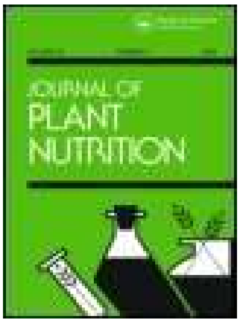

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Dunite solubilization kinetics in silicon-magnesium fertilization

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ABSTRACT

Dunite [$(MgFe)_2SiO_4$] is an igneous rock that remineralizes and corrects soil acidity as well as providing magnesium (Mg^{2+}) and silicon (Si^{4+}) to several crops of agricultural interest. However, few studies have verified its solubility in tropical soils, which is important for its use in different tropical conditions. In a greenhouse incubation experiment, this study verified the solubilization kinetics of dunite in clayey, medium texture, and sandy soils. The soil samples were dried and sieved (50 mesh), placed in plastic cups (300 g), and incubated for six months (at 60%–80% of the moisture retention capacity). Samples for pH ($CaCl_2$ extractor), Mg^{2+} (resin extractor), and Si^{4+} ($CaCl_2$ extractor) analysis were taken at 10, 20, 30, 40, 50, 60, 70, 85, 100, 115, 130, 150, and 180 days after incubation. The application of dunite led to an increase in Mg^{2+} and Si^{4+} contents and an acidity correction in the three studied soils. Its controlled release presents method for increasing the efficiency of magnesium fertilization. It allows for fewer losses due to Mg^{2+} leaching, as the dissolution kinetics are slower than soluble sources and have a residual effect for subsequent crops.

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Agromineral; magnesium fertilization; remineralizer; silicon; stonemeal

Introduction

Magnesium (Mg) fertilization is often neglected, and the lack of this mineral affects plant growth. Many essential plant functions require adequate sources of nutrients; the role of such nutrients is most obvious in root formation, chlorophyll, and photosynthesis. Plant functions that depend on an adequate Mg supply are often less visible (Cakmak and Kirkby 2008). Magnesium deficiency may be associated with the excessive use of calcite limestone and gypsum, and continuous potassium (K) application, conditions such as excessive fertilization that cause imbalance, or both (Moreira et al. 2003; Malavolta 2006).

The role of Mg^{2+} is well understood in several plant functions (Cakmak and Kirkby 2008). Magnesium deficiency in plants can be caused by soil depletion, but it can also be induced by the low assimilation of Mg^{2+} by the roots caused by competitive inhibition due to calcium (Ca^{2+}), potassium (K^{+}), manganese (Mn), and zinc (Mn) imbalances (Moreira et al. 2003; White and Broadley 2009; Cakmak and Yazici 2010; Tränkner, Tavakol, and Jákli 2018). Therefore, it is important to maintain an adequate balance between Mg and Ca, in addition to potassium, to ensure that an excess of one does not affect the availability of the other via antagonism.

Dunite is an igneous rock, being a peridot, magmatic, or eruptive, and resulting from the cooling of melted or semi-retained magma. It consists primarily of olivine, $(MgFe)_2SiO_4$, which

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usually has a grayish-olive color, although it may have a reddish color due to the oxidation of iron. It contains approximately 40% magnesium oxide (MgO) and 34% silica (SiO_2; Assis and Dias 2007). Dunite can be transformed into serpentine, which is a rock formed by the serpentinization process and composed of hydrated Mg and silica, with certain chemical characteristics and some variability in the structure. In serpentine, the dominant silicate mineral increases due to the hydration of olivine (forsterites), enstatite pyroxene, or both, in low-grade metamorphic conditions. It remains stable during the regional metamorphism of amphibolite facies, selecting pseudomorphically olivine crystals and preserving the original textures of rock and ore (Assis and Dias 2007).

According to Crusciol et al. (2019), dunite consists of a controlled source of Mg and Si, where the natural release mechanism may or may not be used industrially to increase the reactivity of soil material, in addition to simple grinding. The release mechanism of Mg and Si in remineralizers is known to be regulated by plant demand, since it is dependent on the removal of the products from the balance of dissolution, along with hydrolysis of the minerals that make up the rock. This is an even more important factor than soil acidity (Moretti et al. 2019).

The mechanism of Mg and Si release to plants depends on the removal of products from the equilibrium of dissolution and hydrolysis of minerals that make up the rock (Castro and Crusciol 2013; Castro et al. 2016). The main consequence of controlled release is the potential to increase the magnesium fertilization efficiency; the dissolution kinetics are slower than soluble sources, leading to reduced leaching of Mg^{2+}. However, soil texture is also a factor in this process, as soil clay content affects water storage, organic matter contents, and, consequently, microbiological activity (Cai et al. 2013).

Thus, the objective of this study was to evaluate dunite solubilization kinetics, pH changes, and Mg and Si content changes in sandy, medium texture, and clay soils over a period of six months.

Materials and methods

The incubation experiment was conducted under greenhouse conditions at the Department of Crop Science, College of Agricultural Sciences, São Paulo State University, Botucatu, São Paulo State, Brazil. According to the Brazilian soil classification system (Santos et al. 2018), the three soils used in the experiment are classified as clayey Latosol, medium texture Latosol, and sandy Latosol, which corresponds to a Typical Ferralsol (FAO (Food and Agriculture Organization) 2006) and an Oxisol (Soil Survey Staff 2014). The physical and chemical properties of the soils (0.00–0.20 m depth) are presented in Table 1. The physical properties were determined according to the methods of Donagema et al. (2017), while the chemical properties were determined according to the methods of Raij et al. (2001).

The chemical characteristics of the dunite used in this study are shown in Table 2. X-ray diffraction analysis (XRD) was performed to determine and quantify the mineralogical phases (Table 2). The identification of the crystalline phases was obtained by comparing the sample with the PDF2 database of the International Center for Diffraction Data (ICDD) and the Inorganic Crystal Structure Database (ICSD) (see Moretti et al. 2019). The values were calculated by the Rietveld method (Young 1993), using the standard ICSD crystalline structures and internal fluorite (CaF_2) to determine the amorphous phase.

All soil samples were dried and sieved (50 mesh). For each sample, the incubation experiment was set up with 300 g of soil in a 500 mL plastic pot. The dunite dose was 1.7 g kg^{-1} of soil, adapted from Camargo, Korndörfer, and Pereira (2007) and Buck, Korndörfer, and Datnoff (2010). The pot lids were perforated to allow gas exchange and microbial respiration (Cai et al. 2013). Soil water retention capacity was determined on a tension table using a Richards extractor

Table 1. The mean of physical-chemical properties of three soil types (0.0–0.2 m depth) from Botucatu, São Paulo State, Brazil.

Properties			Soils		
			Clayey	Medium	Sandy
Physical		Unit		Value	
Clay		g kg^{-1}	602	353	259
Silt		g kg^{-1}	281	478	724
Sand		g kg^{-1}	117	169	17
Bulk density		g cm^{-3}	1.13	1.23	1.3
Chemical					
pH, $CaCl_2$		–	4.3	4.0	4.3
Total organic carbon		g kg^{-1}	15.2	14.3	13.5
Total nitrogen (N)		g kg^{-1}	1.00	0.94	0.85
Phosphorus–available ($P_{Mehlich\ 1}$)		mg kg^{-1}	24.0	18.0	13.0
Exchangeable	Calcium ($Ca^{2+}{}_{resin}$)	$mmol_c$ kg^{-1}	20.0	12.0	9.0
	Magnesium ($Mg^{2+}{}_{resin}$)	$mmol_c$ kg^{-1}	5.7	5.2	3.1
	Potassium ($K^{+}{}_{resin}$)	$mmol_c$ kg^{-1}	1.7	1.5	1.1
	Aluminum ($Al^{3+}{}_{KCl}$)	$mmol_c$ kg^{-1}	4.0	2.0	2.0
Potential acidity (H + Al)		$mmol_c$ kg^{-1}	35.0	27.0	22.0
S-Sulfate ($S–SO_4^{2-}{}_{Ca(H2PO4)2}$)		mg kg^{-1}	5.6	3.0	1.5
Boron ($B_{Hot\ water}$)		mg kg^{-1}	0.4	0.3	0.3
Copper ($Cu_{DTPA-TEA}{}^{a}$)		mg kg^{-1}	8.4	2.7	1.6
Iron ($Fe_{DTPA-TEA}$)		mg kg^{-1}	50	43	35
Manganese ($Mn_{DTPA-TEA}$)		mg kg^{-1}	15.3	8.8	1.9
Zinc ($Zn_{DTPA-TEA}$)		mg kg^{-1}	0.9	0.9	0.3
Base saturation (BS)		%	43.9	40.9	37.5
Cation exchange capacity ($CEC_{pH\ 7.0}$)		$mmol_c$ kg^{-1}	62.4	45.7	35.2

[a] DTPA-TEA = diethylenetriaminepentaacetic acid-triethanolamine.

Table 2. Mean of heavy metals, mineralogical phases, and composition of two dunite sources. Botucatu, São Paulo State, Brazil.

Description	Chemical symbol	Value	Unit
Heavy metals			
Arsenic	As^{3+}	3.79	mg kg^{-1}
Cadmium	Cd^{2+}	0.8	mg kg^{-1}
Lead	Pb^{2+}	2.5	mg kg^{-1}
Mercury	Hg^{2+}	<0.20	mg kg^{-1}
Selenium	Se^{2+}	<0.10	mg kg^{-1}
Chromium	Cr^{3+}	0.037	%
Nickel	Ni^{2+}	0.239	%
Mineralogical phases			
Lizardite	$Mg_3(Si_2O_5(OH)_4)$	48	%
Magnetite	Fe_3O_4	9	%
Brucite	$Mg(OH)_2$	26	%
Magnesioferrite	$MgFe_2O_4$	17	%
Composition			
Magnesium oxide	MgO	40	%
Silicon dioxide	SiO_2	34	%

device (Richards and Fireman 1943), which constructed water retention curves. Soil water potential (Ψw) was determined for each soil sample.

Before the fertilizers were applied, soil moisture was adjusted to 80% of the field capacity. The soil moisture was monitored daily by weighing the pots on an electronic scale, and was maintained between 60% and 80% of the field capacity. The greenhouse was held at a constant temperature (27–32 °C; Janzen and Bettany 1987), had natural lighting, and relative agriculture humidity ranged from 40% during the afternoon to 80% in the morning. For all samples, the soil was air-dried, sieved, and mixed, and the pH, Mg, and Si values were obtained for each incubation period. The soil pH was determined in calcium chloride ($CaCl_2$) suspensions at 0.01 mol L^{-1} (a soil to solution ratio of 1:2.5). Mg content was determined using resin according to the method

Table 3. The *F* probability as a function of the treatments used. Botucatu, São Paulo, Brazil, 2019.

Factors	pH	Mg	Si
ANOVA (*F probability*)		Clayey Soil	
Dunite Application (DA)[a]	≤ 0.001	≤ 0.001	≤ 0.001
Incubation period (IP)[b]	≤ 0.001	≤ 0.001	≤ 0.001
DA × IP	≤ 0.001	≤ 0.001	≤ 0.001
C.V.	0.8	2.9	3.1
LSD	0.05	0.4	0.4
		Medium Texture Soil	
Dunite Application (DA)	≤ 0.001	≤ 0.001	≤ 0.001
Incubation period (IP)	≤ 0.001	≤ 0.001	≤ 0.001
DA × IP	≤ 0.001	≤ 0.001	≤ 0.001
C.V.	0.7	4.3	2.9
LSD	0.05	0.4	0.5
		Sandy Soil	
Dunite Application (DA)	≤ 0.001	≤ 0.001	≤ 0.001
Incubation period (IP)	≤ 0.001	≤ 0.001	≤ 0.001
DA × IP	≤ 0.001	≤ 0.001	≤ 0.001
C.V.	0.7	3.2	3.0
LSD	0.06	0.4	0.4

[a] Significantly different: Fisher's least significant (LSD) test at $p \leq 0.05$.

[b] Significant polynomial regression was selected with the highest logarithmic model determination coefficients.

used by EMBRAPA (Brazilian Agricultural Research Corporation) (1997). Si content was determined by spectrophotometry after extraction with 0.01 mol L^{-1} $CaCl_2$ (Korndörfer, Pereira, and Nolla 2004).

Experimental design and sampling

The treatments were arranged in a completely randomized design with 3 soils × 2 levels of dunite (with- and without-) × 13 sampling intervals (Days 10, 20, 30, 40, 50, 60, 70, 85, 100, 115, 130, 150, and 180) were employed with four replicates each. The incubation period was adapted from MAPA (Ministry of Agriculture and Livestock and Supply) (2019) using a sequence of evaluations. The first seven samples were taken every 10 days until 70 days and the next four samples were taken every 15 days from 70 to 130 days. The subsequent sample was taken after 20 days (day 150) and the final sample was taken after 30 days (day 180). Thirteen samples were taken overall.

Statistical analyses

All the data were initially analyzed using the Shapiro-Wilk test (Shapiro and Wilk 1965) for normality and the Levene test for homoscedasticity (Levene 1960), both at 0.05 probability ($p \leq 0.05$) levels. The UNIVARIATE procedure of SAS version 9.4 was used for these analyses (SAS Institute 2015). The data were also tested for sphericity using the Bartlett test (Bartlett 1937) via the FACTOR procedure of SAS version 9.4 (SAS Institute 2015). The results indicated that all the data were distributed normally ($W > 0.90$) and exhibited no sphericity. The data were also subjected to analysis of variance (ANOVA). Polynomial regression analysis was performed to determine the response curves of the solubilization kinetics for soil characteristics and significant polynomial regression equations were used.

Results and discussion

Results indicated a significant interaction (Table 3) between dunite application and incubation period. Our results also demonstrated that pH values increased in tropical acid soils (Figures 1, 2,

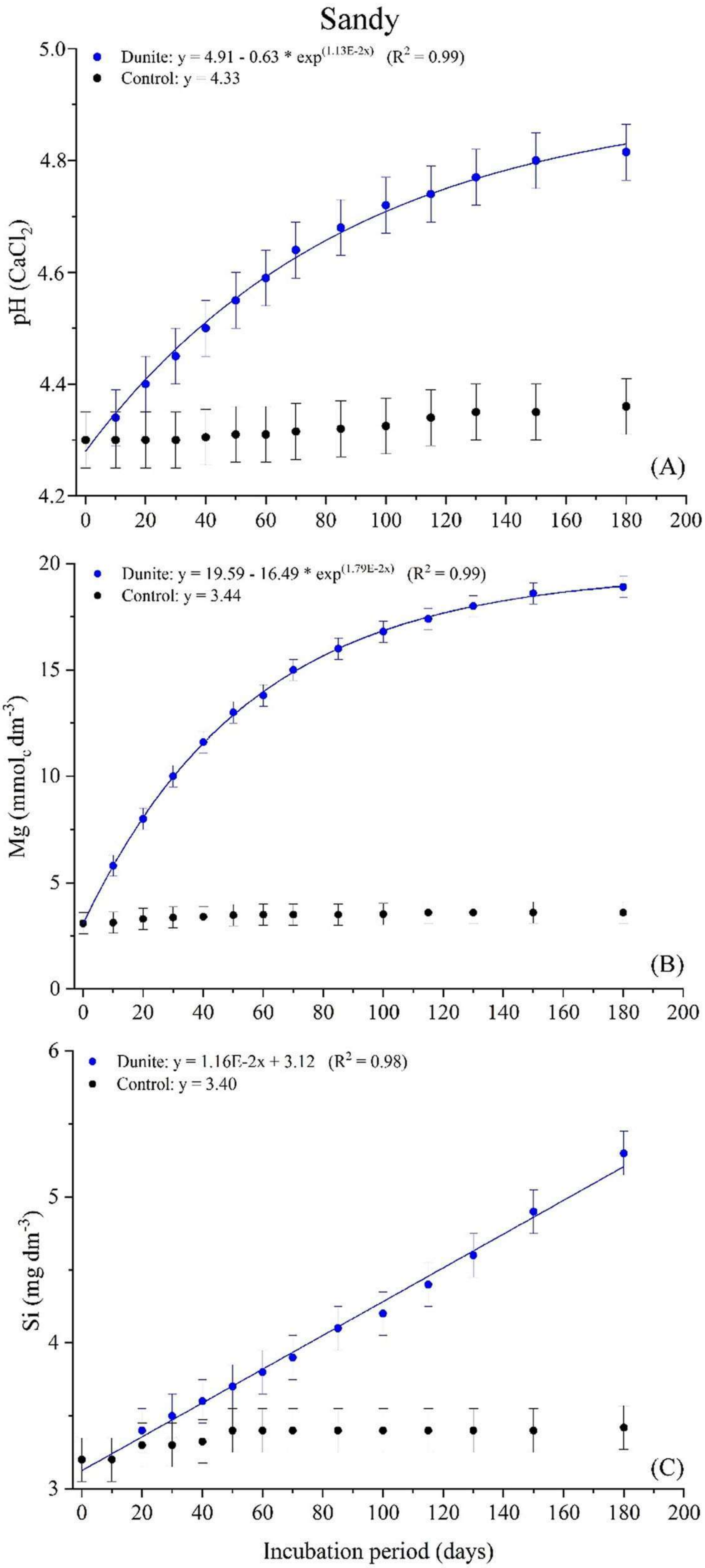

Figure 1. The pH, Mg content, and Si content in soil with sand texture, in relation to dunite application and incubation period. Botucatu, São Paulo, Brazil.

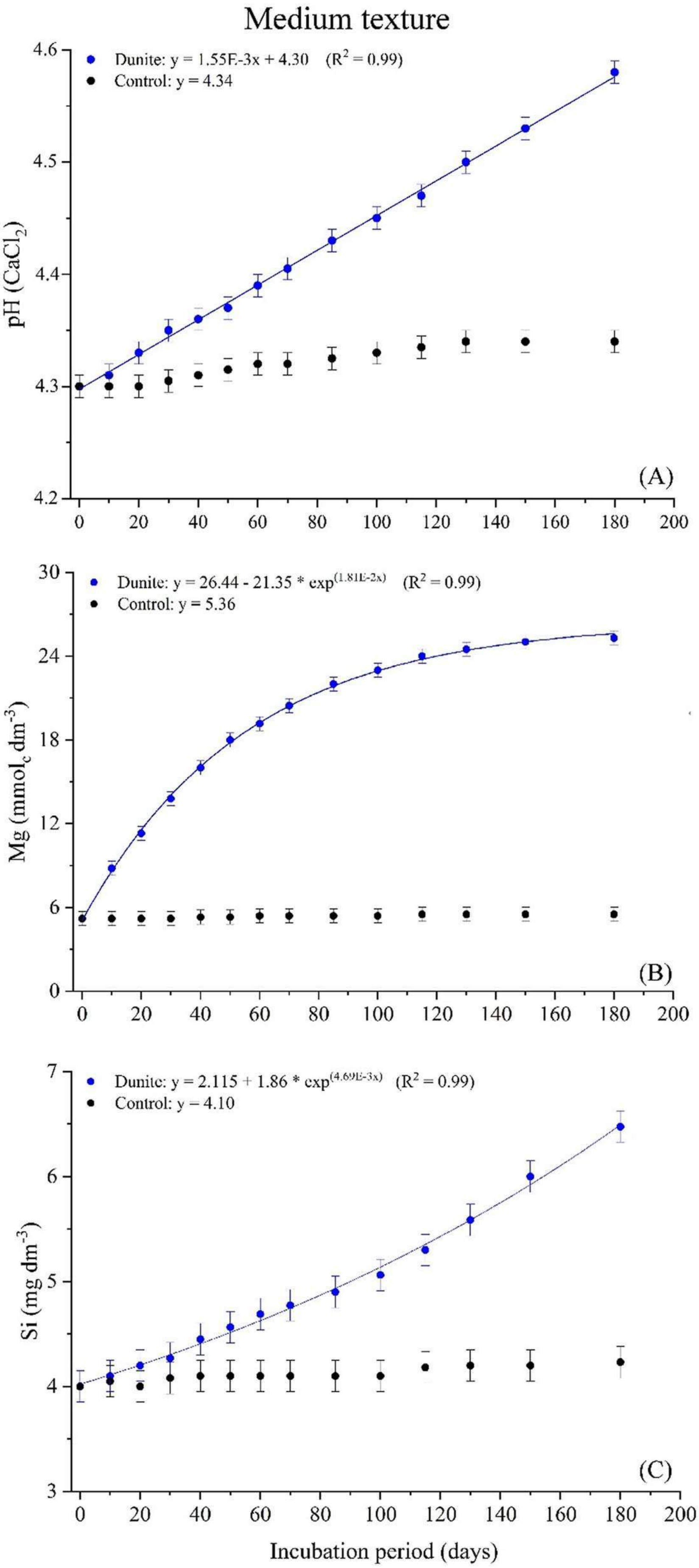

Figure 2. The pH, Mg content, and Si content in soil with medium texture, in relation to dunite application and incubation period. Botucatu, São Paulo, Brazil.

and 3) in all periods analyzed, which characterizes the reactivity of the remineralizer. The pH behavior in the sandy and medium texture soils was like a quadratic curve and maximum point. This was different from that of the clayey soil, where the pH linearly increased with the application of dunite, without a 180-day evaluation period (Figure 3A). This is due to the buffering power of soils; in general, clay soils have higher clay and organic matter contents and, therefore, higher cation exchange capacity (CEC) values. This factor influences the resistance to pH change, requiring a greater amount of corrective material to promote the increase of this index.

In the soil solution, after the magnesium silicate crystals ($MgSiO_3$) undergo dissolution and hydrolysis, the Mg^{2+} cation leaves the metasilicate coordination (SiO_3^{2-}) and the anion tends to stabilize, coordinating the H^+ ions originating from the hydrolysis of water (H_2O). This causes an increase in pH values, with subsequent hydration to H_4SiO_4, orthosilicate, or monosilicic acid. According to Alcarde (2005), the increase in pH is based on the neutralizing action of SiO_3^{-2}, through equations I to IV:

$$MgSiO_3 \leftrightarrow \ Mg^{+2}{+}SiO_3{}^{-2} \qquad [I]$$

$$SiO_3{}^{-2} + H_2O\ (soil)\ \leftrightarrow\ HSiO_3{}^{-} + OH^{-} \qquad [II]$$

$$HSiO_3{}^{-} + H_2O\ (soil)\ \leftrightarrow\ H_2SiO_3 + OH^{-} \qquad [III]$$

$$H_2SiO_3{+}H_2O\ (soil)\ \leftrightarrow\ H_4SiO_4(monosilicic\ acid) \qquad [IV]$$

The final product, $H_4SiO_4{}^{\circ}$, remains in the system until the polymerization process begins. This is where acidity is released, according to Alves (2014), through equation V:

$$4H_4SiO_4{}^{0} \leftrightarrow\ H_6SiO_4O_{12}{}^{2-}{+}2H^{+} + 4H_2O \qquad [V]$$

The polymerization of orthosilicate affects the final balance of neutralization of acidity by $MgSiO_3$ in soils. In general, four moles of $MgSiO_3$ will neutralize only six moles of H^+ due to the generation of two moles of H^+ resulting from the polymerization of Si, following equation VI:

$$4MgSiO_3{+}6H^{+} \leftrightarrow\ 4Mg^{2+} + H_6SiO_4O_{12}{}^{2-} \qquad [VI]$$

The result of this aqueous polymerization of the $H_4SiO_4{}^{\circ}$ species explains the low magnitude of the pH increase over 180 days in the three soils with added dunite. According to Moretti et al. (2019), the remineralizer has features of acidity correction, providing the soil with a controlled pH increase, partially due to the formation of Si polymers that have an acid character. The solubilization is a little slower than lime and steel slag, which has a smaller impact on the microbiota that decomposes soil organic matter (SOM) and is extremely sensitive to changes in acidity (Imaizumi, Rossi, and Fortes 2018).

Dunite has a greater capacity to undergo dissolution and hydrolysis processes than coarser granulometries because of its greater contact surface. According to Korndörfer (2002), macrominerals should have a high content of soluble Si, adequate physical properties, ease of mechanized application, low cost, satisfactory levels of nutrient(s), absence of heavy metals, and adequate availability for plants. The latter is influenced by the grain size of the material. According to Carvalho (2012), particles larger than 0.2 mm negatively affect the speed of solubilization, such that the larger the surface area, the greater the reactivity.

According to Carvalho (2012), agrominerals particles larger than 0.2 mm negatively affect the speed of solubilization, allowing us to infer that the larger the specific surface, the greater the reactivity. In addition to grain size, other factors related to the genesis of the rock are relevant to solubility and need to be understood to define the susceptibility of the mineral contents to weathering that occurs in the soil. Harley and Gilkes (2000) and Manning (2010) also suggested that assessments of the potential of rocks to be used in plant supplementation are based on their

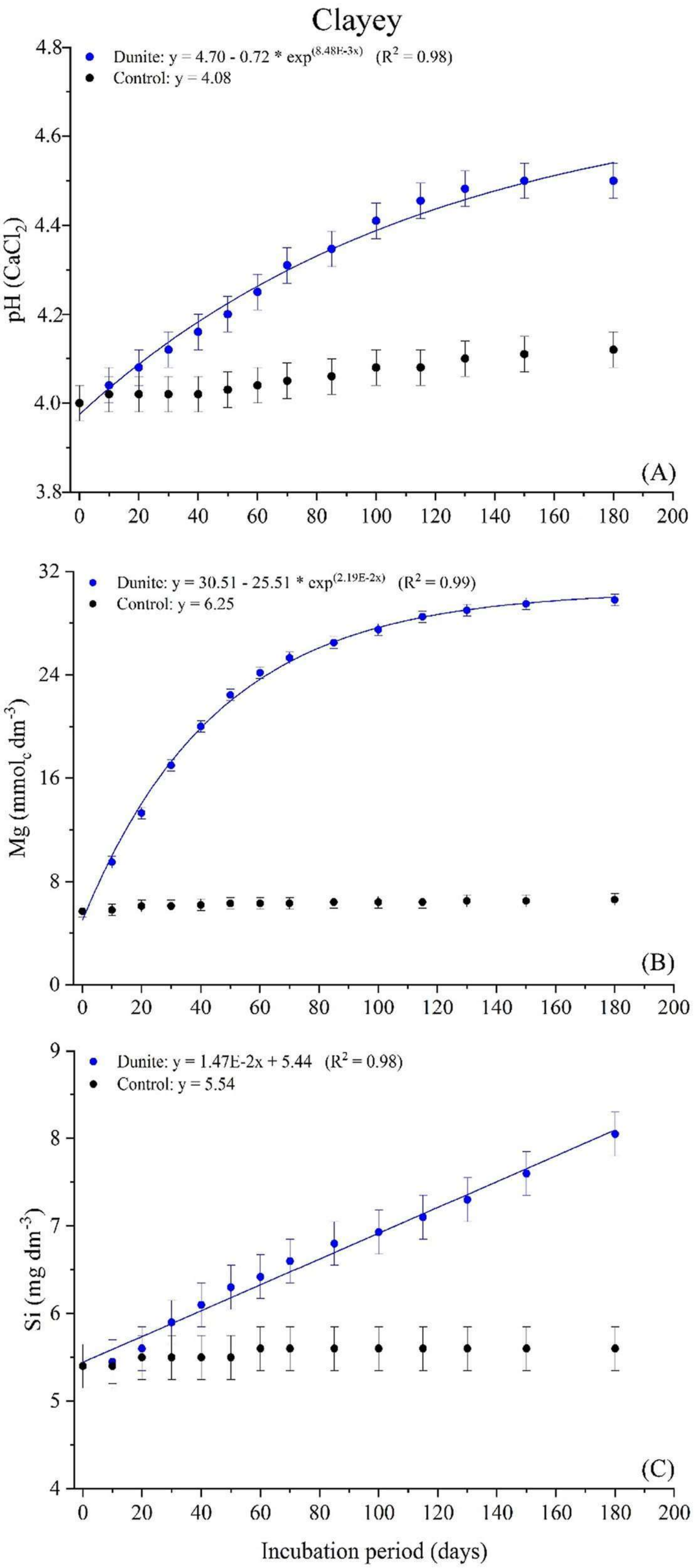

Figure 3. The pH, Mg content, and Si content in soil with clayed texture, in relation to dunite application and incubation period. Botucatu, São Paulo, Brazil.

geochemical composition to avoid doubts about the results. Nascimento and Lápido-Loureiro (2004) reported that mineralogy can indicate the solubility potential of minerals and their ability to release nutrients. Therefore, understanding the mineral and geochemical composition of rocks is essential for choosing which ones have the greatest effect as a source of nutrients.

Dunite is predominantly made up of minerals from the olivine and serpentine groups, such as lizardite ($Mg_3Si_2O_5(OH)_4$); Table 2), comprising 46% of the remineralizer. Lizardite consists of a magnesium silicate with moderate resistance to weathering, guaranteeing initial and medium-term responses to the availability of Mg and Si. Another important mineral in the constitution of dunite is brucite ($Mg(OH)_2$; Table 2), comprising 26% of the remineralizer. Brucite is formed from the serpentinization of dunite and has low resistance to weathering, guaranteeing rapid responses in the field in relation to the availability of Mg. Lizardite and brucite are largely responsible for the supply of Mg and Si from the dunite fertilizer, which is the subject of the present study.

Regarding the availability of Mg^{2+} (Figures 1B, 2B, and 3B), for the three soils analyzed, the remineralizer was extremely reactive. In the first 30 days, the Mg availability in sandy soils of medium texture and clayey soils were improved, promoting significant amounts of the referred nutrients to the soil solution. However, over time, the weathering of the remineralizer stabilized and, consequently, the supply of Mg to the soil system also stabilized. The present study used soils only, without crop cultivation. Therefore, as the balance between the solid phase of the mineral and the liquid agriculture phase of the soil solution was established, the weathering rate and consequent Mg solubilization decreased. This gradually led to lower levels of this element in later soil samples, which tended to stabilize over time.

According to Carvalho (2012), this stabilization is expected, since in agricultural soil the dissolution of minerals is reinforced by an imbalance between the solution and the mineral surfaces through the removal of ions. This occurs through processes such as leaching and nutrient uptake by plants. The rhizosphere's own interference and other biological activities can also increase the dissolution of minerals through the release of H^+ ions and the complexation carried out by organic compounds that react with their surfaces. Such situations were not applied in the present study due is only related to the dynamics of Dunite solubilization in the soil.

Studies on the efficiency of agrominerals in supplying Mg are especially important because of the few known sources of this nutrient on the market, such as magnesian and dolomitic limestones. Furthermore, inadequate acidity corrections (supplying only Ca) and high K fertilization rates often negatively interfere with the interactions between plant and soil, especially in the uptake of Mg.

Crusciol et al. (2019) upon supplementing corn crops with dunite, observed a positive effect in the increase of Mg and Si in the soil and leaf tissue, causing translocation of sucrose and starch from the leaves to the grains and subsequently increasing grain yield. Moretti et al. (2019) observed reduced sugars and foliar glucose as well as pH, Mg, and Si of the soil when a soybean crop was dosed with dunite, with yield components showing a positive response due to the increase in input rates. Mg nutrition leads to lower foliar starch levels; consequently, a better partitioning of metabolites to plants leads to better development, filling, and yield of soybeans.

Ramos et al. (2006) compared the effect of Ca and Mg silicate with calcitic limestone incubated in the soil for 40 days and found that these inputs had similar capacities to increase the exchangeable Ca content in the soil, along with a greater capacity for making Mg available as a silicate. Castro and Crusciol (2015) found that, after 180 days of superficial dolomitic limestone application and Ca and Mg silicate on soil under a no-tillage system, both limestone and silicate increased the levels of Ca^{2+} (at 0.0–0.2 m depth), and Mg^{2+} (0.0–0.1 m depth) compared to the control treatment, with no significant difference between corrections.

In relation to Si, the application of dunite in this study increased the content of this element in the three soil types over time (Figures 1C, 2C, and 3C). However, unlike the behavior of Mg, this rise in availability occurred more slowly, with an increasing linear trend. The gradual solubility of dunite Si can again be explained by mineralogy. Si is part of all the magnesium silicate mineral structures that make up the remineralizer, which provide, in their majority, moderate stability against weathering in the soil. It can even form intermediate fractions, such as clay minerals, before complete dissolution. However, its availability occurs in a controlled and constant manner. This is important for semi-perennial and perennial crops such as sugarcane, eucalyptus, pastures, coffee, oranges, and fruit in general, as nutrient release rates can be better adjusted to the demand of cultures over time.

Several field and greenhouse studies have been conducted to verify the effectiveness of Mg and Si sources. In general, sources of Si such as wollastonite, blast furnace slag (Carvalho-Pupatto et al. 2003), steel aggregates, shale and thermophosphate (Moreira, Malavolta, and Moraes 2002; Pereira et al. 2004), and dunite (Crusciol et al. 2019; Moretti et al. 2019) increase soil Si and Mg content and increase soil pH, reducing the concentration of phytotoxic Al^{3+}. However, there is still a lack of knowledge regarding which materials are the most promising, which methods are most suitable for analysis, dosages, ideal granulometry characteristics, ways to increase the solubility of these materials, and performance in the cultivation of different species. In addition, Pádua (2012) stresses that the interaction of agrominerals characteristics (mineralogy, chemistry, granulometry, and solubility), soil properties (pH, texture, organic matter content, presence of microorganisms, and moisture), and crop characteristics (species, crop cycle, and nutritional requirements), among other environmental and management factors, have a great influence on the agrominerals effects.

Conclusions

As a remineralizer, dunite provides an option for more sustainable agriculture, increasing soil Mg, Si, and pH in sandy, medium, and clayey soils over a six-month period. Further research should investigate the benefits of this to crop growth in rotational crop agriculture.

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Disclosure statement

No potential competing interest is reported by the authors.

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