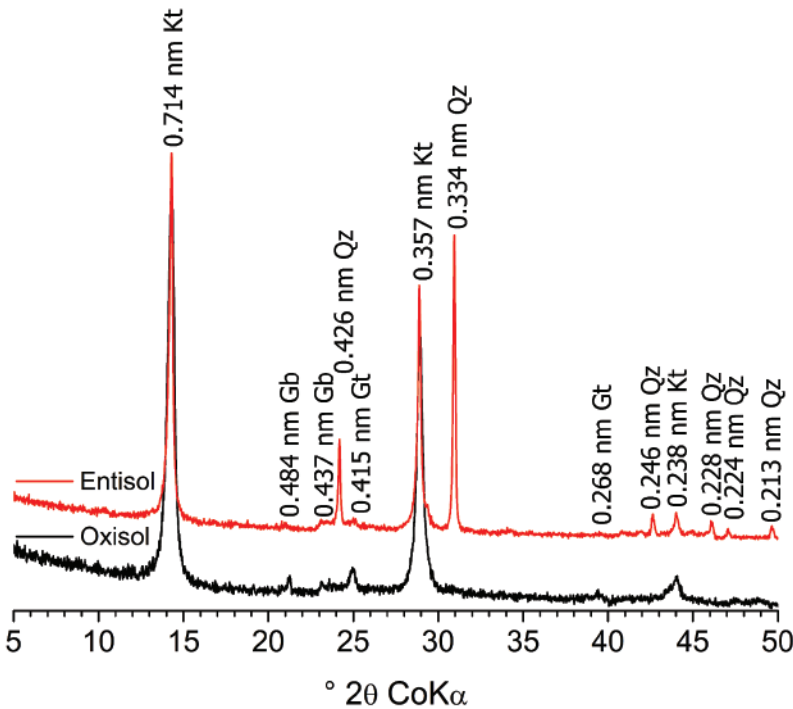
Figure 1. X-ray diffraction patterns of fraction <2 μm (natural) of the of neossolo Quartzarênico_NQ (Entisol_en), from Barreiras, Bahia state, and Latossolo Vermelho Amarelo_LVA (Oxisol_ox), from Viçosa, Minas Gerais state. Minerals: kt, kaolinite; gb, gibbsite; gt, goethite; and qz, quartz (interplanar distance in nm).

humidity was kept constant. The plastic bags were gently shaken periodically to avoid soil crusting and to facilitate gas exchange. Ninety days after limestone application, the soil samples were dried, passed through a 2 mm sieve, and the chemical analyses were carried out.

Chemical analyses

The pH in water (water-pH) was determined in soil suspensions with a 1:2.5 (soil: water) volumetric ratio. Exchangeable Ca^{2+} and Mg^{2+} were extracted with 1 mol L^{-1} KCl solution (Tedesco et al. 1995 and the residual forms (Ca_{res} and Mg_{res}) through solubilization with 0.5 mol L^{-1} HCl, according to the procedures described below. Five grams of soil samples were placed in an Erlenmeyer flask containing 50 mL of KCl solution. The material was incubated in an orbital shaker (200 rpm) for 5 min under stirring. Then, the sample was filtered through a fast-quantitative filter paper (∼25 μm pore size). The filtrate was used to measure Ca^{2+} and Mg^{2+} by atomic absorption spectrophotometry (AAS). Subsequently, 50 mL of deionized water was passed through the solid material withheld on the filter paper, aiming to remove possible retained exchangeable cations. The filter paper and the soil were transferred to a 125 mL Erlenmeyer flask containing 50 mL of HCl solution. The material was boiled (250–300°C) for 20 min using a hot plate. After filtration, the acid extract was transferred to a 50 mL volumetric flask, and the volume was adjusted with deionized water. The amount of Ca_{res} and Mg_{res} was determined by AAS. The limestone dissolution was assessed based on the mass balance between exchangeable and residual forms of Ca or Mg by using the following equations:

$$M_{exc} + M_{res} = M_{tot}, \tag{1}$$

$$M \ reacted \ (\%) = \frac{M_{exc}}{M_{tot}} \times 100 \qquad (2)$$

$$M \ unreacted \ (\%) = 100\% - M \ reacted, \qquad (3)$$

where M_{exc} is the amount of exchangeable metal (Ca^{2+} or Mg^{2+}), M_{res} is the amount of residual metal (Ca_{res} or Mg_{res}), and M_{tot} is the total amount. $M \ reacted$ is the proportion of metal from limestone dissolution and $M \ unreacted$ is the proportion of undissolved metal. The amount of native metal (Ca or Mg) was not discounted in the soil because it is negligible.

The results of the chemical analyses were not subjected to statistical tests because the objective was to detect the behavior of limestone dissolution under different conditions.

Chemical equilibrium approach

The Visual Minteq 3.1 software was used to simulate equilibrium conditions for partial pressure of carbon dioxide (pCO_2) of the dolomite-magnesite-calcite-atmosphere system, aiming to support the experimental results.

Results

pH

As expected, the water-pH increased with the limestone rate that is, liming decreased soil active acidity (Figure 2). The pH ranged from 4.2 to 8.5. The equilibrium pH in sandy soils (~8.2) was reached with ~ 6 t ha^{-1} of limestone. For clayey soils, the equilibrium pH (~7.8) was reached with ~ 20 t ha^{-1} of limestone. Therefore, sandy soils reached a higher equilibrium pH compared with clayey soils. Moreover, there was greater pH variation in sandy soils per applied limestone unit, because these soils have a low buffering capacity (Bloom 2000).

Exchangeable and residual ca and mg

The exchangeable Ca^{2+} content was positively responsive up to the dose of ~ 6 t ha^{-1} of limestone in sandy soils (0–20 mmol$_c$ kg^{-1}) (Figure 3); it then remained constant for subsequent limestone rates. In clayey soils, Ca^{2+} was responsive up to 20 t ha^{-1} of limestone (45–90 mmol$_c$ kg^{-1}), and then remained constant. Mg^{2+} was positively responsive up to ~ 1.5 t ha^{-1} of limestone in sandy soils, but from that dose
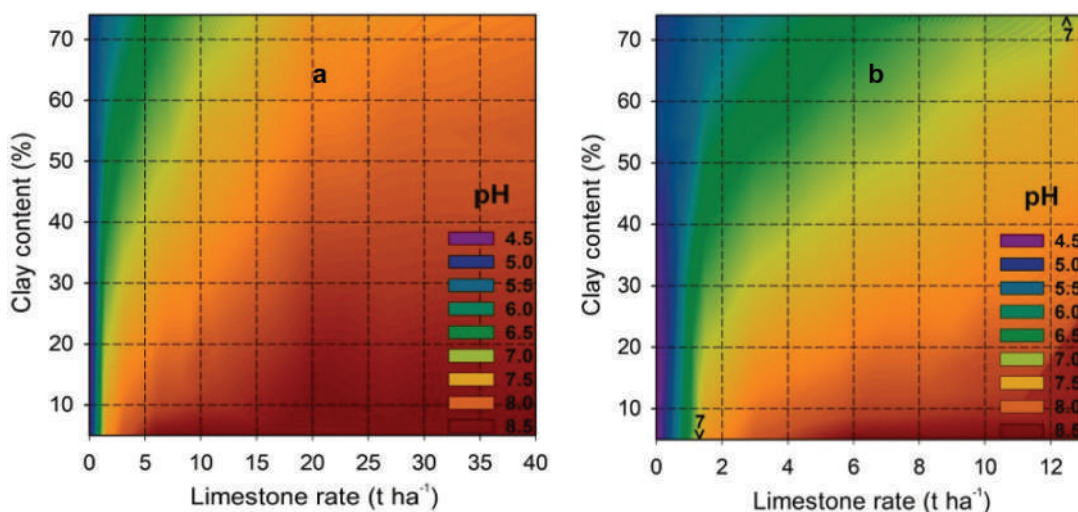

Figure 2. Soil texture and limestone rate affecting the water-pH. Panel B is a closer view of panel A; the arrow indicates pH 7.
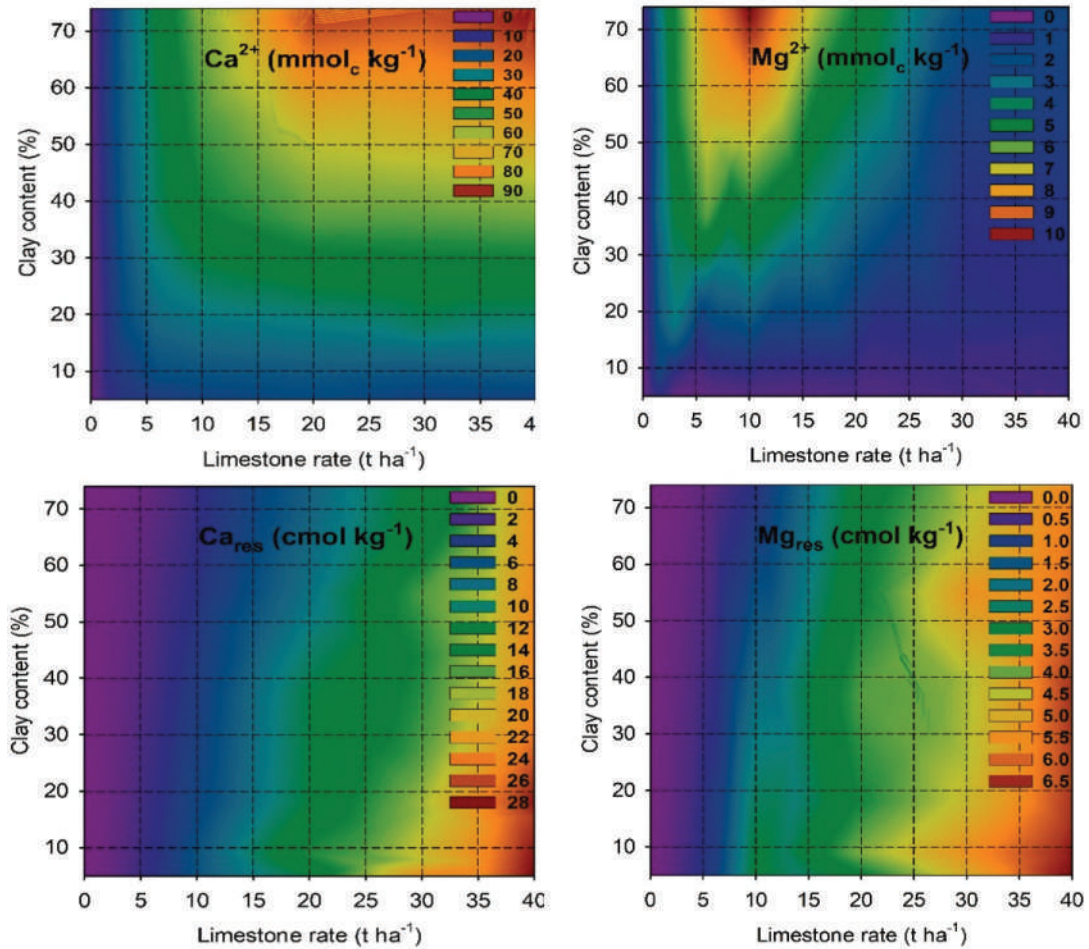
Figure 3. The soil texture and limestone rate affect the exchangeable forms of Ca^{2+} or Mg^{2+} (A and B) and the residuals forms (Ca_{res} or Mg_{res}) (C and D).

it decreased and stabilized around 0.3 $mmol_c\ kg^{-1}$. In clayey soils, Mg^{2+} was responsive up to ~ 10 t ha^{-1} of limestone (5–10 $mmol_c\ kg^{-1}$); however, at subsequent rates it gradually decreased and stabilized around ~ 1.5 $mmol_c\ kg^{-1}$ (Figure 3). Furthermore, regardless of the soil type, Ca_{res} and Mg_{res} increased as the limestone rate increased. However, sandy soils presented higher residual values compared with clayey soils for the same limestone rate (Figure 3). These results indicate that soil texture and the limestone rate affect exchangeable and residuals forms of Ca and Mg, and hence limestone dissolution.

Ca and mg from limestone dissolution

Higher limestone rates decreased the solubilization proportion, assessed as Ca release (Ca reacted, Figure 4), with the following means: 81%, 72%, 59%, 46%, 30%, 23%, and 17% Ca reacted for 1.5, 3, 6, 10, 20, 30, and 40 t ha^1 of limestone, respectively. A higher clay content promoted greater limestone dissolution, as evidenced by the increase in Ca reacted as the clay content increased: 27%, 34%, 41%, 54%, 60%, and 66% for 5%, 9%, 18%, 37%, 55%, and 74% clay, respectively. The Mg reacted was 72%, 54%, 35%, 20%, 5%, 2.3%, and 1.7% for 1.5, 3, 6, 10, 20, 30, and 40 t ha^1 of limestone, respectively. When considering the clay content, there was 7%, 12%, 21%, 34%, 41%, and 46% Mg reacted for 5%, 9%, 18%, 37%, 55%, and 74% clay, respectively. The mass balance between Mg reacted and Mg
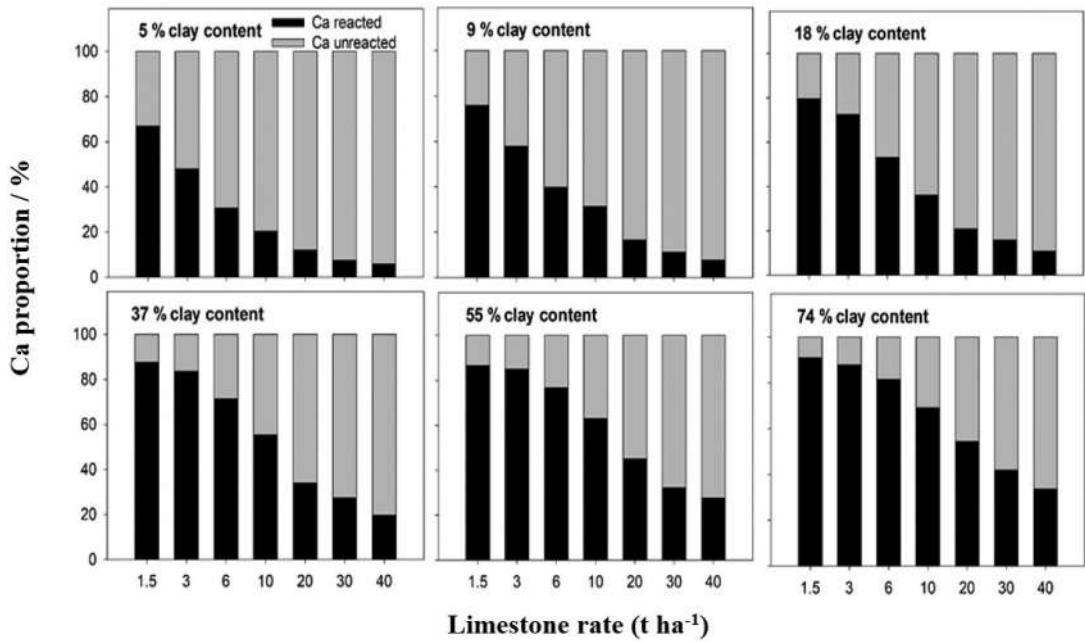
Figure 4. The soil texture and limestone rate affect the mass balance between ca reacted and ca unreacted from limestone.
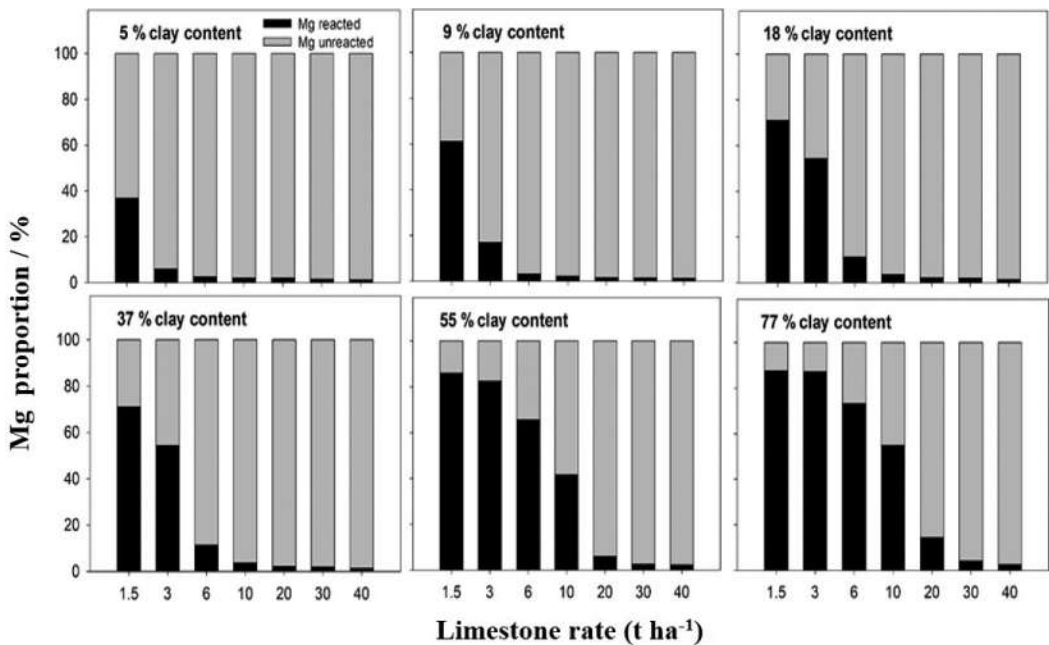

Figure 5. The soil texture and limestone rate affect the mass balance between mg reacted and mg unreacted from limestone.

unreacted (Figure 5) shows a trend similar to Ca mass balance, but to a lesser extent. That is, the Mg phases present in the limestone showed less dissolution (27% Mg reacted) than the Ca phases (47% Ca reacted). These results showed minor limestone dissolution when it was applied at higher rates, especially in sandy soils.

Discussion

pH

The maximum pH of the soil system in the context of overliming depends on pCO_2, the minerals that make up the lime, and the limestone rate. According to a simulation performed by Visual Minteq 3.1 (Gustafsson 2013), when considering the pCO_2 of the dolomite-magnesite-calcite-atmosphere system at equilibrium (38 Pa), the pH is 8.6, assuming 10 mmol L^{-1} ionic strength (simulating the soil solution) and 25°C. However, a soil system has a higher pCO_2, which reduces the pH at equilibrium. For example, the pCO_2 in the soil system (380 Pa) can be tenfold higher than in the atmosphere (38 Pa) (Lindsay 1979); thereby, the pCO_2 of the dolomite-magnesite-calcite-soil system would have pH 7.9 at equilibrium.

Only sandy soils showed an equilibrium pH (~8.2) approximate to that obtained in atmospheric conditions (8.6), indicating that the pCO_2 in sandy soils is close to the atmospheric pCO_2. This is justified because sandy soils have a low OM content and hence lower microbiological activity (CO_2-emitting source), besides greater CO_2 diffusivity due to its poor physical structure (Ball 2013). On the other hand, clayey soils present a higher OM content and microbiological activity and hence greater CO_2 emissions. This gas is trapped in aggregates and micropores (good physical structure), which are filled with water, reducing CO_2 diffusivity and increasing pCO_2 in the soil system. The pCO_2 in clayey soils in this study is similar to that suggested by Lindsay (1979) (380 Pa), because the equilibrium among the minerals resulted in pH ~ 7.8, a value consistent with the Visual Minteq output (7.9).
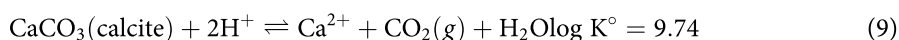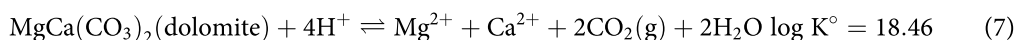
Nonetheless, unlike closed experiments (bag trials as shown here), field experiments in the context of overliming rarely show pH > 7.2. Fageria (2006) reported a pH of 7.2 after application of 24 t ha^{-1} of dolomitic lime (RTNP = 89%) in clayey soil (45% clay, no-tillage system). The lower pH from field experiments is related to the higher pCO_2 of these systems. Based on the crops, fertilization management, irrigation, and other factors, pCO_2 under field conditions is between 1 and 2 kPa (Karberg et al. 2005; Robbins 1986). In field conditions, there is a greater input of CO_2 through root respiration and OM decomposition. In addition, the soil physical structure under field conditions is superior to finely sieved soil samples (bag trials), reducing CO_2 diffusivity (Ball 2013) and hence increasing pCO_2.

In fact, in the context of overliming, even if the pCO_2 is high (2 kPa) the pCO_2 of the dolomite-magnesite-calcite-soil, the dolomite-calcite-soil, or the calcite-soil system provides a high pH at equilibrium (~7.5, ~7.2, and ~ 7.1, respectively) (Gustafsson 2013). Even so, these pH values are not suitable for most tropical crops (Fageria and Baligar 2008), because it reduces the nutrient availability in the soil, especially for metallic micronutrients such as zinc (Zn), copper (Cu), Mn, Fe, and nickel (Ni) (Barrow 2016; Fageria and Baligar 2008; Wang et al. 2006). In addition, precipitation of P as P-Ca phases in these conditions is highly expected. Therefore, liming must be carefully dimensioned, avoiding pH ≥ 7.0 (overliming).

In this sense, the potential acidity (H + Al) is an indicator that prevents overliming because it summarizes the different soil acidity pools at pH ~ 7. That is, the limestone rate (t ha^{-1}, RTNP = 100%) should not exceed the H + Al value ($cmol_c$ dm^{-3}). This approach is consistent with the results, as evidenced in Figure 1b, where the arrows indicate the limestone rates (RTNP = ~ 103%) that promoted ~ pH 7 in soil sample with 5% clay (Entisol, ~ 1.3 t ha^{-1} of limestone to 1.3 $cmol_c$ dm^{-3} of H + Al) and 74% clay (Oxisol, ~ 12 t ha^{-1} of limestone to 6.9 $cmol_c$ dm^{-3} of H + Al). In the soil with 74% clay, the H + Al value seems to underestimate the rate that promotes pH 7, because clayey soils are rich in Fe and Al hydro(oxides) and present a high buffering capacity. Consequently, these soils overcome calcium acetate solution buffering capacity.

Overliming affecting soil properties and limestone dissolution

Overliming promotes an excessive increase in soil pH, especially in sandy soils, which reduces the solubility of carbonates present in limestone, as reflected in equations 7, 8 and 9 (Lindsay 1979).

$$O_2(g) + H_2O \rightleftharpoons H_2CO_3{}^\circ \qquad\qquad \log K^\circ = -1.46 \qquad (4)$$

$$H_2CO_3{}^\circ \rightleftharpoons H^+ + HCO_3{}^- \qquad\qquad \log K^\circ = -6.36 \qquad (5)$$

$$HCO_3{}^- \rightleftharpoons H^+ + CO_3{}^{2-} \qquad\qquad \log K^\circ = -10.33 \qquad (6)$$

$$MgCa(CO_3)_2(dolomite) + 4H^+ \rightleftharpoons Mg^{2+} + Ca^{2+} + 2CO_2(g) + 2H_2O \quad \log K^\circ = 18.46 \qquad (7)$$

$$Mg(CO_3)(magnesite) + 2H^+ \rightleftharpoons Mg^{2+} + CO_2(g) + H_2O \quad \log K^\circ = 10.69 \qquad (8)$$

$$CaCO_3(calcite) + 2H^+ \rightleftharpoons Ca^{2+} + CO_2(g) + H_2O \quad \log K^\circ = 9.74 \qquad (9)$$

According to the equations, dolomite is the mineral most sensitive to pH variation: its solubility drastically reduces as pH increases. This explains the lower Mg reacted compared with Ca reacted in the context of overliming. Moreover, pCO_2 also influences the solubility of theses carbonates (Figure 7).

In sandy soils, where the pCO_2 is similar to atmospheric conditions ($pCO_2 = 38$ Pa), the Mg^{2+} activity maintained by dolomite is negligible at pH 7.1 (red arrow, sandy soils, Figure 7). This means that at pH < 7.1, dolomite and magnesite supply Mg^{2+} ions to the soil solution, and these ions are adsorbed by the CEC; consequently, exchangeable Mg reaches its maximum at around pH 7.1 (Figure 5). From that value, only magnesite maintains the Mg^{2+} activity, which decreases and becomes negligible at around pH 8.6. At pH > 8.6, the Mg^{2+} activity remains constant because it is controlled by the CEC (Lindsay 1979). This behavior of Mg^{2+} activity was the same as that of the exchangeable Mg, as evidenced by arrows of the same color in the Figures 6 and 7. This behavior also occurs in clayey soils, but at lower pH due to higher pCO_2. In a field experiment, Fageria (2006) evaluated the effect of limestone rates (0–24 t ha^{-1}) on the properties of Brazilian Oxisol (45% clay), and this author reported constant values of exchangeable Mg from the rate of 12 t ha^{-1} (pH 6.8), a result similar to the present study.

In addition, the Ca^{2+} activity maintained by dolomite and calcite follows the same pattern (see Figure 7): Ca^{2+} activity is reduced as the pH increases. However, the behavior of exchangeable Ca was
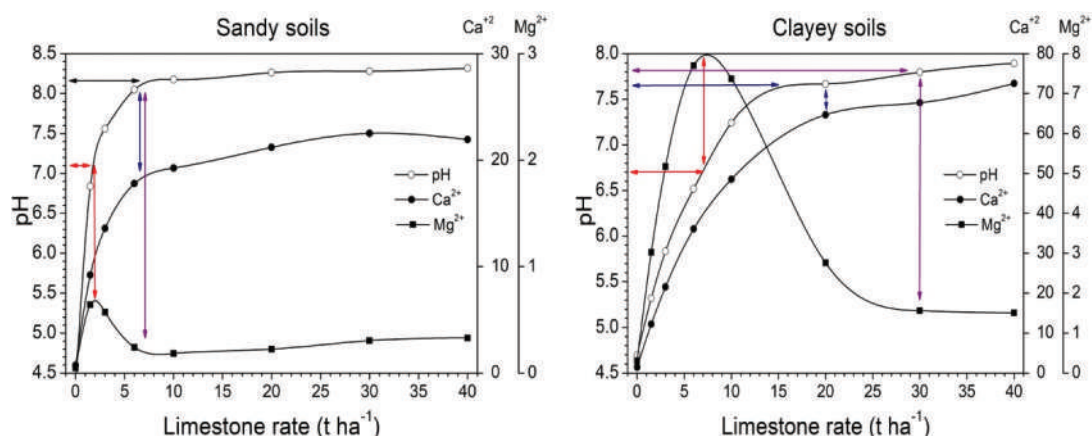

Figure 6. The limestone rate affects the water-pH and exchangeable Ca^{2+} and Mg^{2+} (mmol$_c$ kg^{-1}) in clayey soils (37%, 55%, and 74% clay) and sandy soils (5%, 9%, and 18% clay). The arrows indicate the point at which the variable changes its trend.
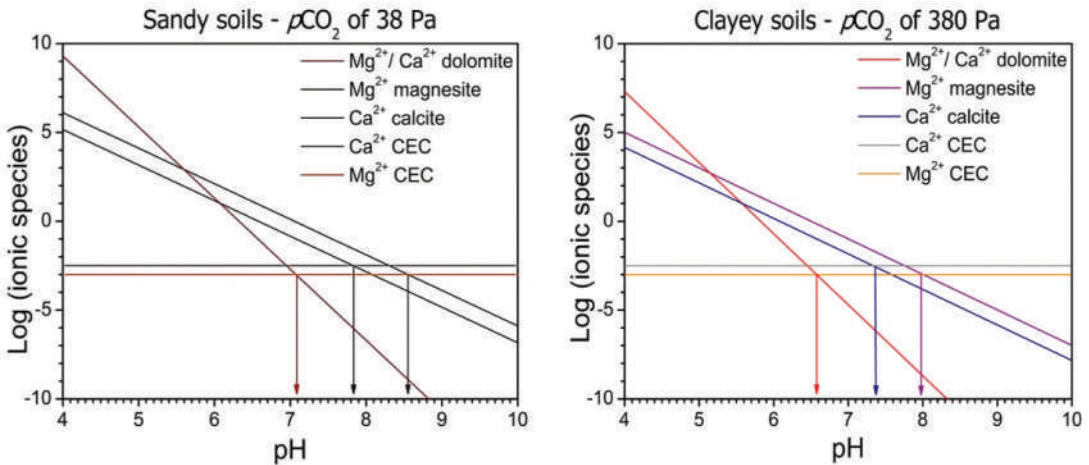
Figure 7. The pH and pCO_2 effects on the activity of ionic species (Ca^{2+} and Mg^{2+}) from dolomite-water (red line, eq. 7), magnesite-water (purple, eq. 8), calcite-water (blue, eq. 9), Ca^{2+} (gray), and Mg^{2+} (orange) maintained in the soil solution by the cation exchange capacity (Lindsay 1979).

not similar to that of exchangeable Mg. Exchangeable Ca continued to increase until calcite became insoluble. The distinct behavior of exchangeable Ca and Mg in the context of overliming (low ionic activity) may be linked to the lyotropic series, because the exchange sites have greater affinity for Ca. Consequently, the Ca/Mg ratio becomes larger in the context of overliming. Fageria (2006) reported the same behavior for exchangeable Ca, which was responsive up to 24 t ha^{-1} of lime (pH 7.1).

Soil texture affects limestone dissolution

From a physical point of view, sandy soils present a low specific surface area (Ersahin et al. 2006) to interact with limestone, besides greater moisture fluctuation due to its poor structure, setting a system of low reactivity, which promotes lower limestone dissolution. From a chemical point of view, equations 7, 8, and 9 demonstrate that H$^+$ activity is a key factor in lime dissolution. Thereby, soils with a low buffering capacity, such as sandy soils, show lower limestone dissolution due to the limited source of H$^+$ (clay edges and OM). Furthermore, the low clay and OM content of sandy soils mean they have a lower CEC. The soil's CEC acts as a cation sink contained in the soil solution, shifting the carbonate reactions (equations 7, 8 and 9) toward the products – that is, increasing limestone dissolution. Indeed, the soil buffering capacity and CEC are directly related to the amount of dissolved limestone (Warfvinge and Sverdrup 1989). These factors explain the lower values of Ca^{2+} and Mg^{2+}: Ca and Mg reacted in sandy soils and, hence, these soils have a lower capacity of limestone dissolution compared with clayey soils.

Conclusions

The low buffering capacity of sandy soils associated to the easy CO_2 exchange make them more sensible to overliming and with higher residual effect of liming. Furthermore, the high pH reduces carbonates solubility that make up lime, especially dolomite.

The greater lime dissolution and consequently a lower residual effect of liming in clayey soils is supported by their high buffering capacity for acidity, including their high CO_2 partial pressure compared to sandy soils.

Under overliming conditions, Mg availability decreases while Ca increases until the pH where calcite becomes poorly soluble.

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Disclosure statement

No potential conflict of interest was reported by the author(s).

ORCID

Fabiane Ballotin http://orcid.org/0000-0003-2872-6690

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