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ANO: 2021

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* Evento de Classificação:

CLASSIFICAÇÕES DE PERIÓDICOS QUADRIÊNIO 2017-2020 ▼

Área de Avaliação:

☒ CIÊNCIAS AGRÁRIAS I ▼ +

ISSN:

☐

Título:

☒ Revista Brasileira de Ciências Agrárias

Classificação:

☐ -- SELECIONE -- ▼

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Periódicos

ISSN	Título	Área com publicação no quadriênio	Classificação	Área mãe
1981-0997	REVISTA BRASILEIRA DE CIENCIAS AGRARIAS	CIÊNCIAS AGRÁRIAS I	B2	CIÊNCIAS AGRÁRIAS I

Mineralogy and genesis of soils developed from ultrabasic and alkaline rocks of Lages Alkaline Complex, Brazil

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ABSTRACT: In the Plateau of Lages, intrusions of alkaline and ultrabasic rocks occur, rocks of rare occurrence in Brazil, whose soils derived from them are practically unknown. The study aimed to evaluate the chemical composition, mineralogy, genesis, and classification of the soils developed from the rocks Nepheline Sienite (P1), Volcanic Breccia (P2), Kimberlitic Breccia (P3), Phonolitic Tephrite (P4), and Phonolite (P5). After the morphological description (field) and the collection of soil samples, chemical analyses of the soils, mineralogical of the clay fraction and the total elements of the soils and the corresponding rocks that gave rise to them were carried out. Three Humidepts (Cambissolos), a Hapludolls (Chernossolo), and a Kandiodox (Nitossolo) were identified. Except for the P3 profile, the others are acidic soils, with high levels of exchangeable aluminum, poor in basic cations, with low sum and base saturation, showing a high degree of weathering and strong leaching. In the clay fraction, the presence of 2:1 clay minerals predominated with kaolinite with interlayered hydroxy-Al polymers. The P3 profile showed a slightly acidic reaction, high sum and base saturation, and practically zero exchangeable aluminum, a situation compatible with mineralogy, where trioctahedral vermiculite predominated, revealing that it was a young, slightly weathered soil.

Key words: chemical properties; genesis; mineralogy; soils derived from alkaline and ultrabasic rocks

Mineralogia e gênese de solos desenvolvidos de rochas alcalinas e ultrabásicas do Complexo Alcalino de Lages, Brasil

RESUMO: No Planalto de Lages, ocorrem intrusões de rochas alcalinas e ultrabásicas, rochas de rara ocorrência no Brasil, cujos solos delas derivados são praticamente desconhecidos. O estudo objetivou avaliar a composição química, a mineralogia, a gênese e a classificação dos solos desenvolvidos das rochas Nefelina Sienito (P1), Brecha Vulcânica (P2), Brecha Kimberlítica (P3), Tefrito Fonolítico (P4) e Fonolito (P5). Para isso, após a descrição morfológica (campo) e a coleta de amostras de solos, procedeu-se as análises químicas dos solos, mineralógicas da fração argila e dos elementos totais dos solos e das rochas correspondentes que lhes deram origem. Foram identificados três Cambissolos, um Chernossolo e um Nitossolo. Com exceção do perfil P3, os demais são solos ácidos, com elevados teores de alumínio trocável, pobres em cátions básicos, com baixa soma e saturação por bases, evidenciando alto grau de intemperismo e forte lixiviação. Na fração argila predominou a caulinita a presença de argilominerais 2:1 com polímeros hidróxi-Al entrecamadas. O perfil P3 apresentou reação ligeiramente ácida, elevada soma e saturação por bases, e alumínio trocável praticamente nulo, situação compatível com a mineralogia, onde predominou a vermiculita trioctaedral, revelando tratar-se de um solo jovem, pouco intemperizado.

Palavras-chave: propriedades químicas; gênese; mineralogia; solos derivados de rochas alcalinas e ultrabásicas

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Associate Editor: Clístenes Williams Araújo Nascimento

Introduction

In the region of Plateau of Lages, SC, lies the Lages Alkaline Complex (Complexo Alcalino de Lages) (CPRM, 2014), a formation composed of intrusions of alkaline and ultrabasic rocks. Although they occupy a small geographic extension, the rocks in the complex are described by Scheibe (1986) as peculiar, with great lithological diversity, where phonolites, nepheline syenites, olivine-melilithites, lamprophyres, in addition to carbonatites, kimberlites, and breccia pipe stand out (Machado et al., 2012).

Characteristics of soils developed from rocks derived from the lava flow from the Serra Geral Formation and Gondwana sedimentary rocks, dominant in the region, are reasonably well known (Ferreira, 2013; Teske et al., 2013). However, for rocks developed from the Lages Alkaline Complex, there is a single study conducted by Ferreira (2013), in which soils derived from phonolite, syenite, and olivine melilithite were studied.

Almeida et al. (2019) mainly identified Cambissolos Húricos e Háplicos derived from Gondwana sedimentary rocks, all with strongly acidic reaction and low natural fertility, which presented clay mineralogy dominated by kaolinite, but with variable and expressive proportions of 2:1 clay minerals with hydroxy-Al interlayer polymers, (2:1HE) interstratified, and illites. All had very high levels of exchangeable Al (2.20 to $13 \text{ cmol}_c \text{ kg}^{-1}$) and very low sum (0.13 to $2.56 \text{ cmol}_c \text{ kg}^{-1}$) and base saturation (3 to 20%). For soils derived from the lava flow rocks of the Serra Geral Formation (basalt and rhyodacites), the mineralogy is generally similar to soils derived from sedimentary rocks, but with lower crystallinity of kaolinites, lower exchangeable Al contents (1.01 to $2.65 \text{ cmol}_c \text{ kg}^{-1}$) and with slightly higher sum (0.13 a $1.02 \text{ cmol}_c \text{ kg}^{-1}$) and base saturation (3 to 13%) values (Teske et al. 2013; Ferreira, 2013; Almeida et al., 2019).

Soils derived from syenite and phonolite studied by Ferreira (2013) had similar mineralogy and chemical properties to those developed from basalt and rhyodacites; on the other hand, the ultrabasic olivine melilithite derivatives stood out for their high natural fertility and mineralogy dominated by vermiculites, but they occupy an inexpressive area in the regional context. However, this suggests that rocks of more basic constitution (alkaline, basic, and ultrabasic), occurring in the Alkaline Complex, can originate more fertile soils compared to those originated from tephrite and nepheline syenite rocks, which in their chemical composition may contain lows contents, mainly of calcium and magnesium oxides.

Several occurrences of phonolites, syenites, and nepheline syenites are currently being identified in prospecting work to use these materials as a potential source of nutrients, in the form of rock powders or soil remineralizers, to supply mainly potassium for plants in alternatives processes for agricultural production (Martins et al., 2015; Ribeiro, 2018). Also, other rocks, not yet properly mapped and classified, have been identified in preliminary field studies, such as trachytes, tephrites, volcanic, and kimberlite breccias, of which details

of the chemical and mineralogical composition, as well as the characteristics of the soils derived from them are unknown.

Due to the lack of information on the chemical and mineralogical composition of soils developed from rocks of the Lages Alkaline Complex (Ferreira, 2013), this study aimed to evaluate five soil profiles developed from different rocks of the Complex mentioned above, with alkaline, basic, and intermediate, seeking to characterize in detail the aspects of its genesis, chemical properties, and mineralogical composition, as well as proceeding with the classification of these soils by the Brazilian Classification System (Santos et al., 2018)

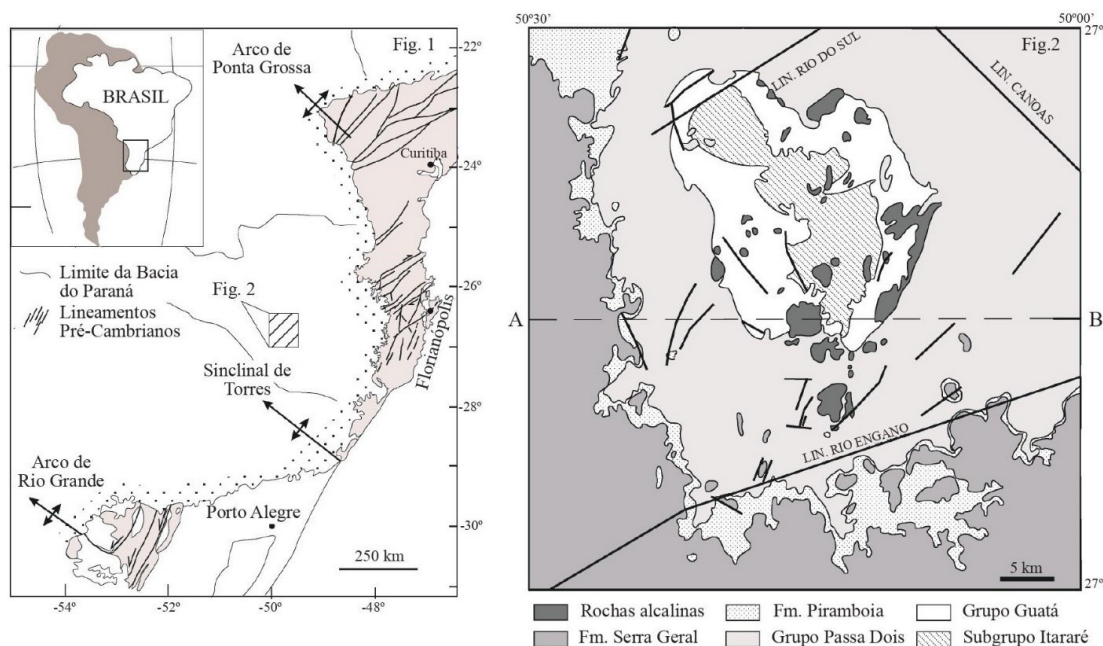
Materials and Methods

According to Köppen's classification, the climate of the Lages Plateau region, located in Santa Catarina, is of the Cfb type, humid mesothermal, with cold winter and mild summer, with an average temperature of 16°C and average annual rainfall of 1,500 mm. The altitude ranges from 850 to 950m. The region comprises mixed ombrophilous forest (pine forests), and the phytogeographic classification is of general grasslands associated with subtropical subdeciduous forest and araucaria forest (Santa Catarina, 2015).

The study was carried out with samples of five soil profiles, all developed from rocks of the Lages Alkaline Complex (Nepheline syenite –P1; Kimberlites - P2 and P3); Trachyte – P4, and; Phonolite – P5). The formation comprises alkaline and ultrabasic rocks resulting from late magmatism occurring around 75 Ma BP (Machado et al., 2012). The outcrops of the rocks occur as intrusions amid the Gondwana sedimentary rocks in the vicinity of the Serra Geral Formation escarpments. Also called Domo, or Alkaline District of Lages, this corresponds to a stratigraphic window, where the oldest geological occurrences outcrop at levels similar to the alkaline and ultrabasic, younger ones (Scheibe, 1986; Machado et al., 2012). Such rocks occupy approximately 50 km² but are dispersed over a larger area, approximately 1200 km² (Figure 1). Profiles P1, P2, and P5 were in the lower third of the slope in undulating relief; the P3 profile on top of elevation in soft undulating relief; and the P4 profile in the middle third of the slope in undulating relief.

These soil profiles were selected based on different lithologies that occur in the Lages Alkaline Complex, which may have influenced the genesis and properties of the different soils. The identification of rocks in the field and the respective observation sites were made based on the existing geological literature on the region, notably on the maps prepared by Scheibe (1986), Machado et al. (2012) and crossing with approximate geographic coordinates.

A general and morphological description was made for each profile, and samples were collected from the horizons and the underlying rock, carried out in ravines in road cuts, as described in Santos et al. (2015). All environments were under natural vegetation conditions. Afterward, the soil sample collection (deformed) from each horizon identified in the profile was carried out to subsequently carry out chemical,



Source: Machado et al. (2012).

Figure 1. Geological map of the alkaline rocks of the Lages Alkaline Complex, SC.

physical, and mineralogical analyses of the soils. Samples of the underlying rocks were also collected for elemental chemical analyses.

After the collections, the soil samples were dried in a forced circulation oven at 60 °C for 48 hours, chipped with a wooden roller, and sieved in a 2 mm mesh to separate the air-dried fine earth (ADFE). At ADFE, physical, chemical, and mineralogical analyses were performed.

Physical analyses were performed as described in Embrapa (2017), with some adaptations. In summary, 50 g of soil were weighed in a 150 mL snap-cap, then two acrylic pellets and 10 mL of 1 mol L⁻¹ and 75 mL of distilled water were added. Afterward, the samples were stirred on a horizontal stirrer for 4 h at 140 rpm. After this procedure, the samples were washed with fine jets over a 0.053 mm sieve to separate the silt and clay from the sand fraction, quantified by weighing after drying at 105 °C.

To quantify the clay, the Bouyoucus densimeter method (Embrapa, 1979) was used. The sand was quantified gravimetrically after drying at 105 °C, and silt was calculated by difference.

In ADFE, the pH in water and in a KCl 1 mol L⁻¹ solution was determined by potentiometry in a 1:1 ratio. The exchangeable Ca²⁺, Mg²⁺ e Al³⁺ contents were extracted with a 1 mol L⁻¹ KCl solution. Al³⁺ was quantified by neutralization titration with 0.025 mol L⁻¹ NaOH, using phenolphthalein (2%) as an indicator. Ca²⁺ and Mg²⁺ were determined by atomic absorption spectrophotometry (model: AAnalyst 200 – Perkin Elmer), using lanthanum oxide to control interferences. H+Al was extracted with a 0.5 mol L⁻¹ calcium acetate solution buffered at pH 7.0 and quantified by neutralization titration. K⁺ and Na⁺, extracted by ammonium acetate solution, were quantified by flame photometry. All analyses were performed according to

the criteria established in the Soil Analysis Methods Manual (Embrapa, 2017). The total carbon in the soil samples was determined in a Total Carbon Analyzer (TOC) equipment.

Mineralogical analyzes were carried out in the clay fraction, in 2 representative horizons of each profile (P1: horizons B and C1; P2: BAi and C; P3: Bi and Cr; P4: Bt1 and BC e; P5: Bi and C2), by X-ray diffraction. For this, the silt and clay from the particle size analysis were separated by successive decantations based on Stokes' Law (Jackson, 1965). Next, the clay samples were saturated with KCl 1 mol L⁻¹ and MgCl₂ 1 mol L⁻¹ solutions separately. Subsequently, excess salts were eliminated using dialysis bags, submerged in distilled water (DW) with previously measured electrical conductivity (EC). The water change was performed every 3 h until the DW, in which the samples were submerged, reached the initially measured EC (Almeida et al., 2019).

The samples saturated with K⁺ and Mg²⁺ were analyzed in the form of oriented aggregates, and those of K⁺ were read after drying at room temperature, then heated in an oven to read the samples at 110 °C and in the muffle to read the samples at the temperatures of 350 and 550 °C. Those with Mg²⁺ were subjected to air drying (25 °C) and saturation with ethylene glycol vapor (ETG) at 60 °C for 48 h.

Mineralogical analyses were performed using a Philips X-ray diffractometer, model PW 3710, equipped with a copper tube, with a $\theta/2\theta$ compensation angle, vertical goniometer, and a graphite monochromator, with angular variation from 3 to 42 for the samples of oriented clay, with an angular velocity of 0.02 ° 2 θ /s, in step mode, with a reading time of 0.5 seconds per step. To identify the minerals, the parameters of Brown & Brindley (1980), Whittig & Allardice (1986) were used, as well as specific literature indicated in the text, when applicable. For semi-quantification of clay minerals, the

Panalytical X'Pert High Score Plus software (3.0) was used, measuring the relative areas of the main reflections of each identified mineral.

Elemental analyses were performed using the X-ray fluorescence technique, with the elements expressed as oxides. Such analyses were carried out in the clay fraction powder of the selected horizons of each soil profile, previously mentioned, as well as in the rock powder. These analyzes were used to quantify the oxides in the source material of each soil and in the clay fraction to calculate the Ki index, obtained by the formula: $Ki = (SiO_2/Al_2O_3) \times 1.7$ (Silva, 2020).

The clay fraction and rock samples were ground in an agate mortar until the entire content passed through a 0.053 mm sieve and homogenized. Then, the samples were placed in a cylindrical sampler, with the bottom closed with a polyester film ("mylar") of 3.6 μm . A compact X-ray fluorescence (XRF) spectrometry equipment, model Epilson 3 from Panalytical was used. To calibrate the equipment, the OMNIAN application (Epsilon software package) was used, which are standard samples in the format of cast pellets, analyzing 24 elements simultaneously.

Results and Discussion

Chemical composition of rocks

The SiO_2 content evidenced in the phonolite rock powder (P5) was within the expected range (52 to 65%), with a value of 55.11%, placing this rock in the intermediate group. The CaO and MgO contents for this rock are low, totaling 1.86%. On the other hand, those of Na_2O and K_2O are high, as well as those of aluminum. The same interpretation performed for the phonolite can be admitted for the nepheline syenite (P1), probably because it is the intrusive correspondent of the phonolite. However, it should be noted that the SiO_2 contents (51.20%) (Table 1) were slightly below those commonly reported for this rock, classified as intermediate. Then, these results confirm that based on the general composition, the classification of these two rocks as alkaline. Thus, the composition of these two rocks is compatible with the values normally referenced in studies carried out by Scheibe (1986), Le Maitre (2002), and Motoki et al. (2013).

For P2, the elemental composition of the rock collected in this profile shows high SiO_2 content (56.67%) (intermediate group), very low levels of magnesium oxide (0.4%), chromium and strontium, and abnormal aluminum content (Table 1).

Nevertheless, it had morphological similarity to kimberlites or kimberlite breccias. Therefore, given the elemental composition observed, it was classified as a volcanic breccia, as also evidenced by Scheibe (1986).

Regarding the rock collected in P3, the elemental composition shows a relatively high content of SiO_2 (47%), high contents of CaO and MgO (sum of these oxides around 22%), titanium, chromium, strontium, and nickel. However, the CaO content exceeded that of MgO (Table 1). Although there was a great morphological similarity between this rock and a kimberlite, it differed from this rock by the higher contents of SiO_2 and MgO and the normal pattern of most kimberlites studied by Costa et al. (1997). Given these results, the rock was classified as a kimberlite breccia.

The chemical composition of the rock collected in P4 has a SiO_2 content of around 49%. Therefore, it belongs to the basic group. Furthermore, this rock has high contents of K, Ca, Al, and P_2O_5 oxides, relatively high Na, and low trace elements. Furthermore, based on the chemical composition data of samples collected in the Lages region by Scheibe (1986) and Aumond & Scheibe (1996), the selected rock would fit better as a porphyritic nepheline syenite. However, for the first author, this rock would be located in the field of phonolitic tephrites. The elementary composition of a tephrite analyzed by Comin-Chiaramonti et al. (2001) in the region of Lages is very similar to the composition of the studied rock.

Similar results regarding the classification of rocks collected in the Lages Alkaline Complex, evidenced in this study, were also verified by Scheibe (1986) and Roldan (2007). After evaluating some rocks from the aforementioned Complex, these authors separated them into leucocratic alkaline (phonolites, porphyritic phonolites, analcime, trachytes, and porphyritic nephelines syenites), alkaline ultrabasic (olivine melilithites and lamprophyres), carbonatites, kimberlites, and volcanic breccias. In addition, the elemental composition of the rocks based on SiO_2 contents are in accordance with those reported by Leinz & Amaral (1985) and Ferreira (2013), although this study found higher levels of basic oxides when compared with those obtained by the last author.

Morphology, physical, and chemical properties of soils

All soils studied, except for P1, showed high levels of carbon in the superficial horizons (Table 2), forming relatively thick and dark A horizons (P1: 49 cm; P2: 40 cm; P3: 25 cm; P4: 60 cm and P5: 38 cm), which can be attributed to the cold and

Table 1. Elements in the form of oxides, by X-Ray fluorescence, of the powder of the rocks of profiles P1 (nepheline syenite), P2 (volcanic breccia), P3 (kimberlite breccia), P4 (trachyte), and P5 (phonolite).

Sample	Na_2O	K_2O	CaO	MgO	Al_2O_3	SiO_2	P_2O_5	TiO_2	Cr_2O_3	NiO	CuO	Sr
	(%)											
P1	2.47	5.08	2.98	1.78	28.48	51.20	0.61	0.64	0.02	0.006	-	0.12
P2	4.30	8.03	1.74	0.41	23.34	56.67	0.40	0.25	0.01	-	-	0.05
P3	-	3.28	17.50	4.74	11.53	47.27	0.99	1.72	0.06	0.06	0.008	0.23
P4	2.47	5.27	6.71	1.33	22.13	48.73	1.30	1.46	-	-	0.003	0.36
P5	-	6.7	1.44	0.42	28.66	55.11	0.51	0.59	0.01	0.001	-	0.04

- not detected.

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ARTIGO PUBLICADO REVISTA ACTA SCIENTIARUM AGRONOMY (ONLINE)

ANO: 2022

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* Evento de Classificação:
CLASSIFICAÇÕES DE PERIÓDICOS QUADRIÊNIO 2017-2020

Área de Avaliação:
☒ CIÊNCIAS AGRÁRIAS I

ISSN:
☒ 1807-8621

Título:
☐

Classificação:
☐ -- SELECIONE --

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Periódicos

ISSN	Título	Área com publicação no quadriênio	Classificação	Área mãe
1807-8621	ACTA SCIENTIARUM. AGRONOMY (ONLINE)	CIÊNCIAS AGRÁRIAS I	A4	CIÊNCIAS AGRÁRIAS I



Chemical composition of soybean seeds subjected to fertilization with rock dusts

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ABSTRACT. Rock dusts in association with organic sources have been used in agriculture to improve soil chemical characteristics and food composition. In this context, this study aimed to evaluate the effect of the rock dusts siltstone, tephrite, and olivine melilitite pure or combined and associated or not with cattle manure on the chemical composition of soybean seeds of the cultivar BRS 232 grown on a Cambisol and Nitisol subjected to these treatments and conventional fertilization. For this, an experiment was carried out with pots in a greenhouse using a Cambisol and Nitisol to cultivate the soybean from January to May 2019 until seed production. The seeds were harvested, ground, and sieved through a 1 mm mesh sieve to determine the nutrient, phytate, and crude protein contents. The effects of the rock dusts on the chemical composition of seeds were directly related to the chemical characteristics of the evaluated soils. The tested rock dusts had a more pronounced response in the Cambisol (poorer soil) than in the Nitisol (more fertile). The contents and accumulation of nutrients in the seeds, as well as the total dry matter production in both soils subjected to the treatments with the rock dusts olivine melilitite and siltstone + olivine melilitite, pure and combined with manure, were similar and, in some cases, higher than the treatments subjected to conventional fertilization. The combination of manure and rock dusts provided an increase in the concentration of nutrients in both soils, resulting in their higher availability for plants and directly reflecting on the chemical composition of seeds.

Keywords: siltstone; tephrite; olivine melilitite; siltstone + olivine melilitite; cattle manure.

Received on April 27, 2020.

Accepted on July 24, 2020.

Introduction

The soybean grown in Brazil and several parts of the world is used to obtain oil, protein, and other by-products necessary to meet both human and animal demand. For this, soil management practices need to be carried out to meet the requirements of plants at different stages of development, mainly during seed production, directly reflecting on its chemical composition.

Most of the Brazilian territory is composed of weathered and acidic soils, with low cation exchange capacity (CEC), high Al^{3+} and Mn contents, low Ca^{2+} , Mg^{2+} , K^+ , the sum of bases, and base saturation contents, and P as the most limiting nutrient, which are undesirable characteristics for good development of the main crops (Yang, Zhang, Cong, Wang, & Shi, 2013; Tandzi, Mutengwa, Ngonkeu, & Gracen, 2018). Thus, the use of high amounts of soil acidity correctives and soluble fertilizers is necessary to achieve good productivity in these soils, resulting in an increased dependence on external inputs, high production costs for farmers, and soil and water pollution when misused (Brito, Batista, Moreira, Morais, & Silva, 2019).

In this context, rock dusts may represent an alternative option to soluble mineral fertilizers, as they are sources of various nutrients and can also maintain a fraction of weatherable minerals in the soil, serving as a reserve of mineral nutrients. Ground rocks applied to soils can provide a high amount of nutrients lost during the weathering and leaching processes or soil management practices (Brito et al., 2019). The supply of nutrients to the soil using rock dusts occurs more slowly than industrial soluble fertilizers, but it occurs continuously, usually releasing only the amount of nutrients that the plant requires at each stage of development until its cycle is complete (Cola & Simão, 2012; Toscani & Campos, 2017).

Although the release of these nutrients depends on the chemical composition, particle size, mineralogy, rock origin, and soil pH, among other factors, studies developed by Ferreira, Almeida, and Mafra (2009), Melo, Uchôa, Dias, and Barbosa (2012), and Duarte, Mafra, Foresti, Piccolla, and Almeida (2013) showed that the

productivity of plants, mainly those of long cycles, subjected to the use of rock dust can have a satisfactory performance compared to plants subjected to the conventional fertilization. This behavior is probably due to the release of macro- and micronutrients contained in the rock dusts and their ability to increase the sum of bases and soil pH. This performance can be enhanced by mixing these products with organic compounds (manure, sewage sludge, and crop residues) to obtain nutrient-enriched materials, which are required for plant production (dry matter and grain production), besides accelerating the rock dissolution processes and supplying N to the soils (Florentino et al., 2017; Miranda et al., 2018).

Few studies have evaluated the chemical composition of the seeds produced in systems using the rock dusts siltstone, tephrite, and olivine melilitite, as well as the mixture of siltstone + olivine melilitite (Santos, Oliveira, Oliveira, & Oliveira, 2010), although their benefits when associated or not with organic sources are known to provide soils with improved chemical characteristics and plant productivity (Meena & Biswas, 2014; Wolschick, Schuch, Gerber, & Sartoretto, 2016).

In this sense, it is important to evaluate whether the nutrients present in the seeds produced by plants subjected to treatments with these rock dusts are within the ranges considered appropriate in the literature (Tedesco, Gianello, Bissani, Bohnen, & Volkweiss, 1995; Vargas et al., 2018). This evaluation is essential because seeds with a good nutrient supply can originate vigorous plants. Phosphorus, as well as its inorganic and phytate fractions, is among the nutrients with an essential role in the germination process and potential to form and establish seedlings (Coelho, Santos, Tsai, & Vitorello, 2002; Marin, Bahry, Nardino, & Zimmer, 2015).

Moreover, poor soil management associated with excessive P fertilization may increase the risk of losing this nutrient in the surface and groundwater, leading to water eutrophication and phytate accumulation in the seeds (Taliman, Dong, Echigo, Raboy, & Saneoka, 2019). Phytate is the main form of P storage in the seeds and its presence under adequate amounts leads to a good initial development of soybean seedlings, providing plants more vigorous and with higher productive potential (Corrêa, Mauad, & Rosolem, 2004; Marin et al., 2015). However, according to Martinez, Martinez, Souza, and Brazaca (2011) and Deak and Johnson (2007), high phytate content accumulated in the seeds is considered an anti-nutritional factor that contributes to reducing the digestibility of proteins by humans and animals (Martinez et al., 2011).

In this sense, the use of rock dusts can be an interesting alternative for the good supply of this nutrient to plants, as its release is slow but continuous, nourishing the plants during their different growth stages and providing seeds with a good supply of nutrients.

Thus, this study aimed to evaluate the effect of the rock dusts siltstone, tephrite, olivine melilitite, and siltstone + olivine melilitite pure or combined and associated or not with cattle manure on the chemical composition of soybean seeds of the cultivar BRS 232 grown on two soils with different physicochemical characteristics subjected to these rock dusts and conventional fertilization.

Material and methods

The rock dusts tested in this study consisted of siltstone, tephrite, olivine melilitite, and the mixture (1:1) of siltstone + olivine melilitite. These products were collected in the municipality of Palmeira, Santa Catarina State, Brazil, being supplied by the company Dinamisa S.A. in the powder form with a particle diameter lower than 0.3 mm. The cured cattle manure was collected in a stable of animals destined for milk production.

The first step of the chemical analysis consisted of sieving the rock dusts through a 0.25 mm mesh sieve to obtain the filler particle size. Subsequently, the samples of each rock dust were ground in an agate mortar, sieved in a 0.053 mm mesh sieve, and analyzed as uncompressed powder. A total of 2 g of the ground samples were placed in a standard sample holder with the bottom covered with a 3.6 µm Mylar polyester film.

The equipment used for these analyses was a compact PANalytical Epsilon 3 energy dispersible x-ray spectrometer (EDX) set for a maximum high-voltage power generator of 9000 mW, voltage from 4 to 30 kV, and current intensity from 1 to 1000 µA, and with rhodium anode (Rh) x-ray tubes, a 50 µm thick beryllium window, and a Si-PIN diode detector, and data processing using a multichannel analyzer (MCA). The system used pressurized helium and a Peltier electronic thermoelectric air-cooling. The quantification of the elements was carried out through the creation of an application called Omnian, which is part of the software package Epsilon, calibrated from standard samples of molten inserts.

The following results were obtained from the analyses for each rock dust: i) siltstone (sedimentary rock present in the surroundings and intermingled of the outcrops of the olivine melilitite rock; Dome or Alkaline Complex of Lages) – CaO = 6.8%, MgO = 4.00%, P₂O₅ = 0.56%, K₂O = 3.38%, SiO₂ = 57%, and Al₂O₃ = 14.06%;

ii) tephrite (basic igneous rock; dome or Alkaline Complex of Lages) – CaO = 6.72%, MgO = 1.36%, P₂O₅ = 0.99%, K₂O = 5.70%, SiO₂ = 50%, and Al₂O₃ = 21.06%; iii) olivine melilitite (magmatic, ultrabasic magmatic rock; Dome or Alkaline Complex of Lages) – CaO = 15.28%, MgO = 14.91%, P₂O₅ = 1.55%, K₂O = 3.59%, SiO₂ = 37.7%, and Al₂O₃ = 8.22%; and iv) siltstone + olivine melilitite (Dome or Alkaline Complex of Lages) – CaO = 12%, MgO = 10.65%, P₂O₅ = 1.31%, K₂O = 3.70%, SiO₂ = 45%, and Al₂O₃ = 12.05%.

The cattle manure samples were dried in a greenhouse until constant weight, ground, and sieved through a 2 mm mesh sieve to quantify the Ca²⁺, Mg²⁺, P, and K contents (Tedesco et al., 1995). The total organic carbon and nitrogen contents were determined using a Total Carbon Analyzer. The chemical composition of the cattle manure was obtained as follows: Ca²⁺ = 0.20%, Mg²⁺ = 0.06%, P₂O₅ = 2.51%, K₂O = 0.77%, C = 24.73%, and N = 1.053%.

To test the potential of rock dusts, pure or combined with cattle manure, was carried out using soil samples collected at a depth of 0-20 cm from the surface horizon on the roadsides of the highways SC-114, located in Pinheiro Seco (Cambissolo Háplico Alumínico Típico, Cambisol), and BR-282, located in Amola Faca (Nitossolo Bruno Distroférrico Típico, Nitisol). Both soil sampling areas were located in the municipality of Lages and were not cultivated. The soil samples were air-dried in a greenhouse and sieved using a 4 mm mesh sieve. These samples passed through a 2 mm mesh sieve for the chemical analysis before incubation.

After sample processing, the pH in water and SMP were determined by potentiometry at the 1:1 soil to solution ratio. P and K were extracted using the Mehlich-1 solution and quantified by colorimetry (Murphy & Riley, 1962) and flame photometry (Tedesco et al., 1995), respectively. These analyses were performed to calculate the need for limestone and soil fertilization in conventional treatment, respectively. The field capacity was determined as described by Casaroli and van Lier (2008). The Cambisol presented the following chemical characteristics before incubation: pH in water = 4.64, pH SMP = 4.93, and P = 2.91 mg kg⁻¹, and K = 0.31 cmol_c kg⁻¹. In addition, the Nitisol presented the following chemical characteristics: pH in water = 5.23, pH SMP = 5.3, and P = 1.21 mg kg⁻¹, and K = 0.13 cmol_c kg⁻¹.

Soil incubation was carried out in a greenhouse. Treatments consisted of application of three doses of pure dusts from siltstone and tephrite rocks (2.5, 5, and 10 t ha⁻¹); and two doses of: a) a combination of the aforementioned rocks with cattle manure (5 and 10 t ha⁻¹), b) pure dusts from olivine melilitite and siltstone + olivine melilitite rocks (5 and 10 t ha⁻¹), c) a mixture of siltstone + olivine melilitite rock dust with cattle manure (5 and 10 t ha⁻¹), and pure cattle manure. The amount of manure corresponded to 5 t ha⁻¹ of dry matter.

The amounts of rock dusts, pure or combined with manure, used in the treatments totaled 5 and 10 t ha⁻¹ in their respective doses. Moreover, no source from conventional fertilizers was applied to the treatments that received the rock dusts, pure or combined with manure. Besides these treatments, a control was added to each soil, that is, under natural conditions without application of limestone and soluble fertilizers. The soils, in the corresponding treatments, were corrected using limestone (Total Neutralization Relative Power [PRNT in Portuguese] = 90%, CaO = 29%, and MgO = 19%, filler grain size after sieving through a 0.25 mm mesh) to raise pH to 5.5. In this step, limestone was added only to the corresponding treatments.

Each treatment was carried out with four replications, and the doses were homogenized in samples of 20 kg of soil (dry basis). Distilled water was applied to increase the moisture to 80% of the field capacity. Subsequently, the samples were placed in 50 L plastic bags protected by a raffia bag. The bags were opened every 10 days to allow the homogenization of the samples and the moisture was corrected with deionized water when necessary. This step was carried out for 45 days.

An amount of 140 g of moist soil was collected from each treatment at the end of the incubation period. The samples were dried in an oven at 60°C for 24 hours to obtain the equivalent to the air-dried fine earth, which was used to determine the pH in water and 0.01 mol L⁻¹ CaCl₂ by potentiometry at the 1:1 soil to solution ratio. The exchangeable Ca²⁺, Mg²⁺, and Al³⁺ contents were determined using the 1:10 soil to solution ratio after extraction with 1 mol L⁻¹ KCl solution (Empresa Brasileira de Pesquisa Agropecuária [Embrapa], 2017). The Ca²⁺ and Mg²⁺ quantification were performed by atomic absorption spectrophotometry (Tedesco et al., 1995), while Al³⁺ was determined by titration with 0.02 mol L⁻¹ NaOH in 20 mL of the extract. The P, K, and Na contents were extracted using the Mehlich-1 solution (Tedesco et al., 1995), the former being quantified by colorimetry (Murphy & Riley, 1962) and the two latter by flame photometry. The H+Al content was obtained after extraction with calcium acetate at pH 7 (Embrapa, 2017). The other soil chemical attributes were calculated from the results of these analyses.

After incubation, the samples of 20 kg of soil (dry basis) of each treatment, representing the four replications, were homogenized and only the samples that received limestone were fertilized with P and K,

using triple superphosphate and KCl as sources, respectively. The calculations for P and K fertilization were performed using the 20 kg of soil (dry basis). Nitrogen was not used because the soybean seeds were inoculated.

The P and K fertilization followed the recommendations of the *Comissão de Química e Fertilidade do Solo* of the States of Rio Grande do Sul and Santa Catarina (CQFS RS/SC, 2016) for the soybean crop, taking into account the results of the soil chemical analysis before incubation. The calculation of the P and K fertilization aimed at the productivity of 3 t ha⁻¹ of grains.

The experiment was carried out for 127 days from January to May 2019 in a greenhouse using 7 L pots filled with 5 kg of soil (dry basis). Eight soybean seeds of the cultivar BRS 232, without pre-germination, were sown in each pot (experimental unit). Seedlings were thinned to one per pot until the end of the cycle, that is, one seedling was thinned every 10 days, totaling four thinning procedures. The pots were weighed daily aiming at maintaining soil moisture close to 80% of the filed capacity, being corrected with deionized water whenever necessary. The weights corresponding to the plant growth were added to replace the water. The experimental design was completely randomized with 19 treatments and four replications for the two soils (two experiments with 76 experimental units).

The evaluation of the plant's nutritional status consisted of collecting two index leaves from each treatment at the phenological stage of full flowering (R2), as recommended by *Empresa Brasileira de Pesquisa Agropecuária* (Embrapa, 2009). Subsequently, the leaves were packed in paper bags and dried in a forced-air circulation oven at 60°C until constant weight. The weights of each sample were added to the shoot dry matter (SDM) of each experimental unit after the experiment was collected. The samples were then ground and digested in a digester block (Tedesco et al., 1995) for later chemical analysis. The Ca, Mg, K, and N contents in the index leaves were determined according to the procedures described by Tedesco et al. (1995), while P contents were determined as described by Murphy and Riley (1962). Selenium was not used for sample digestion.

Pods, leaves, and stems were collected at the end of the experimental period from each experimental unit, and the sample processing was carried out as previously described. The roots were manually separated from the soil and washed under running water, receiving the same treatment as the other plant parts. After separating the roots, the soil samples were homogenized and 140 g of moist soil was collected from each experimental unit for chemical analysis. The sample processing and analyses carried out for the soil chemical characterization followed the same procedure described after the incubation period (45 days) of the soil samples in the greenhouse.

The production per plant in each experimental unit considered the following characteristics: i) number of pods and seeds; ii) seed weight; iii) thousand-seed weight; iv) shoot (SDM), root (RDM) and total dry matter (TDM); and v) harvest dry matter. In this study, only TDM was presented and discussed.

The nutrient content accumulated in the grains was calculated from these results and the seed weight, using the equation adapted from Cunha, Almeida, Souza, and Ernani (2019).

$$NAS (mg) = SW (mg) \times \% \text{ nutrient in the seed} / 100 \quad (1)$$

where:

N_{AS} represents the nutrients accumulated in the seeds and SW is the seed weight.

After obtaining the data regarding seed production, the samples were milled and sieved in a 1 mm mesh sieve to determine the Ca, Mg, N, P (total phosphorus), and K contents, as described for the index leaves. Crude protein (CP) was obtained by multiplying the total N (N%) by the factor 6.25 (Association of Official Analytical Chemists [AOAC], 1995).

Inorganic phosphorus (Pi) and phytate were also determined. The Pi extraction was performed according to the method described by Raboy and Dickinson (1984), being determined by colorimetry according to the method described in Chen and Charalampous (1966). Phytate was determined according to the procedures described by Latta and Eskin (1980). However, some adaptations of these methods have been made.

For extraction and subsequent quantification of Pi, the extracts were obtained by weighing 0.1 g of each sample in 15 mL Falcon tubes. Subsequently, 4 mL of the solution composed of the mixture of 12.5% trichloroacetic acid (12.5 g in a 100 mL volumetric flask) and 0.025 mol L⁻¹ MgCl₂ were added. That is, the volume of the volumetric flask was completed with this solution. The samples were stirred at 120 rpm for five minutes on a horizontal stirrer and centrifuged for 1 hour and 30 min to reach the g force (10,000 g for 10 min.), as indicated by Raboy and Dickinson (1984). Then, the samples were filtered through filters paper (quantitative filter with an 11 cm diameter and medium filtration). This procedure was performed twice with the same amounts of solution and sample (remaining residue from the previous extraction), totaling 8 mL of

extract at the end. An aliquot of 4 mL was taken from this extract and diluted to 12.6 mL of ultrapure water. Then, a mix composed of a mixture of 10% ascorbic acid and 0.42% ammonium molybdate (1:6) was dissolved in 0.01 N H₂SO₄ (stored in the dark). An aliquot of 2.1 mL of the mix + 0.9 mL of the extracts was added to plastic cups. The concentrations in the curve, prepared from an 80-ppm stock solution, were 0, 0.5, 1.0, 1.5, 2.0, 2.5, and 3.0 ppm in 100 mL flasks. The volume was made up with a 0.01 N H₂SO₄ solution. After preparation, both samples and curve points were immediately placed in a previously heated oven at 45°C for 20 min. The readings were performed on a UV-visible spectrometer at a wavelength of 820 nm. The absorbance results obtained at each point on the curve are shown in Table 1.

The calculation procedures for obtaining Pi in the seed samples evaluated in the different treatments are shown as follows.

- 1- Dilution factor (DF1) = mix volume (mL) + extract volume (mL)/extract volume (mL).
- 2- Real P = Curve point/DF1.
- 3- Slope: Real P × Absorbance.

Table 1. Curve points and their respective absorbance values.

Curve point	Absorbance
ppm	
0	0.000
0.5	0.136
1.0	0.262
1.5	0.375
2.0	0.529
2.5	0.675
3.0	0.798

For the Pi calculations a few steps were performed, namely.

- 1- DF1 = solution volume (4mL)/Sample weight.
- 2- DF2 = solution volume (4mL)/Sample weight.
- 3- DF3 = solution volume (4 mL) + 12.46 mL/solution volume (4 mL).
- 4- Considering the readings and dilutions = Sample absorbance (SA; ppm) × FD1 × FD2 × FD3.
- 5- Considering the Blank Test = Result found in item 4 - BT;
- 6- Pi (%) = (Result obtained in item 5/Slope of the line)/10000.

The extraction and subsequent quantification of phytate in soybean seeds were performed as follows. In summary, weighing of 1.0 g of the Dowex 200-400 anionic mesh resin (Sigma) in 50 mL Falcon tubes. The resin was hydrated with ultrapure water for 1 h. The water was discarded after this period and 8 mL of 0.7 mol L⁻¹ NaCl was added with subsequent stirring at 150 rpm for 5 min and resting for 1 h for saturation with Cl⁻. The NaCl was discarded. Removing excess salt from the resin was performed with the addition of 8 mL of ultrapure water, stirring the samples manually, and letting them stand for 1 h. This procedure was performed twice.

Phytate extraction, 0.25 g of each sample was weighed in 15-mL Falcon tubes; 8 mL of 2.4% HCl was added to them. The samples were stirred at 150 rpm on a horizontal stirrer for 3 h and centrifuged at 2000 rpm for 15 min. Then, we proceed to filtering and subsequent conditioning of the extracts in 15 mL Falcon tubes. To phytate recovery, 1 g of resin (obtained by pipetting a volume of 2.5 mL in 50-mL Falcon tubes, after washing the excess salt) plus 3 mL of the extract (obtained after phytate extraction) plus 8 mL of ultrapure water. The samples were then stirred at 150 rpm for 1 h and centrifuged at 2000 rpm for 15 min. Removal of impurities (including Pi): the supernatant obtained in the previous item was discarded and the resin of each sample (treatment) was washed with 0.07 mol L⁻¹ NaCl, as follows: 8 mL of the previous solution was added, stirring them at 120 rpm for 10 min followed by centrifugation at 2000 rpm for 15 min. Subsequently, the supernatant was discarded. To remove phytate retained in the resin, 8 mL of the 0.7 mol L⁻¹ NaCl solution was added and the samples were stirred for 30 min and centrifuged at 2000 rpm for 10 min. A volume close to 8 mL of each extract was pipetted into 50-mL Falcon tubes.

Wade's solution was prepared (0.03 g of ferric chloride plus 0.3 g of 5-sulfosalicylic acid dihydrate in 100 mL of ultrapure water). Before readings: 3 mL (the final sample volume must have this volume, regardless of the dilution performed and the addition of Wade's solution) was pipetted from the extract obtained in item 9 into 15 mL Falcon tubes and 2 mL of Wade's solution was added. Then, the samples were centrifuged at 2000 rpm for 15 min for the precipitation of iron phytate.

Preparation of the curve: preparation of a 300-ppm stock solution (0.03 g of sodium phytate (phytic acid)) in a 100-mL volumetric flask. Then, 35 mL of the previous solution was pipetted in a 50-mL flask to obtain the 210-ppm stock solution. The curve points were then obtained, with final concentrations of 0, 35, 70, 105, 140, 175, and 210-ppm, calculated for a final volume of 3 mL. The volume for preparing these concentrations was pipetted into 1 mL Falcon tubes with the addition, respectively, of 3, 2.5, 2.0, 1.5, 1.0, 0.5, and 0 mL of ultrapure water and 2 mL of the solution prepared in item 11 (total of 5 mL). Wade's solution was added to the samples and curve points only to provide differences in the pink color with an increase in the phytate concentration, thus not being considered in the calculation to obtain the phytate in the samples.

Sample reading was carried out by pipetting 3 mL of the extract plus 2 mL of Wade's solution into 15-mL Falcon tubes, being then centrifuged at 2000 rpm for 15 min. Although the final sample volume was 5 mL, only 3 mL of each sample was pipetted into 50 mL plastic cups to perform the readings.

The reading of the curve points and the phytate quantification of each sample were performed on a UV-Visible spectrometer at a wavelength of 500 nm, using 3 mm cuvettes. The device was zeroed with ultrapure water before performing this last procedure. The absorbance results obtained at each point on the curve are shown in Table 2.

Table 2. Curve points and their respective absorbance values.

Curve point	Absorbance
ppm	
0	0.785
35	0.674
70	0.562
105	0.462
140	0.356
175	0.260
210	0.182

For the phytate calculations a few steps were performed, namely.

- 1- Calculation of the blank test (BT) = $(-343.84 \times \text{BT reading}) + 266.16$.
- 2- Reading of each sample (RSA) $\times$ Line equation = $(-343.84 \times \text{RSA reading}) + 266.16$.
- 3- RSA – BT reading value (obtained in item 1).
- 4- Quantity of phytate in 3 mL = $(\text{Result obtained in item 3} \times 3\text{mL})/1000$.
- 5- mg of phytate in 8 mL = $(\text{Result obtained in item 4} \times 8\text{ mL})/3$.
- 6- mg in the extract = $(\text{Result obtained in item 5} \times 8\text{ mL})/3$.
- 7- Phytate (%) = $[\text{Result obtained in item 6}/\text{sample weight (g)}]/1000 \times 100 \times 0.7144$.

The resin can be used three times. After use, after analysis, and after cleaning, it can be stored in a plastic container filled with water. It is recommended to pipette 2.5 mL to perform new analyses. Subsequently, a saturation with NaCl must be performed, and so on.

The data regarding the nutrient contents and their accumulation in TDM, as well as phytate, CP, and the results of the soil chemical analysis, were subjected to analysis of variance (F-test) using the software Sisvar 5.6 (Ferreira, 2014). The Scott-Knott test at the 5% probability error was applied when a significant effect was observed. Moreover, the normality and homoscedasticity tests were performed and all the conditions were met.

Results and discussion

Soil chemical attributes

In general, the addition of limestone and rock dusts, associated or not with cattle manure, to both soils increased the pH values in water, Ca^{2+} and Mg^{2+} contents (some treatments presented Mg contents lower than the control in the Cambisol), the sum of bases, base saturation, and P contents, with a concomitant reduction in the contents of H+Al, exchangeable Al^{3+} , and Al saturation compared to the control treatment, regardless of the used dose. On the other hand, K and Na contents in most treatments were similar and even lower than the values observed in the control. Even with an increase in the values of pH in water and the contents of Mg^{2+} , K^+ , Na^+ , and base saturation compared to the control, little variation was also observed in their absolute values, regardless of the products, doses, and soils, with or without statistical difference between the applied doses and between treatments (Table 3).

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Potential of olivine melilitite as a soil remineralizer according to particle size and rates

Abstract – The objective of this work was to evaluate the potential of olivine melilitite rock powder, in two particle sizes and in increasing rates, to improve the chemical properties of the soil and the growth and nutrient accumulation of soybean (*Glycine max*) and sorghum (*Sorghum bicolor*) plants. The treatments consisted of three rates of the rock powder, equivalent to 2.5, 5.0, and 10 Mg ha⁻¹, in the powder and filler particle sizes of the commercial product. Physicochemical and mineralogical analyses were carried out using, as a basis, the Brazilian normative ruling on rock powder as a soil remineralizer. The soybean and sorghum plants were cultivated for 60 and 45 days, respectively, in a greenhouse on a Humic Dystrudept and a Typic Hapludult. The olivine melilitite rock powder applied in the tested increasing rates and two particle sizes improved soil chemical properties and promoted plant growth. However, the filler particle size is more efficient than that of the powder to improve soil chemical properties and plant growth and nutrient accumulation.

Index terms: agromineral, natural inputs, plant nutrition, rock powder, soil fertility.

Potencial do pó da rocha olivina melilitito como remineralizador de solos de acordo com tamanho de partículas e doses

Resumo – O objetivo deste trabalho foi avaliar o potencial de pó da rocha olivina melilitito, em duas granulometrias e em doses crescentes, para melhorar as características químicas do solo e o crescimento e o acúmulo de nutrientes em plantas de soja (*Glycine max*) e sorgo (*Sorghum bicolor*). Os tratamentos consistiram de três doses de olivina melilitito, equivalentes a 2,5, 5,0 e 10 Mg ha⁻¹, nas granulometrias de pó e filler do produto comercial. Foram realizadas análises físico-químicas e mineralógicas, tendo-se utilizado, como base, instrução normativa para pó de rocha como remineralizador de solos. As plantas de soja e sorgo foram cultivadas por 60 e 45 dias, respectivamente, em casa de vegetação, em Cambissolo Háplico Aluminico e Argissolo Vermelho Distrófico sombrio. O pó da rocha olivina melilitito aplicado nas doses crescentes e nas duas granulometrias testadas melhorou as características químicas dos dois solos estudados e promoveu o desenvolvimento das plantas. No entanto, a granulometria filler é mais eficiente que a do pó para melhorar as características químicas dos solos e o desenvolvimento e o acúmulo de nutrientes nas plantas.

Termos para indexação: agromineral, insumos naturais, nutrição de plantas, rochagem, fertilidade do solo.

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Received
April 27, 2019

Accepted
December 30, 2021

How to cite
ALMEIDA, J.A. de; CUNHA, G.O. de M.
HEBERLE, D.A.; MAFRA, Á.L. Potential
of olivine melilitite as a soil remineralizer
according to particle size and rates. **Pesquisa
Agropecuária Brasileira**, v.57, e01445, 2022.
DOI: <https://doi.org/10.1590/S1678-3921.pab2022.v57.01445>.

Introduction

Although Brazil stands out in agricultural production worldwide due to the great size of its territory and high crop yields associated to favorable climatic conditions, most soils of the country are highly weathered and acidic and have a low nutrient availability (Barbosa et al., 2017; Rabel et al., 2018).

To improve the production capacity of these soils, in conventional agriculture, high amounts of soluble fertilizers are used, which is linked to a great dependency on external sources of raw materials (Manning & Theodoro, 2020). An alternative for the country to increase its autonomy in supplying nutrients for its own crops is the application of rock powders, whose effects, however, still need to be further researched for the adequate use of each rock for this purpose in agriculture (Manning, 2015; Silva et al., 2017; Beerling et al., 2018; Manning & Theodoro, 2020; Brito et al., 2019).

Researches in Brazil and abroad have shown that, when rock powders are applied to a soil as a source of nutrients, the agronomic responses are comparable to those obtained with soluble fertilizers (Manning et al., 2017; Zhang et al., 2018; Silva et al., 2019; Aguilera et al., 2020). Despite this positive result, there are important limitations to the wider use of rock powders, especially related to the scarcity of scientific studies evaluating their effects on improving soil quality and crop yield. Therefore, it is necessary to identify, according to their composition and solubility, the rocks that are most effective in each soil and crop condition, as well as the best products to be used, to improve soil chemical properties for a suitable plant development (Theodoro, 2017; Beerling et al., 2018; Manning & Theodoro, 2020). Another challenge is that, in Brazil, since the legislation for the use of rock powders is still recent, there are not yet adequate requirements for estimating the amount and rate of nutrient release from these rocks to plants.

Considering this scenario and the interest of companies in the commercial production of soil remineralizers, a possible material to be tested for this use is the ultrabasic rock olivine melilitite, which presents high contents of calcium and magnesium and expressive contents of potassium and phosphorus, with a localized occurrence in the highlands of Southern Brazil, especially in the alkaline complex of the municipality of Lages, in the state of Santa

Catarina (Scheibe, 1986). As other rock powders, this one should be evaluated to determine its most adequate particle size distribution, nutrient content, solubility and reaction rate in the soil, as well as its effectiveness as an alternative to soluble fertilizers in crops (Beerling et al., 2018; Zhang et al., 2018; Silva et al., 2019; Aguilera et al., 2020).

For a more detailed characterization of the melilitite olivine rock and identification of its benefits for both soil and plants, the hypotheses of the present study were: the filler particle size of the olivine melilitite rock powder improves the chemical properties of two soils from Santa Catarina, when compared with the powder particle size; nutrient accumulation by soybean [*Glycine max* (L.) Merr.] and sorghum [*Sorghum bicolor* (L.) Moench] plants is higher in the filler than in the powder particle size; nutrient release from the rock powder in both particle sizes provides an adequate nutrition for soybean and sorghum; and the response of soils and plants subjected to the conventional treatment will be better than that under the different particle sizes of the olivine melilitite rock powder.

The objective of this work was to evaluate the potential of olivine melilitite rock powder, in two particle sizes and in increasing rates, to improve the chemical properties of the soil and the growth and nutrient accumulation of soybean and sorghum plants.

Materials and Methods

The experiment was conducted from November 2017 to February 2018, using a commercial product of olivine melilitite rock powder (Dinamisa Agrominerais, Curitiba, PR, Brazil) with two levels of grinding: powder, preliminary grinding, with 100% of the rock particles passed through a 2.0 mm sieve, 87% through a 0.84 mm sieve, and 60% through a 0.3 mm sieve; and filler, fine grinding, with 100% of the particles passed through a 0.3 mm sieve, as recommended in Instrução Normativa Nº 5 (IN5) of Ministério da Agricultura, Pecuária e Abastecimento (Brasil, 2016), the Brazilian normative ruling for plant remineralizers and substrates. For its characterization, the olivine melilitite rock was subjected to petrographic, chemical, and mineralogical analyses.

The petrographic characterization was done through analyses of thin sections of the rock in the Axio Imager.

A2m petrographic microscope, using the AxioVision system of image capture and processing (Carl Zeiss Microscopy, LLC, White Plains, NY, USA). The analyses showed that the rock was a dark-gray color, with a very fine to thick granulation, grain size from 0.1 to 1.5 mm, a massive structure, a very fine to thick inequigranular phaneritic texture, and submillimeter fractures with different orientations, probably filled with clay minerals. The mineral composition of the rock included 40% melilitite, 35% clinopyroxene (diopside), 15% phlogopite, 5% olivine, and 5% opaque minerals, as well as traces of apatite, altering minerals, and clay minerals.

For the chemical characterization of the olivine melilitite rock, analyses were first carried out using the X-ray fluorescence (XRF) analyzer as described in Heberle (2017). Then, in a laboratory with ALS Global certification, chemical analyses were performed to quantify the present elements through plasma atomic emission spectrophotometry induced by argon.

The values obtained by XRF for the oxides of Si (SiO_2), Ca (CaO), Mg (MgO), K (K_2O), and P (P_2O_5) of the tested rock powder were similar to those found by the chemical analyses, except those of P, which were higher (Table 1).

The elemental chemical analyses revealed low contents of SiO_2 in the rock, confirming its classification in the ultrabasic group. However, the levels of CaO and MgO were very high and those of K_2O were expressive, meeting the criteria of IN5 (Brasil, 2016). In addition, the tested rock powder showed relatively high levels of total P (1.18% P_2O_5) (Table 1), which may contribute to the release of low amounts of the element

to the plants during the process of rock dissolution. A complementary and independent sample of crude rock was also analyzed in the laboratory, showing similar values to those of the tested rock powder, in order to guarantee the reliability of the obtained results.

Regarding potentially toxic elements, the tested material showed levels of 2.5, >0.5, 0.01, and 13 ppm arsenic, cadmium, mercury, and lead, respectively, which are below the allowed maximum limits of 15, 10, 0.1, and 200 ppm (Brasil, 2016).

For the mineralogical analysis of the rock, X-ray diffraction (XRD) was carried out in the PW 3710 diffractometer (Philips/PANalytical, Almelo, Netherlands), equipped with a copper tube, with a compensation angle of $\theta/2\theta$, and a graphite monochromator with an angular variation from 3.2 to $42^\circ 2\theta$. The angular velocity was $0.02^\circ 2\theta/\text{s}$ in step mode, with a reading time of 1 s per step. The diffractogram was made in the X'Pert Highscore Plus, version 3.0, software (PANalytical, Almelo, Netherlands). The criteria used for the interpretation of the diffractogram and for the identification of the minerals of the rock powder were based on the interplanar spacing and behavior of the diffraction reflexes presented by Jackson (1969), Brindley & Brown (1980), and Whittig & Allardice (1986), as well as on the complete tables containing the peaks of various minerals in the RRUFF database (Lafuente et al., 2015).

The mineralogical analysis revealed a mineral assemblage very similar to that found by the petrographic analysis, besides identifying the characteristic peaks of the following minerals: melilitite, clinopyroxene of the diopside type, olivine, and phlogopite (Figure 1). Small

Table 1. Characterization of two samples of olivine melilitite rock (OM4 and OM DIN 7) by chemical analyses and by X-ray fluorescence (XRF).

Method	SiO_2	CaO	MgO	K_2O	P_2O_5
	-----(%)-----				
	ICP-AES ⁽¹⁾				
OM4 ⁽²⁾	38.2	13.55	15.05	2.84	1.11
OM DIN 7 ⁽³⁾	35.7	14.85	17.4	2.73	1.18
	XRF				
OM DIN 7	37.7	15.28	14.91	3.59	1.55
	Content (%) of element in the rock ⁽⁴⁾				
	16.7	10.6	10.4	2.3	0.52

⁽¹⁾Plasma atomic emission spectrophotometry induced by argon.

⁽²⁾Supplementary sample of rock powder. ⁽³⁾Sample of rock powder particle size range. ⁽⁴⁾Calculated from the values obtained by OM DIN 7.

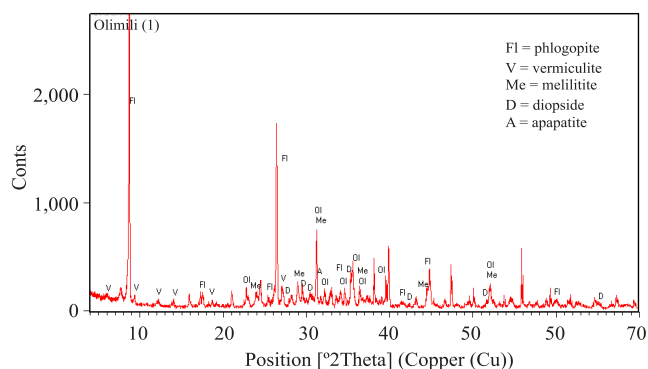


Figure 1. X-ray diffractogram of the applied olivine melilitite rock powder.

amounts of vermiculite were also detected by XRD, indicating a possible transformation of the phlogopite in solid state to vermiculite, which is consistent with the results of the petrographic analysis, showing a small amount of clay minerals in the submillimetric fractures of the rock. Moreover, the low-intensity peak around 2.81 Å indicated the presence of small amounts of apatite in the rock, compatible with the relatively high levels of P (around 1.2% P_2O_5) confirmed by the elemental analysis. Although the most intense peaks in the diffractogram were those of the phlogopite, which could indicate that it was the dominant mineral in the studied sample, the chemical and petrographic analyses showed, respectively, low amounts of K and phlogopite (Table 1). Therefore, there must have been segregation and/or orientation of the phlogopite mineral particles on the surface portion of the sample holder during the powder analysis. According to Dunworth & Wilson (1998), the presence of relatively high quantities of phlogopite and apatite are unusual in the olivine melilitite characterized in other environments.

For the study, besides the rock powder, two very acidic soils, in a unfertilized natural condition, were tested: a Cambissolo Háplico Alumínico típico, i.e., a Humic Dystrudept, with a clayey texture, 4.69 pH in water, 2.97 mg kg⁻¹ P, and 0.18 cmol_c kg⁻¹ K, obtained from the municipality of Lages, in the state of Santa Catarina; and an Argissolo Vermelho Distrófico sômbrico, i.e., a Typic Hapludult, with medium/clayey texture, 4.60 pH in water, 3.40 mg kg⁻¹ P, and 0.20 cmol_c kg⁻¹ K, from the municipality of Içara, located in the same state. Samples from both soils were collected at a depth of 0–20 cm from the surface horizon, air dried in a greenhouse, crushed, milled, sieved with a 4.0 mm mesh, and then incubated separately in a greenhouse for 60 days, being homogenized every 10 days.

Before incubation, the soils were characterized by chemical analyses for pH in water, pH in the 1:1 soil:solution ratio, and SMP pH, in order to calculate the rate of limestone to be used. Both soils were then corrected to pH 5.5 by applying 33.6 and 76.8 g dolomite limestone filler (Silva et al., 2016), respectively, calculated for 16 kg soil (dry basis). The organic carbon content was obtained by wet oxidation according to Tedesco et al. (1995), whereas P and K were extracted with the Mehlich-1 solution and quantified, respectively, by colorimetry (Murphy &

Riley, 1962) and flame photometry (Tedesco et al., 1995). Field capacity was determined as described in Casaroli & Jong van Lier (2008).

The experimental design was completely randomized in a (3x2+2)x2x2 factorial arrangement, with three rates olivine melilitite rock powder x two particle sizes + control + limestone and soluble fertilizers x two soils x two plant species.

The treatments consisted of the three rates of olivine melilitite rock powder, equivalent to 2.5, 5.0, and 10 Mg ha⁻¹, in two particle size ranges, called powder and filler, which were applied to the two different soils, on which the soybean and sorghum plants were cultivated. The control were the soils in natural and unfertilized condition, which was used, as a basis, to stipulate the chosen rock powder rates. Each treatment was carried out with four replicates, and the rates were homogenized in 16 kg soil, to which distilled water was applied to increase water content up to 80% field capacity throughout the experiment, which was done by replacing daily the amount of water lost after weighing the experimental units; the weight corresponding to plant growth in each soil and treatment was included in the water replacement.

The experimental unit was a 5.5 L pot containing 4.0 kg of each incubated soil (dry basis) used to cultivate the soybean and sorghum plants. Seven to eight seed of each species were sown, without pre-germination, per pot in both soil types. Ten days after emergence, plants were thinned out to two soybean and four sorghum plants per pot. The pots were randomized every ten days so that the plants had the same growth conditions. Furthermore, both the soybean and sorghum plants received sources of the conventional N, P, and K soluble fertilizers (Resende et al., 2012).

The soils subjected to the conventional treatment (limestone + soluble fertilizers) and cultivated with sorghum were fertilized with 2.09 g P (triple superphosphate) and 0.9 g K (potassium chloride), as recommended in the protocol for the agronomic evaluation of rock powders and derived products as sources of nutrients to plants or as soil conditioners (Resende et al., 2012). This procedure was necessary since it was a greenhouse experiment, where the volume of root exploitation was limited by the volume of the pot. The same amounts of P and K were applied to both soils before soybean cultivation, but not to the soils in the treatments with the rock powders. In

addition, nitrogen (0.35 g urea) was only applied to the soils cultivated with sorghum, also according to Resende et al. (2012), regardless of the treatment (limestone + soluble fertilizers and rock powder alone), totaling eight applications (0.044 g N per week) during the experiment with this plant species. Urea was not applied to soybean because its seeds were inoculated with rhizobia.

For sampling, all plants were collected on the same day but in different stages: soybean, when most plants were in the full-bloom phenological stage; and sorghum, in stage four (visible flag leaf), in the treatments with the two particle sizes of olivine melilitite rock powder, and, in stage five (booting, when all leaves were completely developed), in the treatments corrected with limestone and fertilized with N, P, and K.

From the plant samples, aboveground biomass was collected, kept in paper bags, and dried in a forced-circulation oven, at 65°C, until constant weight. After weighing, the dry matter of the aerial part (APDM) was obtained. Plant roots were separated manually, washed in running water, and dried as the aerial part to obtain root dry matter (RDM). The total dry matter (TDM) produced by the soybean and sorghum plants was considered the sum of the APDM and RDM; only the results of TDM were presented.

After the APDM and RDM were determined, the plant tissue of both soybean and sorghum was ground and then the samples were digested as described in Tedesco et al. (1995). The concentrations of Ca and Mg in the plant tissue were quantified in the Optima 8300 inductively coupled plasma emission optical spectrometer (PerkinElmer Inc., Waltham, MA, USA). Colorimetry (Murphy & Riley, 1962), flame photometry, and steam distillation in the semi-micro Kjeldahl equipment (Tedesco et al., 1995) were used to obtain P, K, and N concentrations, respectively.

From TDM and the N, P, K, Ca, and Mg contents, the accumulated amounts of these nutrients in the plant tissue were calculated according to the equation: $N_{\text{Macro}} (\text{mg}) = \text{TDM} (\text{mg}) \times \text{nutrient concentration} (\%) / 100$ (Cunha et al., 2019), where N_{Macro} corresponds to the amount of macronutrient accumulated in the vegetal tissue (aerial part + roots) of the tested plants.

After the collection of the aerial part and roots, the soils were homogenized for sampling, being air dried, crushed, milled, and sifted through a 2.0 mm mesh sieve to obtain the air-dried fine earth (ADFE).

In the ADFE samples, pH in water and in CaCl_2 0.01 mol L⁻¹ (1:1 soil:solution ratio) was determined according to Tedesco et al. (1995), as well as the contents of exchangeable Ca^{2+} , Mg^{2+} , K^+ , and Al^{3+} . The P, K⁺, and Na⁺ elements were extracted using the Mehlich-1 solution. Moreover, P was quantified by colorimetry (Murphy & Riley, 1962), and K⁺ and Na⁺ by flame photometry (Tedesco et al., 1995).

Soil chemical properties, TDM production, and the nutrient contents accumulated in the vegetal tissue of the soybean and sorghum plants were subjected to the analysis of variance with the aid of the SISVAR, version 5.6, software (Ferreira, 2014). For plant yield results, means were compared by the F-test for orthogonal contrasts, at 5% probability. These contrasts aimed at comparing the following treatments: control with the rock powder at different rates and particle sizes; limestone + soluble fertilizers with the rock powder at different rates and particle sizes; and filler and powder at different rates and particle sizes. The data obtained for the soils were subjected to the Scott-Knott test, also at 5% probability.

Results and Discussion

In most cases, when compared with the control treatments, the use of limestone and rock powder in the two particle size ranges, regardless of the plant species, soil type, and applied rates of the tested products, increased the values of pH in water and in CaCl_2 0.01 mol L⁻¹, as well as the contents of Ca^{2+} , Mg^{2+} , Na⁺, and P and the sum of bases and saturation, with a concomitant decrease in H⁺/Al, exchangeable Al^{3+} , and Al saturation (Table 2). These results are indicative that the applied products, even in a short period of time, were efficient in improving the fertility of the two evaluated soils.

Higher values were found for pH in water especially after the cultivation of soybean in the Typic Hapludult, probably because this soil is less acidic and buffered. As this is a less buffered soil, the measured pH was higher in the treatments with limestone + PK for soybean and limestone + NPK for sorghum, when compared with those with the rock powder in the two particle size ranges. In the Humic Dystrudept, the pH values in water, at the highest rate and finer particle size of the rock powder, were superior to those obtained in all other treatments, regardless of the plant species,