



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.

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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 Distroférico 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 (COFS 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^{5+} , 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).

Table 3. Soil chemical attributes after soybean cultivation in a greenhouse.

Soil	Treatment t ha ⁻¹	pH	Al ³⁺	H+Al	Ca ²⁺	Mg ²⁺	K ⁺	Na ⁺	V	m	P
		Water	1:1	cmol _c kg ⁻¹			%			mg kg ⁻¹	
Cambisol	Control	4.57f	4.73a	10.55a	0.51h	0.69d	0.27d	0.16d	13j	74a	6.94h
	Limestone	5.61a	0.19i	5.17e	5.20a	3.95a	0.27d	0.20d	65a	2j	8.89g
	Siltstone – 2.5	4.73e	4.35b	10.39a	0.68h	0.59e	0.25e	0.14d	14i	72a	8.68g
	Siltstone – 5.0	4.85e	4.01c	10.14b	0.77h	0.70d	0.27d	0.18d	16h	68b	9.98f
	Siltstone – 10	4.82e	3.89c	9.93b	0.98g	0.80c	0.26d	0.18d	18g	64c	11.81e
	Manure	4.79e	3.99c	10.93a	0.70h	0.53e	0.22g	0.21d	13i	71b	11.01e
	Manure + Siltstone – 2.5	4.77e	3.98c	10.23a	0.72h	0.57e	0.24e	0.18d	14i	70b	10.19f
	Manure + Siltstone – 5.0	4.98d	3.69d	9.68b	0.89g	0.69d	0.21g	0.23d	17g	65c	11.77e
	Tephrite – 2.5	4.74e	4.29b	10.40a	0.57h	0.53e	0.27d	0.28c	14i	72a	9.83f
	Tephrite – 5.0	4.76e	4.14b	10.01b	0.60h	0.54e	0.28c	0.42c	16h	69b	11.45e
	Tephrite – 10	4.82e	3.87c	9.77b	1.35f	0.55e	0.27d	0.33c	20f	60d	11.85e
	Manure + Tephrite – 2.5	4.91d	3.78c	10.03b	0.63h	0.55e	0.23f	0.32c	15i	69b	10.97e
	Manure + Tephrite – 5.0	4.98d	3.54d	9.64b	0.63h	0.55e	0.23f	0.25d	15i	68b	14.73d
	Olivine Melilitite – 5.0	5.04c	2.90e	8.90c	1.35f	0.85c	0.31b	0.37c	24e	50e	11.74e
	Olivine Melilitite – 10	5.13c	1.75h	7.66d	3.75b	0.99b	0.33a	0.76a	43b	23i	18.92b
	Siltstone + O.Meli. – 5.0 ⁽⁵⁾	4.91d	3.42d	9.33c	0.69h	0.78c	0.28c	0.27d	18g	63c	12.04e
	Siltstone + O.Meli. – 10 ⁽⁴⁾	5.01c	2.63f	8.83c	2.89c	0.93b	0.29c	0.55b	35c	36g	15.69c
	Manure + siltstone + O.Meli. – 5.0 ⁽⁵⁾	5.07c	2.64f	9.09c	1.84d	0.82c	0.26d	0.33c	26d	44f	14.33d
	Manure + siltstone + O.Meli. – 10 ⁽⁶⁾	5.30b	2.13g	8.75c	2.66d	0.96b	0.24e	0.62b	34c	32h	22.08a
	Nitisol	Control	5.12f	0.68a	7.12a	0.67d	1.24d	0.15f	0.17f	24f	23a
Limestone		6.29a	0.05g	5.12b	3.81a	4.30a	0.16e	0.20f	62a	1f	5.79b
Siltstone – 2.5		5.17f	0.65a	7.29a	1.86c	1.65c	0.16e	0.17f	34e	14b	4.81c
Siltstone – 5.0		5.54d	0.65a	7.12a	1.99c	1.44d	0.16e	0.17f	35e	15b	5.78b
Siltstone – 10		5.75c	0.52b	6.78a	2.10b	1.67c	0.16e	0.21f	38d	11c	6.23b
Manure		5.34e	0.47c	6.83a	1.95c	1.70c	0.14f	0.26f	37e	10c	3.59d
Manure + Siltstone – 2.5		5.65d	0.45c	6.50a	1.89c	1.81c	0.13f	0.22f	38d	10c	3.14d
Manure + Siltstone – 5.0		5.76c	0.38d	6.48a	2.33b	1.75c	0.13f	0.23f	41d	8d	4.11d
Tephrite – 2.5		5.51d	0.47c	6.67a	1.76c	1.64c	0.16e	0.30e	36e	11c	3.52d
Tephrite – 5.0		5.68d	0.45c	6.62a	1.83c	1.85c	0.16e	0.59c	40d	9d	4.68c
Tephrite – 10		5.63d	0.48c	6.29a	1.88c	2.66b	0.18c	0.89a	47b	8d	4.86c
Manure + Tephrite – 2.5		5.73c	0.47c	6.91a	1.91c	1.77c	0.16e	0.42d	38d	10c	4.45c
Manure + Tephrite – 5.0		5.57d	0.51b	6.84a	1.73c	2.01c	0.16e	0.66b	40d	10c	4.69c
Olivine Melilitite – 5.0		5.75c	0.20e	5.93b	2.04b	1.63c	0.19b	0.57c	43c	4e	5.03c
Olivine Melilitite – 10		5.87b	0.18f	5.61b	2.12b	1.40d	0.21a	0.68b	44c	4e	7.16a
Siltstone + O.Meli. – 5.0 ⁽⁷⁾		5.77c	0.38d	6.57a	1.97c	1.55d	0.17d	0.30e	38d	9d	5.79b
Siltstone + O.Meli. – 10 ⁽⁸⁾		5.76c	0.22e	5.54b	2.19b	1.84c	0.18c	0.57c	46b	4e	5.68b
Manure + Siltstone + O.Meli. – 5.0 ⁽⁹⁾		5.93b	0.15f	5.65b	2.33b	1.99c	0.16e	0.65b	48b	3e	7.35a
Manure + Siltstone + O.Meli. – 10 ⁽¹⁰⁾		6.33a	0.17f	5.66b	1.89c	2.56b	0.13f	0.34e	46b	3e	5.07c

⁽¹⁾Base saturation; ⁽²⁾Aluminum saturation; ⁽³⁾and ⁽⁷⁾Siltstone + Olivine Melilitite at the dose of 5 t ha⁻¹; ⁽⁴⁾and ⁽⁸⁾Siltstone + Olivine Melilitite at the dose of 10 t ha⁻¹; ⁽⁵⁾and ⁽⁹⁾Manure + Siltstone + Olivine Melilitite at the dose of 5 t ha⁻¹; ⁽⁶⁾and ⁽¹⁰⁾Manure + Siltstone + Olivine Melilitite at the dose of 10 t ha⁻¹.

In the Cambisol, the most buffered soil, the values of pH in water in the treatments with limestone and rock dusts associated or not with manure increased from 0.19 to 1.04 pH units compared to the control. This increase was observed in all treatments, regardless of the products and doses. However, the pH values showed no statistical difference between doses and, in some cases, between treatments. All the pH values were lower than the values determined in the treatments that received limestone (Table 3).

In the Nitisol, the least buffered soil, the pH in water was higher than the values obtained in the Cambisol. The pH values of all treatments were higher than those found in the control, with values ranging from 0.22 to 1.17 pH units, except for the treatment that received the rock dust pure siltstone at the dose of 2.5 t ha⁻¹, which did not differ statistically from the control. This increase was more evident with the increased doses of the rock dusts, pure or combined with manure, differing statistically between doses and between treatments, except for the pure tephrite, and in treatments that received the conventional fertilization. Moreover, the pH in water of the treatments that received manure + siltstone + olivine melilitite were similar to the values obtained in the treatments with limestone (Table 3). Similar behavior was found with the pH values obtained with 0.01 mol L⁻¹ CaCl₂ solution (data not shown).

Although the rock dusts have reduced Al³⁺ contents compared to the control treatments, the decrease in the Cambisol was less pronounced than in the Nitisol, remaining close to the critical limit of 4 cmol_c kg⁻¹ in several treatments (Table 3), as recommended by the CQFS-RS/SC (2016).

Treatments subjected to liming and increasing doses of rock dusts, pure or combined with manure, provided a decrease in Al^{3+} contents in both soils, especially at the highest doses applied in most treatments, compared to the control. In addition, treatments that received the rock dusts olivine melilitite and siltstone + olivine melilitite, pure and combined with manure, had the highest reductions regarding the contents of this element in the evaluated soils, except for the mixture of these rock dusts at the dose of 5 t ha^{-1} in the Cambisol (Table 3).

Because the Al contents are directly influenced by pH, the values observed in this study had the same statistical behavior, with statistical differences as the doses of the products applied to the soils increased and between treatments, but always with lower values than the control. However, the Nitisol presented no statistical difference in the treatments that received the rock dusts pure siltstone at doses of 2.5 and 5 t ha^{-1} and tephrite at the three applied doses (Table 3).

The same interpretation made for Al can be applied for Al saturation (m%) because this soil chemical attribute is calculated considering its content and the effective cation exchange capacity. However, the value of m% was above that allowed for a good crop development (20-30%) in the first evaluated soil (Hashimoto, Smyth, Israel, & Hesterberg, 2010) in most treatments, except for treatments subjected to liming. On the other hand, m% was below the tolerable limit for the good development of soybean plants in the Nitisol, regardless of the treatment (Table 3).

The Ca^{2+} contents in both soils were lower than the critical limit considered adequate by CQFS-RS/SC (2016), i.e., $4 \text{ cmol}_c \text{ kg}^{-1}$, except for treatments that received limestone. However, the highest contents of this nutrient were observed in both soils that also received the pure rock dusts siltstone and tephrite at the dose of 10 t ha^{-1} , olivine melilitite, siltstone + olivine melilitite, and their combination with manure at the two doses, except for the dose of 5 t ha^{-1} of the rock dust siltstone + olivine melilitite in the Cambisol (Table 3). The highest contents of this nutrient were mainly observed with the increased product doses, differing statistically from the lower doses in both soils, except in treatments that received pure tephrite in the Nitisol. In the Nitisol, the maximum doses of the rock dusts, combined or not with manure and limestone, exceeded the control and did not differ statistically from each other. The other treatments in the Cambisol presented no difference from each other and the control, while the other treatments in the Nitisol had no statistical difference from each other in most cases, being higher than the control (Table 3).

The Mg^{2+} contents in the Cambisol were lower than the value considered adequate by the CQFS-RS/SC (2016), i.e., $1 \text{ cmol}_c \text{ kg}^{-1}$. The highest Mg^{2+} contents in this soil were observed in the same treatments previously indicated for Ca^{2+} , except the treatment that received the combination of manure and siltstone at the dose of 5 t ha^{-1} . In addition, the contents of this nutrient in these same treatments increased when the doses were increased. A statistical difference was also observed with the increased doses (Table 3). Seeds produced by soybean plants grown in the soil submitted to the rock dust pure siltstone at a dose of 2.5 t ha^{-1} , as well as pure tephrite and its combination with manure at all doses, showed no statistical difference from each other and had lower values than those obtained in the control, as will be evidenced later on.

The low Mg^{2+} contents in these treatments compared to the control may have occurred due to its higher use for the total dry matter (TDM) production, which was higher than the control and in part translocated and accumulated in the seeds, with no statistical difference with the control and a trend to increase the Mg accumulation, and also due to the physiological functions. On the other hand, the Mg^{2+} contents in the Nitisol and all treatments were above the adequate for good plant development, according to CQFS-RS/SC (2016). Moreover, all contents of this nutrient in the treatments that received limestone and rock dusts combined or not with manure were higher than the control, showing, in general, no statistical differences between doses and between treatments (Table 3).

Treatments submitted to the application of the rock dusts olivine melilitite and siltstone + olivine melilitite at the dose of 10 t ha^{-1} and manure + siltstone + olivine melilitite at the two used doses presented Ca^{2+} contents 3 to 7 and 2 to 4 times higher than the control in the Cambisol and Nitisol, respectively. The ratio between Mg contents and those observed in the control was 1:1, regardless of the soil, products, and doses (Table 3).

The increased Ca^{2+} and Mg^{2+} contents in the soils, especially at the highest doses, may be related to the high CaO and MgO contents in the rock dusts, mainly olivine melilitite and its mixture with siltstone, improving the chemical characteristics of soils that received these pure rocks and their combination with manure.

No substantial changes were observed for the extractable K^+ contents of soils subjected to the different treatments (Table 3). Even the rocks evaluated in this study had high K_2O contents. However, the rock dust olivine melilitite at the dose of 10 t ha^{-1} showed the highest contents of this nutrient compared to the other

treatments in both soils, indicating its promising role in supplying K^+ to the soil and, consequently, to plants. In general, the contents of this nutrient in the other treatments showed no statistical difference between doses and between treatments, but most treatments had values higher than the control in both soils (Table 3). It does not mean that the rocks are not releasing potassium, as this nutrient may have been absorbed during the different stages of plant development, being accumulated in the TDM and then in the seeds.

The Na^+ contents increased in both soils for treatments with the rock dusts pure tephrite and its combination with manure at all doses, except for manure + tephrite at the dose of 5 t ha^{-1} in the Cambisol, olivine melilitite and its mixture with siltstone at the two doses, except for the first dose of siltstone + olivine melilitite in the Cambisol, and the combination of manure + siltstone + olivine melilitite at the two used doses. In most cases, these treatments showed a statistical difference between doses and treatments (Table 3). However, the contents remained below those considered critical (above 6%), showing no sodic character, i.e., a percentage of sodium in CEC between 6 and 15%, according to the criteria established by *Empresa Brasileira de Pesquisa Agropecuária* (Embrapa, 2018). The other treatments presented no difference from each other and were similar to the control (Table 3).

The extractable P contents in both soils and all treatments increased, being higher than those obtained in the control, regardless of the used products and doses (Table 3). The highest increase in P in the Cambisol was observed in treatments that received rock dust (mainly olivine melilitite) with and without manure at the highest doses. The highest P contents in the Nitisol were found in treatments with limestone, siltstone at the last two doses, olivine melilitite at the maximum dose, siltstone + olivine melilitite combined with manure at the dose of 5 t ha^{-1} , pure siltstone, siltstone + olivine melilitite at doses of 5 and 10 t ha^{-1} , and limestone. The other treatments presented no statistical difference from each other, being higher than the control (Table 3).

These results are possibly associated with manure and the presence of apatites in the rocks, promoting an increase in P availability during the experimental period. However, these results should be interpreted with caution, as a double acid solution (Mehlich-1) was used for its extraction, which may be dissolved P fractions not immediately available to the crops, overestimating its contents in the samples of the evaluated soils.

Therefore, the pure siltstone and tephrite possibly need a longer time to solubilize and release the nutrients of its mineralogical composition compared to the other rock dusts, leading to an improvement of soil chemical properties and directly interfering with plant productivity and seed chemical composition. This interpretation is supported by Duarte et al. (2013) and Tavares, Carvalho, Camargo, Pereira, and Cardoso (2018) when evaluating other rock dusts. On the other hand, olivine melilitite and its mixture with siltstone, as well as manure pure and combined with most of the rocks tested in this study, represent a potential faster source of nutrient release (Silva, Pereira, Coelho, Almeida, & Schmitt, 2011; Duarte et al., 2013; Oelkers, Declersq, Saldi, Gislason, & Schott, 2018), as the contents of the evaluated nutrients were higher than or similar to the sufficiency range recommended by CQFS-RS/SC (2016).

Chemical composition, nutrient accumulation in seeds, and soybean production

The chemical composition of nutrients in the soybean seeds, as well as TDM production, varied according to the treatment, that is, the solubility of materials used in the treatments (fast: limestone, soluble fertilizers, and manure; slow: rock dusts) and the plant requirements to use them in their different stages of development until production (Tables 4 and 5, respectively).

The highest Ca contents in the seeds from plants cultivated in the Cambisol were obtained in treatments with limestone, siltstone, and the mixture of this rock with olivine melilitite at the used doses, with no difference from each other, but differing from the control. In the Nitisol, the Ca contents in the pure manure and its association with siltstone at the dose of 2.5 t ha^{-1} , as well as in the olivine melilitite at doses of 5 and 10 t ha^{-1} , were similar to the control. The Ca contents in the other treatments were slightly lower, differing from the control and the treatment with limestone, but with no statistical difference (Table 4).

In general, the Ca contents in the seeds were lower than the other nutrients, regardless of the soil, treatment, and applied product (Table 4). This behavior is probably related to the low Ca mobility (Domingos, Lima, & Braccini, 2015; Fioreze, Tochetto, Coelho, & Melo, 2018; Cunha et al., 2019) due to the functions it has in the different plant structures, such as calcium pectate formation (cell wall strengthening), plasma membrane stabilization (plasmalemma), root growth (Yamamoto, Ferreira, Fernandes, Albuquerque, & Alves, 2011; Fioreze et al., 2018), and Ca accumulation in the endoplasmic reticulum, chloroplasts, and vacuoles (Zeng, Zhang, Zhang, Pi, & Zhu, 2017).

Table 4. Chemical composition (nutrient content) of soybean seeds grown on a Cambisol and Nitisol under greenhouse conditions.

Soil	Treatment t ha ⁻¹	Ca	Mg	K	P Total	P Inorganic	Phytate	N	CP ⁽¹⁾
							%		
Cambisol	Control	0.095b	0.19c	1.08b	0.23e	0.28b	0.29d	3.94a	24.63a
	Limestone	0.103a	0.24b	1.72a	0.53b	0.36a	0.81a	4.23a	26.44a
	Siltstone – 2.5	0.110a	0.18c	1.41b	0.33e	0.29b	0.38d	4.01a	25.06a
	Siltstone – 5.0	0.115a	0.24b	1.39b	0.32e	0.30b	0.36d	4.25a	26.56a
	Siltstone – 10	0.115a	0.23b	1.39b	0.33e	0.29b	0.40d	4.16a	26.00a
	Manure	0.080b	0.24b	1.60a	0.45c	0.31b	0.62b	4.08a	25.50a
	Manure + siltstone – 2.5	0.091b	0.23b	1.56a	0.40d	0.31b	0.31d	4.11a	25.69a
	Manure + siltstone – 5.0	0.086b	0.25a	1.83a	0.54b	0.31b	0.54c	4.43a	27.69a
	Tephrite – 2.5	0.081b	0.21b	1.27b	0.28e	0.28b	0.22d	4.11a	25.69a
	Tephrite – 5.0	0.093b	0.22b	1.44b	0.31e	0.29b	0.27d	4.11a	25.69a
	Tephrite – 10	0.095b	0.26a	1.60a	0.36d	0.32b	0.49c	4.24a	26.50a
	Manure + Tephrite – 2.5	0.093b	0.26a	1.76a	0.43c	0.30b	0.52c	4.38a	27.38a
	Manure + Tephrite – 5.0	0.081b	0.27a	1.75a	0.47c	0.37a	0.53c	4.06a	25.38a
	Olivine Melilitite – 5.0	0.093b	0.25a	1.74a	0.39d	0.30b	0.48c	4.42a	27.63a
	Olivine Melilitite – 10	0.099b	0.23b	1.68a	0.40d	0.31b	0.51c	4.61a	28.81a
	Siltstone + OM – 5.0 ⁽²⁾	0.127a	0.29a	1.78a	0.38d	0.31b	0.45c	4.36a	27.25a
	Siltstone + OM – 10 ⁽³⁾	0.121a	0.27a	1.65a	0.37d	0.32b	0.50c	4.33a	27.06a
	Manure + Siltstone + OM – 5.0 ⁽⁴⁾	0.093b	0.27a	1.91a	0.54b	0.34a	0.49c	4.65a	29.06a
	Manure + Siltstone + OM – 10 ⁽⁵⁾	0.097b	0.26a	1.88a	0.62a	0.37a	0.64b	4.47a	27.94a
	CV (%)		11.88	12.69	9.59	13.05	8.76	12.32	8.64
Nitisol	Control	0.119a	0.20b	1.31b	0.29b	0.29b	0.18c	4.29 ^{ns}	26.81 ^{ns}
	Limestone	0.113a	0.26a	1.74a	0.42a	0.34a	0.63a	4.43	27.69
	Siltstone – 2.5	0.104a	0.18c	1.23b	0.24b	0.35a	0.25c	4.62	28.88
	Siltstone – 5.0	0.094b	0.15c	1.30b	0.24b	0.32a	0.36c	4.57	28.56
	Siltstone – 10	0.109a	0.18c	1.41a	0.27b	0.35a	0.39b	4.47	27.94
	Manure	0.127a	0.24a	1.72a	0.37a	0.34a	0.44b	4.25	26.56
	Manure + Siltstone – 2.5	0.101a	0.19c	1.21b	0.25b	0.34a	0.26c	3.83	23.94
	Manure + Siltstone – 5.0	0.091b	0.21b	1.29b	0.30b	0.35a	0.32c	4.20	26.25
	Tephrite – 2.5	0.065b	0.14c	1.05b	0.20b	0.34a	0.25c	4.29	26.81
	Tephrite – 5.0	0.085b	0.17c	1.09b	0.29b	0.35a	0.28c	3.98	24.88
	Tephrite – 10	0.082b	0.15c	1.02b	0.29b	0.34a	0.26c	4.55	28.44
	Manure + Tephrite – 2.5	0.070b	0.17c	0.97b	0.32b	0.33a	0.37c	4.16	26.00
	Manure + Tephrite – 5.0	0.96b	0.20b	1.33b	0.36a	0.35a	0.28c	3.93	24.56
	Olivine Melilitite – 5.0	0.110a	0.20b	1.11b	0.29b	0.33a	0.40b	3.97	24.81
	Olivine Melilitite – 10	0.105a	0.20b	1.10b	0.34a	0.33a	0.53a	4.05	25.31
	Siltstone + O.Meli. – 5.0 ⁽⁶⁾	0.128a	0.22b	1.14b	0.29b	0.31b	0.29c	4.11	25.69
	Siltstone + O.Meli. – 10 ⁽⁷⁾	0.115a	0.22b	1.57a	0.35a	0.33a	0.46b	4.52	28.25
	Manure + siltstone + O.Meli. – 5.0 ⁽⁸⁾	0.069b	0.22b	1.40a	0.39a	0.31b	0.49b	4.41	27.56
	Manure + siltstone + O.Meli. – 10 ⁽⁹⁾	0.072b	0.21b	1.65a	0.37a	0.36a	0.44b	4.42	27.63
	CV (%)		19.44	12.24	16.65	16.79	7.23	20.75	13.36

⁽¹⁾Crude protein; ⁽²⁾and ⁽⁶⁾Siltstone + Olivine Melilitite at the dose of 5 t ha⁻¹; ⁽³⁾and ⁽⁷⁾Siltstone + Olivine Melilitite at the dose of 10 t ha⁻¹; ⁽⁴⁾and ⁽⁸⁾Manure + Siltstone + Olivine Melilitite at the dose of 5 t ha⁻¹; ⁽⁵⁾and ⁽⁹⁾Manure + Siltstone + Olivine Melilitite at the dose of 10 t ha⁻¹. Means followed by the same letter in the columns do not differ from each other by the Scott-Knott test at 5%. ^{ns}Not significant.

The low Ca contents in the seeds may also be related to its (antagonistic) competition between this nutrient and Mg by root absorption sites, as observed by the 1:1 soil exchangeable ratio in most treatments in both soils (Table 3). According to Guimarães Júnior et al. (2013), this competition may occur due to the similarity of its ionic radii, valences, degrees of hydration, and mobility, which is higher in Mg. The competition hypothesis is plausible, as the Ca contents accumulated in TDM and, subsequently, translocated and accumulated in the soybean seeds was lower among all the evaluated nutrients (Table 5). Therefore, the lower the amount of a certain nutrient the plant accumulates in its vegetative organs during its entire cycle, the lower it will be used in the seed filling stage because the soil has little participation in the plant nutrition at this stage.

In the Cambisol, the Mg contents were higher both in absolute values and statistically in the treatments that received the rock dusts tephrite at the dose of 10 t ha⁻¹, olivine melilitite at the dose of 5 t ha⁻¹, siltstone + olivine melilitite, and manure + siltstone + olivine melilitite at doses of 5 and 10 t ha⁻¹. The other treatments

showed no statistical difference from each other, with higher contents compared to those observed in the control. In the Nitisol, treatments that received limestone and pure manure presented higher Mg contents than the other treatments, being similar to each other. Treatments that received olivine melilitite, its mixture with siltstone associated or not with manure at the two doses, manure + siltstone, and manure + tephrite at doses equivalent to 5 t ha⁻¹ did not differ from the control. The Mg contents in the other treatments were lower than those previously mentioned, not differing from each other (Table 4).

Table 5. Nutrient accumulation in soybean seeds and total dry matter production by soybean plants grown in a Cambisol and Nitisol under greenhouse conditions.

Soil	Treatment	TDM ⁽¹⁾	Ca	Mg	K	P total	P inorganic	N
	t ha ⁻¹	g				mg		
Cambisol	Control	1.21f	1.14c	2.33e	12.85e	2.74g	3.40f	47.70e
	Limestone	10.52b	13.91a	31.69b	232.11b	71.25b	48.37b	567.05b
	Siltstone – 2.5	2.59e	2.30c	3.66e	29.68e	6.91g	6.19f	84.25e
	Siltstone – 5.0	1.67f	2.13c	4.41e	25.70e	5.76g	5.60f	79.77e
	Siltstone – 10	2.39e	3.03c	5.99e	36.70e	8.61g	7.62f	109.68
	Manure	5.98d	7.05b	20.77d	143.17d	39.90e	27.89d	362.28c
	Manure + siltstone – 2.5	5.67d	7.27b	18.68d	126.56d	31.60e	25.59d	332.79d
	Manure + siltstone – 5.0	7.31c	9.84b	28.96b	209.10b	62.20c	34.86c	504.78b
	Tephrite – 2.5	2.45e	1.26c	3.38e	19.42e	4.21g	4.31f	62.70e
	Tephrite – 5.0	2.31e	1.70c	4.13e	26.64e	5.72g	5.22f	75.53e
	Tephrite – 10	3.40e	2.91c	7.58e	49.19e	11.32g	9.68f	130.33e
	Manure + tephrite – 2.5	4.62d	6.49b	18.19d	124.53d	30.71e	21.57e	310.76d
	Manure + tephrite – 5.0	7.17c	8.13b	26.60c	175.54c	46.92d	37.02c	406.37c
	Olivine melilitite – 5.0	5.22d	6.36b	17.23d	119.91d	26.55f	20.69e	302.32d
	Olivine melilitite – 10	6.18d	8.18b	19.16d	138.49d	33.09e	26.08d	377.58c
	Siltstone + OM – 5.0 ⁽⁵⁾	4.79d	8.06b	18.33d	113.59d	24.17f	20.07	277.67d
	Siltstone + OM – 10 ⁽⁴⁾	5.35d	8.31b	18.64d	113.23d	25.08f	22.37e	297.13d
	Manure + siltstone + OM – 5.0 ⁽⁵⁾	7.51c	9.15b	26.24c	184.79c	52.11d	32.65c	450.44c
	Manure + siltstone + OM – 10 ⁽⁶⁾	12.73a	15.54a	41.76a	299.72a	98.99a	58.47a	712.23a
	CV (%)		15.10	25.96	14.37	17.35	19.45	18.95
Nitisol	Control	2.08d	1.30f	2.28e	14.80f	3.38e	5.91d	48.15e
	Limestone	12.39a	14.73a	33.39a	227.88a	54.22a	42.15a	579.98a
	Siltstone – 2.5	2.03d	1.24f	2.15e	14.89f	2.89e	7.06d	56.22e
	Siltstone – 5.0	2.71d	1.47f	2.32e	20.06f	3.82e	8.73d	68.97e
	Siltstone – 10	2.27d	2.47f	4.05e	32.19f	6.14e	8.02d	102.59
	Manure	6.82c	10.61b	20.31b	144.63c	31.24c	23.11c	358.72c
	Manure + siltstone – 2.5	5.34c	6.54d	12.11d	78.39e	16.51d	18.10c	250.35d
	Manure + siltstone – 5.0	8.15b	8.39c	19.28c	119.69d	28.12c	28.66b	387.88c
	Tephrite – 2.5	2.09d	0.98f	2.02e	15.65f	2.98e	7.22d	63.83e
	Tephrite – 5.0	2.46d	1.82f	3.54e	23.49f	6.28e	8.59d	84.79e
	Tephrite – 10	2.73d	1.96f	3.61e	23.92f	6.21e	9.27d	107.68e
	Manure + tephrite – 2.5	5.10c	4.14e	9.86d	56.13e	18.35d	16.60c	245.17d
	Manure + tephrite – 5.0	5.51c	7.59c	16.25c	105.43d	28.02c	19.32c	316.42c
	Olivine melilitite – 5.0	4.38c	5.66d	10.40d	57.24e	15.00d	14.33c	203.98d
	Olivine melilitite – 10	4.84c	5.76d	10.91d	60.61e	18.39d	16.28c	219.88d
	Siltstone + OM – 5.0 ⁽⁷⁾	3.69d	5.54d	9.49d	49.50e	12.52d	11.45	178.22d
	Siltstone + OM – 10 ⁽⁸⁾	5.33c	7.04c	13.51d	99.19d	21.84d	17.76c	275.21d
	Manure + siltstone + OM – 5.0 ⁽⁹⁾	11.65a	7.42c	23.41b	153.52c	43.60b	36.17a	481.98b
	Manure + siltstone + OM – 10 ⁽¹⁰⁾	10.72a	7.65c	21.99b	175.90b	40.12b	39.01a	469.97b
	CV (%)		23.77	25.40	19.92	25.13	24.21	24.55

⁽¹⁾Total dry matter; ⁽²⁾Seed weight; ⁽³⁾and ⁽⁷⁾Siltstone + Olivine Melilitite at the dose of 5 t ha⁻¹; ⁽⁴⁾and ⁽⁸⁾Siltstone + Olivine Melilitite at the dose of 10 t ha⁻¹; ⁽⁵⁾and ⁽⁹⁾Manure + Siltstone + Olivine Melilitite at the dose of 5 t ha⁻¹; ⁽⁶⁾and ⁽¹⁰⁾Manure + Siltstone + Olivine Melilitite at the dose of 10 t ha⁻¹. Means followed by the same letter in the columns do not differ by the Scott-Knott test at 5%. ^{ns}Not significant.

The low Mg contents of the seeds in both soils compared to the other nutrients (except Ca) (Table 4) may be related to its competition with K and Ca for the absorption sites in the plant roots (Senbayram, Gransee, Wahle, & Thiel, 2015; Bruns, 2016; Cunha et al., 2019). Senbayram et al. (2015) reported that K absorption by plants is usually not impaired in soils whose Mg contents are higher than the K contents, as observed in this

study (Table 3). According to Horie et al. (2011), it occurs because plants develop specific transport systems for K that allow its absorption even when its available or soluble contents in the soil solution are low. In addition, the plant absorbs more K than Mg when the concentration of K in the soil-root system is high.

The hypothesis of antagonism between Mg and K can also be supported by the low Mg accumulation in TDM, with subsequent translocation and accumulation in the seeds (Tables 5 and 4, respectively), compared to K, N, and P, probably related to its lower absorption by the roots or its use in other physiological functions, such as protein synthesis and as the main constituent of chlorophyll molecules (Gerendás & Führs, 2013; Senbayram et al., 2015).

Potassium, followed by N, was the nutrient with the highest amount in the seeds, regardless of the soil and the used product. The lowest K contents in the Cambisol were found in the control, siltstone at the three doses, and tephrite at the two highest doses, not differing from each other. The other treatments presented K contents higher than those previously reported, not differing statistically from each other. However, the K contents in the Nitisol were similar regarding the seeds produced from treatments subjected to limestone, siltstone and siltstone + olivine melilitite at the highest dose, and pure manure and its association with siltstone + olivine melilitite at the two doses. The other treatments did not differ from each other. These results show, especially in the Cambisol, that most of the rock dusts provided adequate amounts of K to the plants, with contents similar to those that received the application of soluble fertilizers (Table 4).

The total phosphorus (Pt) contents of soybean seeds grown in the Cambisol were higher in treatments that received manure + siltstone + olivine melilitite at the dose of 10 t ha⁻¹, followed by treatments that received limestone, manure + siltstone, and manure + siltstone + olivine melilitite, both at the dose of 5 t ha⁻¹. The other treatments showed no statistical difference from each other, being similar to those found in the control. In the Nitisol, the highest Pt contents in the seeds were found in treatments subjected to limestone, olivine melilitite and siltstone + olivine melilitite at the highest doses, and pure manure and its association with tephrite at the dose of 5 t ha⁻¹ and siltstone + olivine melilitite at the two used doses, with no statistical difference from each other. The other treatments presented no difference from the control, regardless of the product and dose (Table 4).

The similarity among Pt contents in the treatments that received the previously mentioned rock dusts associated or not with manure with those of the treatment subjected to limestone and soluble fertilizers is probably due to the higher chemical decomposition of the rock dusts provided by the manure addition, favoring an increase in the P availability for the soils (Table 3), which is absorbed, accumulated in the plants (Table 5), and translocated to the seeds (Table 4).

This interpretation is supported by the study carried out by Meena and Biswas (2014), who found that an increase in the available P contents in the soil was verified in the treatments with phosphate rock dusts associated with organic sources. The authors suggested that the increased P contents in the evaluated soils occurred due to the production of organic acids (citric, oxalic, and tartaric acid) and the higher activity of the microbiota, which increased the rock solubility, favoring the release of nutrients. Similar behavior was observed in this study, as the available P contents increased in both soils when using cattle manure (Table 3), being positively correlated with Pt in the seeds (Cambisol: $r = 0.90$ to 0.93 ; Nitisol: $r = 0.1$ to 0.23).

The absence of statistical difference between Pt contents in the different treatments may be related to their accumulation in the TDM of plants, being then translocated and accumulated in the seeds, which was higher than those of Ca and Mg, regardless of the soil and treatment (Tables 4 and 5). According to Silva, Barros, Novais, and Pereira (2002), the increase in the concentration of nutrients in the soil solution provides high absorption and subsequent increasing accumulation in the plant shoot, being proportionally higher than the dry matter produced by plants. Although the rocks evaluated in this study showed low P₂O₅ contents in their composition, the higher P accumulation in plants, according to Tomaz et al. (2009), may be related to the lower need for P in the biochemical reactions of plants, with its higher redistribution to the growth points and P mobilization stored in vacuoles in cells under a deficiency condition.

The inorganic phosphorus (Pi) contents in the Cambisol were higher in treatments that received limestone, manure + siltstone + olivine melilitite at both doses, and manure + tephrite at the dose of 5 t ha⁻¹ than in the other treatments, with no statistical difference from each other. The other treatments presented no difference from the control, as observed for Pt. On the other hand, the Pi contents of seeds produced in the Nitisol subjected to the treatments siltstone + olivine melilitite and manure + siltstone + olivine melilitite, both at the lowest dose, were similar to those of the control. The other treatments showed no statistical difference from each other, regardless of the used product and dose (Table 4). Therefore, the P from organic matter and/or products applied to soils was mineralized to the same magnitude by the microbial activity, increasing

Pi concentration in the solution, being absorbed and accumulated in the dry matter of plants and then translocated to the seeds.

The highest phytate concentration in the seeds was obtained in both soils when subjected to the treatments with limestone and olivine melilitite at the dose of 10 t ha⁻¹ (the latter only in the Nitisol) compared to the others, not differing statistically from each other. The high phytate concentration in the seeds may be directly related to the P contents available in the soil (Taliman et al., 2019) and its accumulation in TDM and later translocation and accumulation in the seeds (Tables 3, 4 and 5, respectively). This interpretation is reinforced by the high positive correlation (Cambisol: 0.50 to 0.99, except for tephrite, with $r = 0.12$; Nitisol: 0.54 to 0.97) observed between the available P (Table 3) in each soil and the treatment with phytate in the seeds. Although there was a high correlation, its contents were constant and below ideal (0.5-0.6%) in most treatments of both soils.

The Cambisol had similarity and high phytate contents in the seeds produced by plants subjected to treatments with manure pure and associated with siltstone + olivine melilitite at the highest dose. The lowest contents, on the other hand, were found in the control, siltstone at the three doses, manure + siltstone at the first dose, and tephrite at doses of 5 and 10 t ha⁻¹, not differing statistically from each other. The phytate content in the Nitisol presented no statistical difference in the treatments with siltstone at the dose of 10 t ha⁻¹, olivine melilitite at the dose of 5 t ha⁻¹, and manure pure and associated with siltstone + olivine melilitite at the two used doses. The other treatments did not differ from the control (Table 4).

According to Coelho et al. (2002), the variation in phytate contents in the seeds may occur due to the CP concentration. The authors found that the high correlation between P and CP was observed in treatments that received the highest P doses. Similar behavior occurred in this study, which showed a high correlation between these variables in the treatments with olivine melilitite and its mixture with siltstone at both doses used in the Cambisol ($r = 0.98$ and 0.92 , respectively), which presented high available contents of this nutrient (Table 3). On the other hand, no correlation was observed between phytate and CP in most of the treatments for both soils, which may be due, according to these authors, to the absence of variation in the CP contents, as also evidenced in this study (Table 4).

Nitrogen, in addition to K, was the nutrient most required by plants, directly reflecting the composition of soybean seeds. However, no statistical difference was found between treatments, regardless of the soil, treatment, and dose (Table 4). The absence of statistical difference between N contents observed in all treatments may be an indication that probably the biological N fixation, provided by seed inoculation, was efficient to nourish the plants satisfactorily, maintaining the leaf (data not shown) and seed contents within the adequate limit recommended by Tedesco et al. (1995) and Vargas et al. (2018).

The similarity between CP contents evidenced in the control treatments with those that received limestone and rock dust (Table 4) may have occurred because a large part of the N in the seeds is present as free amino acids or other non-protein compounds (Silva et al., 2011). However, these values were lower than those found by Santos et al. (2010) and Bavia et al. (2012). According to the first authors, these differences may be related to plant genotypes, environmental conditions, planting sites, and growing seasons. The correlation between Pt and CP contents separately for each treatment and soil showed positive values for Cambisol (0.26 to 0.99) and Nitisol (0.1 to 0.80), indicating that Pt contents were associated with an increase in the CP content of seeds, as also found by Silva et al. (2011).

Although the results show a statistical similarity among the contents of nutrients in the soybean seeds (Table 4) produced in the control treatments and those subjected to pure siltstone and tephrite relative to the other pure products associated or not with manure at different doses, these rocks may not have been effective in releasing nutrients to the soil and, consequently, to the plants compared to the control. This interpretation is supported by the tendency to increase the chemical composition and accumulation of nutrients in the seeds even though no statistical difference was observed between the contents of nutrients and their accumulation in the seeds produced by soybean plants subjected to the treatments with pure siltstone and tephrite compared to those obtained in the control (Tables 4 and 5). This difference may be due to the higher TDM production and, consequently, the higher concentration of nutrients in the TDM, which were possibly translocated to the seeds.

The similarity between the control, pure siltstone, and tephrite treatments may be related to the nutrient dilution (Colombari, Lanna, Guimarães, Martins, & Cardoso, 2018; Cunha et al., 2019) in the plants cultivated in soils managed with limestone, olivine melilitite, and siltstone + olivine melilitite associated or not with manure at their respective doses, which presented the highest TDM productions (Table 5) compared to the

other treatments of pods and seeds (data not shown). In addition, the low TDM production may have led to a higher nutrient concentration, but statistically similar to the control (Table 5), in the different parts of the plant cultivated in the soils subjected to the treatments with the rock dusts siltstone and tephrite. The plants probably accumulated more nutrients and used them little for dry matter (Table 5), pod, and seed production and their physiological functions, being fully translocated to the seeds.

In this sense, the treatments subjected to pure manure and its association with rock dusts stood out. In general, the use of manure at different doses and in both soils increased satisfactorily both the TDM production in plants and the accumulation and composition of nutrients in the seeds, maintaining the values close to and even higher than those observed in the conventional fertilization (Tables 5 and 4, respectively). Thus, the addition of manure with rock dusts may have partially favored an increase in the solubilization and subsequent release of the nutrients present in their structures for the soil solution, increasing their concentrations and providing a high absorption by the plants, directly influencing the TDM production and the chemical composition of seeds. According to Silva et al. (2011) and Tavares et al. (2018), the increased release of nutrients with the use of organic sources may occur due to changes in the soil pH, providing higher activity of the microbiota present in the organic sources and the stimulation of the soil microbiota with the application of these compounds.

The interpretation that the use of rock dusts associated with organic sources can enhance their solubilization is supported by some scientific studies. In this context, Ferreira et al. (2009) evaluated the nutrition of bean plants grown on a Humic Cambisol fertilized with basaltic rock dust associated or not with cattle manure and concluded that the association between them had the potential to increase the bean yield compared to the isolated use of this rock dust, as the microorganisms present in the organic material provided an increase in the rock solubilization. Silva et al. (2011) found that an increase in the proportion of rock dusts increased the P content of seeds, but the phytate content remained constant. Wolschick et al. (2016) and Tavares et al. (2018) concluded that the association of rock dusts with organic compounds can potentiate the beneficial effects of rocks, improving the soil chemical characteristics by increasing the release of nutrients, leading to direct increase in plant productivity.

Although the contents of most of the nutrients in the evaluated soils were characterized as low (Table 3) according to CQFS-RS/SC (2016), we cannot infer that the plants manifested symptoms of nutritional deficiency throughout their cycle, as the nutrient contents in the leaves (data not shown) and seeds were within the sufficiency range recommended by Tedesco et al. (1995) and Vargas et al. (2018), respectively, who evaluated the nutritional composition of seeds from 2453 soybean genotypes, indicating their satisfactory nutrition during all the stages of development.

Therefore, we suggest the conduction of field experiments in soils with characteristics similar to those used in this study to evaluate the plant productivity and chemical composition of seeds subjected to the treatments with pure or combined rock dusts (1:1 proportion) and their association or not with other organic sources.

Conclusion

The effect of pure rock dusts combined with manure was directly related to the chemical characteristics of the evaluated soils. The response of these products was more evident in the Cambisol (poorer soil) than in the Nitisol (more fertile soil).

The contents and accumulation of nutrients in the seeds, as well as the total dry matter production in the two 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 with 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 and total dry matter production.

The products tested in this study were effective in the nutrition of plants at their different stages of development, directly influencing the TDM production and chemical composition of seeds. The rock dust pure olivine melilitite and its combination with siltstone (or other rock dusts) and cattle manure stood out as being a promising source for the supply of nutrients to soil and plants.

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