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The potential of alkaline rocks from the Fortaleza volcanic province (Brazil) as natural fertilizers

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ABSTRACT

This research evaluates the potential of three alkaline rock types to be used as soil remineralizers by measuring the availability of macronutrients and micronutrients in aqueous media and soils after a three-month incubation period. The lithotypes used were (1) phonolite; (2) nepheline syenite; and (3) volcaniclastic breccia, which are from the metropolitan area of Fortaleza, capital of Ceará state, northeast Brazil. All rock types are constituted of alkali feldspar, aegirine-augite, nepheline, kaersutite with minor apatite, and opaque minerals in different proportions. These minerals carry substantial amounts of K, Ca, Mg, P, Fe, Mn, Zn, and Cu, elements that are important for plant nutrition and metabolism. The concentration of the major and trace elements in the rocks was determined to quantify the efficiency of the solubilization relative to the element's original content in the rock. The agronomic tests were conducted as a completely randomized design, which results were submitted to variance analysis. Several important elements were efficiently solubilized by the leaching experiment with citric acid diluted to 2%, indicating the potential of the studied materials to be effectively used as natural fertilizers. A typical surface soil from the region was incubated at room temperature with finely ground nepheline syenite and volcaniclastic breccia for three months. The amendment was applied at rates of 0, 2, 4, 6, 8, and 10 t ha^{-1} to cover situations of moderate to intensive application rates. The incubation results with the treatment using the volcaniclastic breccia indicate a high potential for soil remineralization of P, Fe, and Mn, while the nepheline syenite yields potential for P, K, Mn, Zn, and Cu. The outcomes of this research in laboratory-scale have important implications for fertilizer and soil management and should be tested in the field, including rock blending and long-term experiments to evaluate the effects of soil's improvement with time.

1. Introduction

The importance of sustainable practices in modern agriculture and organic farming is quickly gaining importance among developed and developing countries (Leonardos et al., 2000; Shivay et al., 2010), and the need for alternatives to chemical fertilizers every day becomes more essential. Finding local alternatives to chemical fertilizers is not only important to countries like Brazil, where agriculture is a significant part of the country's economy and is highly dependent on imported chemical fertilizers (up to 73% of the consumption is imported – ANDA, 2019), but also to any country pursuing sustainable agriculture.

The traditional NPK chemical fertilizer requires high application rates in tropical climates of increased rainfall. The phosphate that is not readily used by plants cannot be stored in soil solution for later use and it is either transported out to the drainage system, or it is made unavailable through mineral adsorption (Leonardos et al., 2000). The leachable tendency is even more pronounced for potassium, which is available in chemical fertilizers as KCl. Also, other essential micronutrients such as Mn, Zn, Fe, and Cu may play an essential whole and act as limiting production factors and plant growth (Shivay et al., 2010 and references therein).

One of the main alternatives to the use is of chemical fertilizers is the stonemeal technique, also known as rock mineralizers or rock mineral flour, which has been the object of several recent studies (Theodoro, 2000; Andrade et al., 2002; Almeida et al., 2006; Theodoro and Leonardos, 2006; Beneduzzi, 2011; Nunes, 2012; Cola and Simão, 2012; Ramos et al., 2014, 2015; 2017; Reis, 2015; Borges and Souza, 2015; Medeiros, 2017). The stonemeal technique uses milled rock powder as

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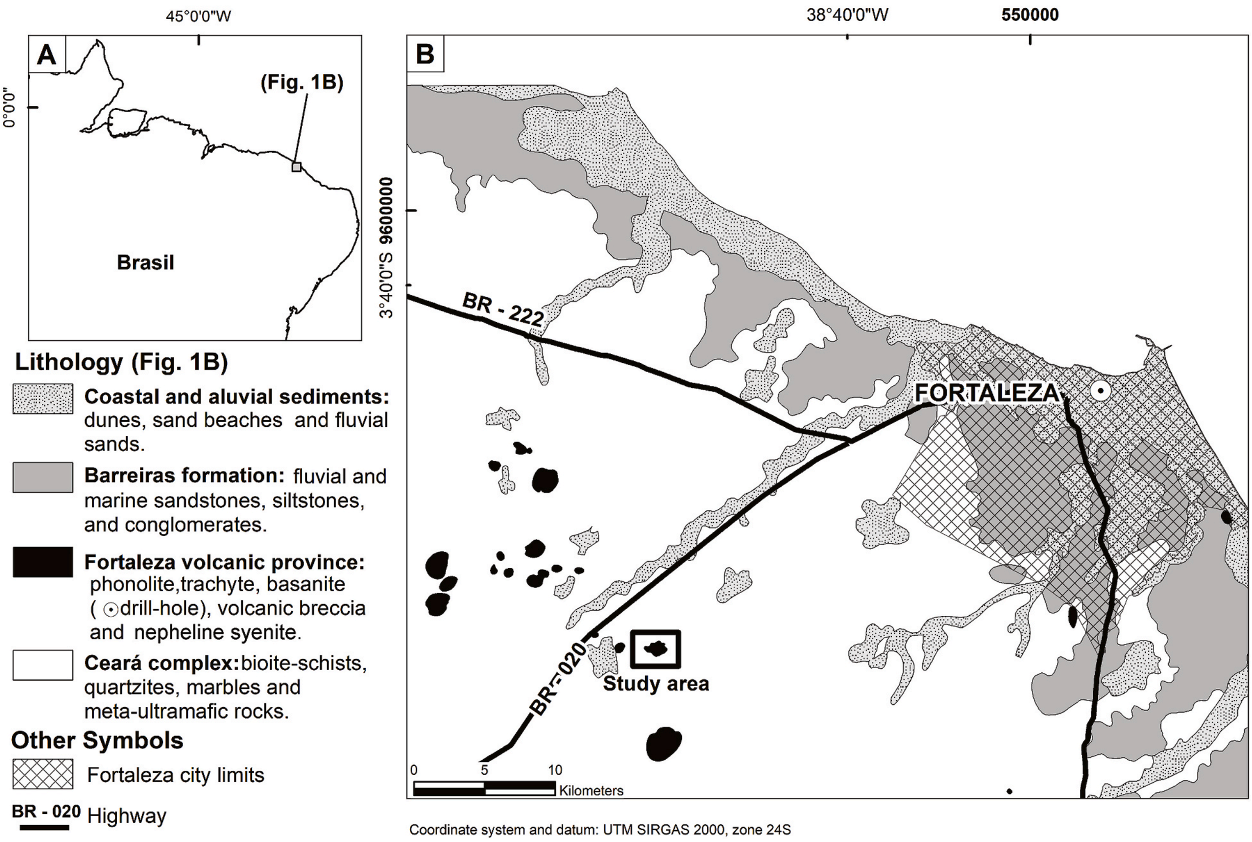

Fig. 1. (A) Location map of the study area on a continental scale. (B) Simplified geological map, highlighting the study area (black rectangle) and the rocks of the Fortaleza Volcanic province (Modified from Cavalcante et al., 2003).

natural soil remineralizers. This technique promotes the slow release of a variety of nutrients to soil and plants (K, P, Fe, Mn, Zn, Cu), which improve fertility, reduce production costs and has long last effects in comparison to the use of chemical fertilizers (Theodoro, 2000) which are normally applied in a yearly basis. Therefore, the evaluation of local rocks wastes with the potential to act as a natural fertilizer is essential for more environmentally friendly, and sustainable agriculture with a lower CO_2 footprint.

The use of alkaline volcanic rocks as soil remineralizer in tropical climate soils have been recently accessed in different locations of Brazil (Leonardos et al., 1987, 2000; Harley and Gilkes, 2000; Theodoro and Leonardos, 2006; Ramos et al., 2017). These rocks contain major quantities of macronutrients and micronutrients important to plant growth and soil fertility such as P, Ca, Mg, Si, Fe Mn, Zn, Cu (CETEM, 2010; Pádua, 2012; Crusciol, 2008) and have the potential of reducing soil acidity (Hildebrand, 1991; Fakengrem-Gerup and Tyler, 1992). Such volcanic rocks occur at the metropolitan region of Fortaleza, the capital city of Ceará state in northeast Brazil, with a current population of approximately 3 million people, with several cashews, coconut and other fruit crops in a short to medium distance radius (50–100 km). The evaluation of the potential of these alkaline rocks as natural remineralizers can, therefore, offer a local, sustainable, and organic alternative to chemical fertilizers.

This research provides new information on the mineralogical characterization of three rock types occurring at the metropolitan region of Fortaleza (Fig. 1A and B) by evaluating the potential of available nutrients in aqueous media dissolution and soil incubation experiments. In addition, we included a commercial natural fertilizer on the experiments for comparing the results with a widely distributed product.

Table 1
Rock types, UTM coordinates, and description of the studied volcanic and plutonic rocks.

Rock Type	Coordinates (X/Y)	Description
Phonolite	523913/ 9569170	Microporphyritic gray volcanic rock, constituted of micro phenocrysts up to 5 mm made of alkali feldspar, nepheline, aegirine-augite, and locally kaersutite within an aphanitic matrix composed of alkali feldspar, aegirine-augite, and nepheline.
Nepheline syenite	523290/ 9569279	Fine grained, phaneritic leucocratic rock composed of alkali feldspar, nepheline, and kaersutite. Locally contains secondary carbonate and clay minerals from weathering processes.
Volcaniclastic breccia	523642/ 9569145	Polymictic volcaniclastic breccia with fragments up to 40 cm within aphanitic matrix which is often weathered to clay minerals. The breccia clasts are composed of lithic fragments from rocks of the volcanic province and its basement. Secondary carbonate minerals occur in veins or filling cavities within the breccias.

2. Materials and methods

2.1. Study area, samples, and reference materials

All materials included in this study are alkaline rocks from the Fortaleza volcanic province, located in the northeast portion of Brazil (Fig. 1A). The volcanic rocks occur as circular to slightly elongated bodies (Fig. 1B) that crosscut the migmatites and gneisses of the Ceará Complex (Cavalcante et al., 2003). This province is mainly composed of alkaline volcanic rocks, minor basic volcanic, and volcaniclastics. It is interpreted as the continental extension of the alignment of seamounts, guyots, and volcanic islands, which include Fernando de Noronha Archipelago, Rocas Atoll, and Ceará Plateau (Almeida, 1955; 1961; 2006). There are also associated intrusive rocks of intermediate composition, such as the nepheline syenite which was used in this study. The other major lithological units in the region are the sedimentary rocks of the Barreiras Formation and the coastal and fluvial quaternary sediments.

Three types of rocks from the Fortaleza volcanic province have been selected for this study, as they all outcrop within a few hundred meters from each other and could potentially be mixed for creating a better product depending on individual results. These are (1) phonolite; (2) nepheline syenite; (3) volcaniclastic breccia. The location and description of the sampled rocks are listed in Table 1.

Additionally, we added to the experiments two well-known materials which are often used in agriculture as a remineralizer agent or a pH neutralizer: (1) Commercial product known as MB4, which is a blend of biotite-schist and serpentinite powders in 1:1 ratio, developed by MIBASA (Mineração Barreto S.A.) from Arapiraca, State of Alagoas, Brazil. It is used as a remineralizer with slow nutrient disposal into the soil. MB4 chemical composition is: 39,73% of SiO_2; 17,82% of MgO; 5,90% of CaO; 0,84% of K_2O; 0,075% of P_2O_5; 0,18% of S; 7,10% of Al_2O_3; 6,86% of Fe_2O_3; 1,48% of Na_2O; 0,074% of Mn; 0,029% of Cu; 0,029% of Co and 0,03% of Zn (Pinheiro and Barreto, 1996; Santos et al., 2011) (2) Limestone, which is used for acidic soil pH neutralization.

2.2. Mineralogical studies

To identify the main and accessory mineral phases of the three selected rock types, we have performed transmitted and reflected light petrography of 19 polished thin sections using a Nikon Eclipse microscope. Additional scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS) were used for observing rock textures and mineral intergrowths, and to acquire semiquantitative compositions of minerals which compose the fine-grained matrix of the volcanic rocks. The analyses were done using a *Quanta* 450 FEG - *FEI* SEM with an attached EDS detector at the Universidade Federal do Ceará.

2.3. Chemical analysis procedures

Multielementary bulk-rock analyses of the three studied rock types were realized by SGS Geosol Laboratories Ldta., Belo Horizonte, according to the following procedures: (1) Loss on ignition (LOI) by calcination; (2) lithium-tetraborate fusion and X-ray fluorescence (XRF) determination of major elements (Si, Al, Ti, Fe, Mn, Mg, Ca, Na, K, and P). (3) Aqua regia digestion and quantification of Ag, As, B, Cd, Co Cr, Cu, Mo, Ni, Pb, Se, Th, U, S, and Zn ICP-MS (inductively coupled plasma mass emission spectroscopy). (4) Hg determination by CVAAS (cold vapor atomic absorption spectroscopy).

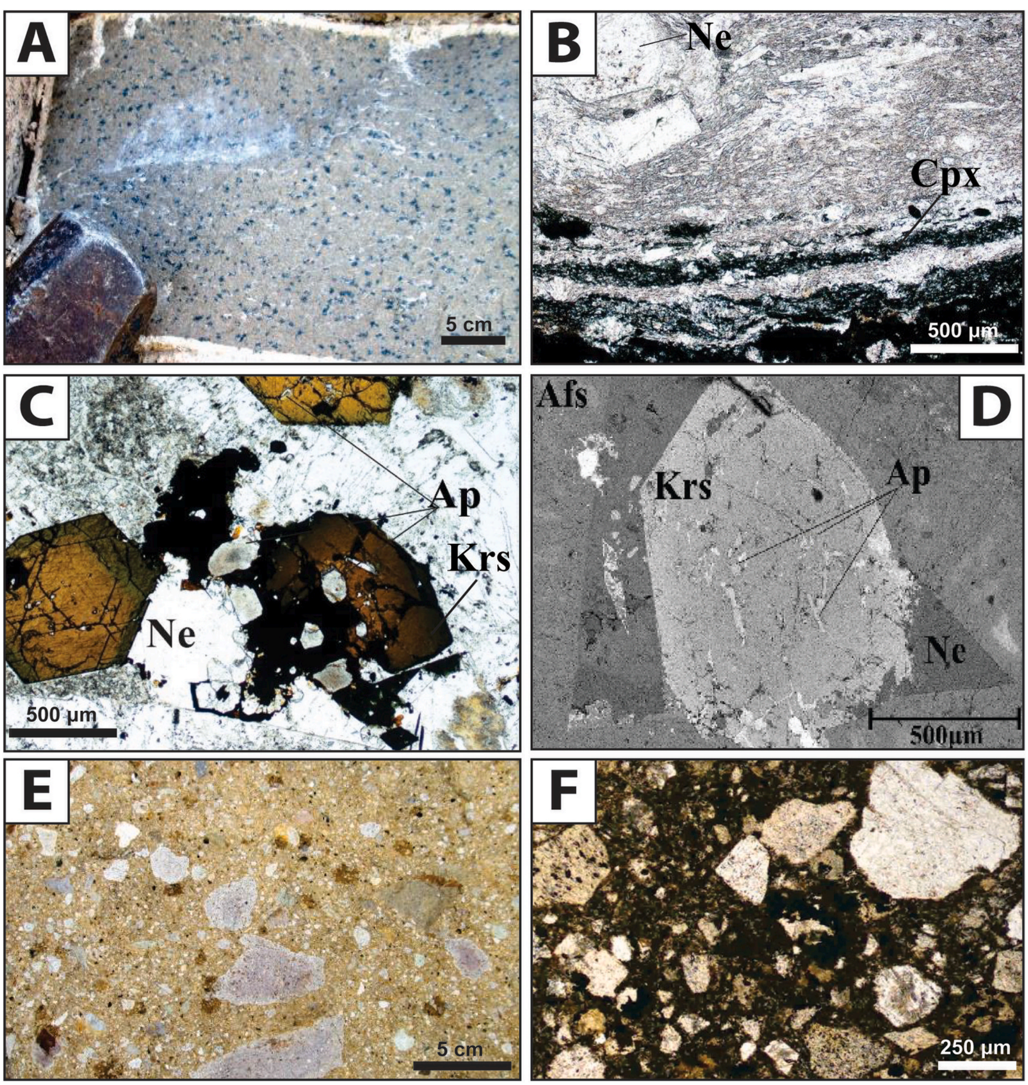

Fig. 2. Textural and petrographic aspects of the studied rocks. (A) Hand specimen of the fine-grained phonolite, the dark minerals are aegirine-augite. (B) Photomicrograph of the phonolite, showing its main mineralogy made of aegirine-augite (Cpx), alkali feldspar (white portions), and nepheline (Ne). (C) Photomicrograph of the nepheline syenite, showing euhedral crystals of kaersutite (Krs) with inclusions of apatite (Ap) and nepheline (Ne) within an alkali feldspar groundmass. (D) Backscattered electron microscope image showing the detail of the various apatite crystals (Ap) included in euhedral kaersutite (Krs) with associated alkali feldspar (Afs) and Nepheline (Ne). (E) Macroscopic aspects of the volcanoclastic breccia with a fine-grained matrix and fragments of various volcanic rock lithologies. (F) Photomicrograph of the volcaniclastic breccia, emphasizing the extremely fine-grained nature of the matrix.

2.4. Experiments

For evaluating the agronomic potential of the studied rocks, we have subjected the specimens to nutrient dissolution in aqueous media and incubation experiments. All experiments, aqueous media, and soil analysis were performed at the Soils Laboratory of Embrapa Agroindústria Tropical located in Fortaleza. For the ICP-OES (inductively coupled plasma optical emission spectrometry) analysis, we used an Agilent 5100 equipment according to the operating parameters: power: 1.1 kW; plasma gas flow: 12 L/min; auxiliary gas flow: 1.0 L/min; spray chamber type: glass cyclonic (single-pass); torch: standard one-piece quartz axial; nebulizer type: OneNeb nebulizer; flow: 0.7 L/min; pump speed: 12 rpm; replicate read time: 5 s; number of replicates: 3; sample uptake delay time: 10 s; stabilization time: 5 s; background correction: fitted. Analyzed elements and wavelength (nm): Al, 396.152; As, 228.812; B, 249.772; Ca, 318.127; Cd, 226.502; Cr, 283.563; Cu, 327.395; Fe, 238.204; Hg, 194.164; K, 769.897; Mg, 279.553; Mn, 257.610; Na, 588.995; Ni, 216.555; P, 213.618; Pb, 220.353; S, 181.972; Se, 196.026; Si, 288.158; and Zn, 213.857.

2.4.1. Nutrient's dissolution in aqueous media

The analysis of nutrient's dissolution in aqueous media was conducted in four different materials, which included the three rocks types (phonolite, the nepheline syenite, and the volcaniclastic breccia) and the MB4. The experiment was conducted as a completely randomized design, with five repetitions. All materials were crushed in a jaw crusher and pulverized in a pan mill into the fraction passing to 200 mesh. The extraction was done by homogenizing 1g of rock powder and 100 mL of citric acid at 2%, following procedures defined by MAPA, 2014. The choice citric acid for the nutrient release study is ideal, due to its capacity of simulating the soil/root environment, as this acid is usually released by the roots of the plants and occur in high concentration at the rhizosphere (Song and Huang, 1988; Ramos et al., 2014). The homogenization was achieved by using an analogic Wagner rotary agitator for 30 min at 35 rotations per minute (rpm).

2.4.2. Incubation study

Incubation experiments were conducted on the nepheline syenite and the volcaniclastic breccia, as these rock types had the most promising results from the nutrient's dissolution experiments. Additionally, the experiments were done using limestone and the MB4 as reference materials. The duration of the incubation period was three months, from the 24th of July to the 24th of October of 2018. The experiments were conducted as a completely randomized design, executed in a factorial scheme ($2 \times 6 + 2$), including four repetitions, totaling 56 samples: two rocks types and six rates (0 – control, 2, 4, 6, 8, and 10 t ha^{-1}) and two additional treatments with the MB4 at the manufacturer recommended rate (2 t ha^{-1}) and the limestone at the rate of 2.11 t ha^{-1}, enough to increase the soil base saturation up to 60%.

We have selected an Arenic Haplustults from the region of Maracanaú, Ceará, nearby the location of the potential rocks being studied. The soil's technical parameters are as follow: pH (water): 4.6; organic matter: 9 g kg^{-1}; clay content: 69 g kg^{-1}; silt content: 105 g kg^{-1}; sand content: 826 g kg^{-1}; available-P: 6 mg dm^{-3}; K^+: 1.1 $mmol_c$ dm^{-3}; Ca^{2+}: 1.3 $mmol_c$ dm^{-3}; Mg^{2+}: 2.5 $mmol_c$ dm^{-3}; Na^+: 0.3 $mmol_c$ dm^{-3}; H+Al: 38.8 $mmol_c$ dm^{-3}; Al^{3+}: 10 $mmol_c$ dm^{-3}; sum of bases (SB): 5.1 $mmol_c.dm^{-3}$; cation exchange capacity (CEC): 44 $mmol_c$ dm^{-3} and base saturation (SB): 12%.

The incubation experiments obeyed the following procedures: (1) the homogenized mixture of soil and the specific rate for each treatment was placed in an unsealed plastic container with a capacity of 0.3 dm^3 (2) De-ionized water was adder in enough quantity to increase soil humidity at 70% of the soil's water retention capacity. (3) Water reposition was done based on the container's weight loss, which was checked every 03 days. During the incubation test, the samples were not exposed to the sun but stored in a room with a climate-controlled temperature between 20 and 24 °C. After an incubation period of 90 days, the samples were dried in a forced-air circulation oven at 45 °C, ground, and sieved to a grain size of 2 mm (10 mesh).

Table 2
Main crystalline phases that constitute the studied volcanic and plutonic rocks.

	Phonolite	Nepheline syenite	Volcaniclastic breccia
Primary rock forming minerals (%)			
Quartz (SiO2)	–	–	[a]
Alkali feldspar ($(Na,K)AlSi_3O_8$)	50–55	47–58	[a]
Aegirine-augite ($(Na,Ca)(Mg,Fe^{+2},Fe^{+3})Si_2O_6$)	20–25	<2	[a]
Nepheline ($(Na,K)AlSiO_4$)	8–10	10–15	[a]
Albite ($NaAlSi_3O_8$)	<1	6–10	[a]
Kaersutite ($NaCa_2(Mg_4Ti)Si_6Al_2O_{23}(OH)_2$)	<1	8–13	[a]
Hornblende ($(Ca,Na)2(Mg,Fe,Al)5(Al,Si)_8O_{22}(OH)_2$)	–	<4	–
Biotite ($K(Mg,Fe)_3AlSi_3O_{10}(OH)_2$)	–	<1	–
Apatite ($Ca_5(PO_4)_3(F,Cl,OH)$)	3–5	2–7	[a]
Zircon ($ZrSiO_4$)	0–1	<1	[a]
Titanite ($CaTiOSiO_5$)	0–1	<1	–
Rutile (TiO_2)	–	<1	[a]
Calcite ($CaCO_3$)			[a]
Opaques (Hematite, ilmenite, pyrite)	0–5	3–10	[a]

[a] Indicates the presence of the mineral only. Mineral proportions are not possible to be determined for the volcaniclastic breccia due to its extreme fine-grained texture.

The micronutrient extraction was done using a DTPA (diethylenetriaminepentaacetic acid) solution (Raij et al., 2001) quantified by ICP-OES. All soil analyses and standardization procedures were performed according to procedures described in Silva et al. (1998). The data were submitted to variance analysis and linear regression, using the AgroEstat software (Barbosa and Maldonado Júnior, 2015).

3. Results and discussion

3.1. Mineral phases

The phonolite is made of well-formed microphenocrysts (up to 2 mm) of sanidine and anorthoclase (30%) within a fine-grained matrix (Fig. 2A). The matrix is composed of alkali feldspar (50–55%), aegirine-augite (20–25%), and nepheline (8–10%). Minor albite may occur within the boundary of alkali feldspar grains. Other accessory minerals comprise apatite (<5%), kaersutite (<1%) opaque minerals (hematite, ilmenite, and pyrite, < 2%), titanite and zircon (both < 1%). The identification of alkali feldspar, aegirine-augite, and nepheline as the major crystalline components indicates the sodic affinity of the rock. It is important to highlight the significant concentration of aegirine-augite (Fig. 2B) and nepheline, both minerals which are easily weathered when compared to feldspar minerals (Wilson, 2004).

The nepheline syenite is constituted by well-formed, up to 5 mm size, and equigranular crystals of alkali feldspar (47–58%), nepheline (10–15%), albite (6–10%), kaersutite (8–15%), hornblende (≤4%), augite (<2%), opaque minerals (hematite, magnetite, and pyrite), apatite (2–7%), with minor (<1%) zircon, rutile, and titanite (Fig. 2C). Kaolinite, sericite, calcite are the main secondary minerals that occur as weathering products of alkali feldspars and nepheline. The major rock-forming minerals of the nepheline syenite are similar to the phonolite, with alkali feldspar as the main crystalline phase, but with a major contribution of kaersutite, albite, and a smaller proportion of aegirine-augite. The relatively high amount of apatite in both phonolite and nepheline syenite (Fig. 2D) is also important as a major P source.

The volcaniclastic breccia is formed by rock fragments that are involved by a tuff-like matrix, which forms from 80 to 60% of the rock

Table 3
Chemical composition of major elements and trace elements of the studied volcanic and plutonic rocks.

Oxide*/Element	Phonolite	Nepheline syenite	Volcaniclastic breccia
	mg kg⁻¹		
SiO_2	579,000	535,000	578,000
Al_2O_3	193,000	199,000	170,000
MgO	4200	12,900	15,000
CaO	18,300	41,700	33,300
Na_2O	67,800	64,100	22,400
K_2O	66,200	53,000	48,500
P_2O_5	1120	4930	2760
S	100	<100	2100
Ti	3957	9053	6055
Fe	25,318	42,314	32,732
Mn	1317	1704	1472
Hg	<0.05	<0.05	<0.05
Ag	<0.01	<0.01	<0.01
As	<1	<1	2.0
B	<10	<10	<10
Cd	0.1	0.1	0.2
Co	1.3	3.6	6.6
Cr	3.5	<1	10.0
Cu	1.6	1.2	7.2
Mo	4.9	6.7	1.4
Ni	1.9	<0.5	11.2
Pb	4.2	2.2	2.4
Se	<1	<1	<1
Th	15.5	12.7	6.7
U	3.3	3.4	0.6
Zn	88	92	106

(Fig. 2E). The rock fragments can go up to 30 cm in size, which are formed by alkaline volcanic rocks such as phonolites, country-rock (migmatites and gneisses), volcanic glass, and mineral fragments (quartz, feldspar, and biotite). Apatite, zircon, rutile, and opaque minerals (hematite, pyrite, and ilmenite) are the main accessory minerals. Due to the extremely fine-grained nature of the volcaniclastic breccia's matrix (Fig. 2F), it was not possible to determine the relative proportion between the major rock-forming minerals of this rock type.

Table 2 summarizes the main crystalline phases identified in the three studied rock types.

3.2. Geochemical analysis and source of nutrients

The major and minor components of the three investigated rocks are listed in Table 3. All three rocks types have Si, Al, Na, K, and Fe as major constituents, with minor amounts of Mg, Ca, P, Ti, and S. The composition reflects the rock's mineralogy and the variable proportions between alkali feldspars, feldspathoids (e.g. nepheline), pyroxenes (aegirine-augite), amphiboles (kaersutite, hornblende), and important accessory minerals such as ilmenite and apatite.

The composition of both the nepheline syenite and the volcaniclastic breccia is within the suggested specifications (MAPA, 2016) for soil remineralizers, which requires that the sum of CaO, MgO, and K_2O is above 9% (90,000 mg kg^{-1}), with K_2O concentration above 1% (10,000 mg kg^{-1}). Regarding potentially toxic elements, the values within the 3 rock types range from 10 to 100 times less than the maximum accepted values (As < 15 mg kg^{-1}, Cd < 10 mg kg^{-1}, Hg < 0.1 mg kg^{-1}, and Pb < 200 mg kg^{-1}). Other elements that could be considered as potentially toxic elements such as U (0.6–3.4 mg kg^{-1}) and Th (7–15 mg kg^{-1}) occur in relatively low concentrations when compared to ordinary rocks but there are no maximum specified values for the concentration of these elements.

The P_2O_5 content of the three rock types varies between 1120, 2760 and 4930 mg kg^{-1} and falls below the recommended concentration values (MAPA, 2016) for listing this element as a macronutrient (>10,000 mg kg^{-1}). Other important macronutrients such as MgO (4200–15,000 mg kg^{-1}) and sulfur (<100-2100 mg kg^{-1}) are of relatively low concentration within these rocks.

Essential micronutrients and other elements important for plant nutrition are present and include Co (3.6–6.6 mg kg^{-1}), Cu (1.2–7.2 mg kg^{-1}), Ni (up to 11 mg kg^{-1}), Zn (88–106 mg kg^{-1}), Fe (25,000–42,000 mg kg^{-1}), Mn (1300–1700 mg kg^{-1}), Mo (1.4–6.7 mg kg^{-1}), and Cr (up to 10 mg kg^{-1}). The concentrations of Fe and Mn on all three rock types are respectively up to 42 and 1.7 times the minimum values required by MAPA (2016) for soil remineralizers.

3.3. Nutrient's dissolution in aqueous media

The results of the nutrient's dissolution in aqueous media are listed in Table 4, which include the three studied rock types from the Fortaleza volcanic province and the MB4. For comparison purposes, published values (Nunes et al., 2014; Ramos et al., 2017) of nutrient dissolution in

Table 4
Levels macronutrients and micronutrients available in extractor solutions for treatments with different volcanic and plutonic rocks.

	Phonolite				Nepheline syenite				Volcaniclastic breccia				MB4				Nunes et al. (2014)	Ramos et al. (2017)	
	Soluble			%	*Soluble*			%	*Soluble*			%	*Soluble*			%			
Si	4377	±	402	1.6	4456	±	245	1.8	556	±	28	0.2	1892	±	247	1.0	n.a.	n.a.	n.a.
Al	23,422	±	193	23	16,013	±	417	15	760	±	40	0.8	1140	±	113	3.0	0.43	541	762
Na	18,566	±	82	36	12,267	±	320	26	380	±	20	2.3	279	±	26	2.5	0.2	276	331
Ca	5887	±	56	45	14,323	±	214	48	20,603	±	1047	87	50,905	±	5142	100	1000	394	1109
Fe	2045	±	61	8.1	1123	±	69	2.7	8590	±	411	26	4563	±	467	10	346	897	1342
K	1323	±	43	2.4	2282	±	78	5.2	262	±	50	0.7	279	±	49	4.0	152	256	385
P	408	±	13	80	863	±	31	40	402	±	8	33	86	±	10	25	100	318	348
S	97	±	14	97	137	±	18	b.d.l.	174	±	9	8.3	461	±	54	26	1.4	n.a.	n.a.
Mn	599	±	7	46	549	±	21	32	779	±	43	53	501	±	47	52	3	159	198
Mg	377	±	3	15	1503	±	29	19	4925	±	209	54	16,216	±	545	15	1	247	341
Zn	43	±	31	49	78	±	76	85	100	±	33	94	38	±	24	36	1.6	0	0
Cr	19	±	0.9	100	21	±	0.8	b.d.l.	12	±	0.9	100	37	±	5	n.a.	n.a.	3	4
B	7	±	3	b.d.l.	5	±	1.0	b.d.l.	8	±	0.8	b.d.l.	6	±	0.9	n.a.	0.2	1	1
Cu	3	±	0.5	100	4			100	3	±	1	47.2	5	±	1.1	2	8.4	8	12
Se	3	±	0.4	b.d.l.	3			b.d.l.	3			b.d.l.	2	±	0.5	n.a.	n.a.	n.a.	n.a.
Ni	2			100	1			b.d.l.	5			43	175	±	20	n.a.	n.a.	0	1
As	<1			b.d.l.	<1			b.d.l.	<1			b.d.l.	<1			n.a.	n.a.	0	0
Cd	<1			b.d.l.	<1			b.d.l.	<1			b.d.l.	<1			n.a.	n.a.	0	0
Hg	1			b.d.l.	1			b.d.l.	1			b.d.l.	1			n.a.	n.a.	0	0
Pb	1.4	±	0.5	33	1.0			45	2.0			83	2.2			n.a.	n.a.	0	1

b.d.l = Values below detection limits on bulk-rock analysis.
n.a = values not available/not analyzed.

Table 5
Levels and standard deviation of pH and exchangeable cations available in the soil after the incubation experiments using treatments with different rates and rock-types.

Rock Type	Rate	pH	± pH	P	± P	K^+	± K^+	Ca^{2+}	± Ca^{2+}	Mg^{2+}	± Mg^{2+}	Na^+	± Na^+	Al^{3+}	± Al^{3+}	SB	± SB
	t ha^{-1}	H_2O		mg dm^{-3}		$mmol_c$ dm^{-3}											
Volcaniclastic breccia	0	4.1	0.1	5.8	0.5	1.3	0.1	5.2	0.7	2.7	0.3	0.2	0.1	2.8	0.1	9.4	1.2
	2	4.2	0.1	6.4	1.1	1.3	0.1	6.0	0.5	3.2	0.3	0.2	0.1	2.6	0.2	10.7	0.9
	4	4.3	0.1	7.1	0.6	1.3	0.0	7.4	0.3	3.8	0.1	0.2	0.1	2.2	0.1	12.7	0.3
	6	4.4	0.1	6.7	0.5	1.2	0.2	8.4	0.8	4.2	0.4	0.2	0.1	1.7	0.3	14.0	1.4
	8	4.5	0.0	6.9	0.1	1.3	0.1	9.6	0.4	4.7	0.2	0.2	0.1	1.6	0.1	15.8	0.6
	10	4.6	0.0	7.6	0.6	1.4	0.1	11.5	0.4	5.6	0.1	0.3	0.1	1.3	0.2	18.7	0.6
Nepheline syenite	0	4.1	0.1	5.8	0.5	1.3	0.1	5.2	0.7	2.7	0.3	0.2	0.1	2.8	0.1	9.4	1.2
	2	4.2	0.1	7.4	0.5	1.4	0.1	5.5	0.3	2.7	0.2	0.6	0.1	2.7	0.2	10.2	0.6
	4	4.3	0.1	10.5	1.3	1.4	0.1	6.3	0.2	2.8	0.1	1.0	0.1	2.5	0.1	11.5	0.5
	6	4.4	0.1	11.3	1.1	1.5	0.1	7.3	0.4	3.0	0.2	1.3	0.1	2.3	0.1	13.2	0.8
	8	4.5	0.0	13.1	1.3	1.6	0.1	8.4	0.4	3.2	0.2	1.6	0.1	2.0	0.1	14.9	0.8
	10	4.4	0.3	15.8	0.9	1.5	0.0	7.6	1.8	3.0	0.3	1.4	0.7	2.0	0.7	13.4	2.9
Limestone	2.1	5.5	0.1	5.5	1.9	1.3	0.1	32.4	1.1	2.5	0.2	0.2	0.1	0.1	0.0	36.5	1.4
MB4	2	4.5	0.1	6.2	1.3	1.3	0.1	8.8	0.3	6.7	0.2	0.2	0.1	1.5	0.2	17.1	0.4

aqueous media experiments at the same conditions using alkaline and basaltic rocks are also listed.

The results on the availability of macronutrients and micronutrients showed that nepheline syenite and the volcaniclastic breccia have a suitable potential for the application as a natural fertilizer, while the phonolite contains high levels of soluble Al^{+3} and Na^+ and needs to be more carefully evaluated. Sodium can benefit plant growth at small concentrations but can lead to salinization and sodification of soils. Aluminum has low geochemical mobility in weathering environments, and its accumulation can become toxic to most plants, reducing crop production and root development (Balbino-Miguel et al., 2010). The nepheline syenite and the volcaniclastic breccia promoted the release of several important macro and micronutrients such as potassium, phosphorous, manganese, iron, and zinc. The results are comparable to or higher than recent studies that evaluated volcanic rocks of basic and acid composition (Nunes et al., 2014; Ramos et al., 2017).

All rocks solubilized between 0.2 and 2% of their silica content, resulting in small concentration values in solution from 500 to 4500 mg kg^{-1}. Despite the similar aluminum content, the phonolite and the nepheline syenite were able to solubilize up to 20x more aluminum than the 1% aluminum solubilized by the volcaniclastic breccia and the MB4. This is due to the high concentration of sodic feldspars in these rocks, which is also responsible for the high values of Na (up to 18,500 mg kg^{-1}) solubilized by the phonolite and the nepheline syenite.

The volcaniclastic breccia also made available the higher concentration of Mg and Ca (up to 20,600 mg kg^{-1}) within the three studied rock types, but with smaller values in comparison to the MB4. Even though, these elements can be considered important by-products as the three studied rock types solubilized up to 20 times more calcium when compared to the basalts studied by Nunes et al. (2014) and acid volcanic rocks studied by Ramos et al. (2017). The experiments using the phonolite solubilized lower amounts of calcium and magnesium when compared to the nepheline syenite and the volcaniclastic breccia, reflecting the differences of the rock's mineralogy. The nepheline syenite and the volcaniclastic breccia contain higher proportions of the less stable hornblende and kaersutite, while the phonolite source of calcium and magnesium is the more stable aegirine-augite, in agreement with the experiment's results. The higher calcium values registered for the volcaniclastic breccia is probably related to the presence of secondary calcite.

Potassium was solubilized in high concentrations in all three-rock types. The volcaniclastic breccia release similar potassium concentrations to the acid volcanic rocks studied by Ramos et al. (2017), which are almost two times higher than basaltic rocks (Nunes et al., 2014). The sample with the nepheline syenite powder solubilized a higher concentration of potassium (2282 mg kg^{-1}), which is up to two times the amount which was released by the phonolite and ten times higher than the MB4 or the volcaniclastic breccia. This highlights the great potential for the use of this rock for fertilization purposes.

The nepheline syenite was able to solubilize the highest phosphorus concentration among the three studied rock types, up to 2 times higher than the phonolite and the volcaniclastic breccia and to 10 times higher than the MB4. These results are also slightly higher than the acid volcanic rocks published by Nunes et al. (2014). Apatite is the most probable source of phosphorous in the rock, corroborating with the rock's mineralogy as it is a mineral that occurs in higher concentration in the nepheline syenite in comparison to phonolite or the volcaniclastic breccia. Additionally, the nepheline syenite contains most of its apatite included in kaersutite, a mineral that is more easily weathered when compared to K-feldspars (Wilson, 2004).

Iron and manganese were also solubilized in high concentrations from all three rock types when compared do basalts or other volcanic rocks. When compared to the, only the volcaniclastic breccia promoted higher iron and manganese solubility (8600 mg kg^{-1} of Fe and 780 mg kg^{-1} of Mn), while the phonolite and the nepheline syenite are considerably lower than the MB4, but still almost two times higher than what is reported for basalts and for acid volcanic rocks (Nunes et al., 2014; Ramos et al., 2017). Manganese levels were similar for the nepheline syenite, the phonolite, and MB4 (approx. 500 mg kg^{-1}), while the volcaniclastic breccia solubilized ca. 1.5 more manganese. The potential for iron and manganese is directly related to low stability ferromagnesian minerals such as aegirine-augite, hornblende, kaersutite, biotite, hematite, and ilmenite, which probably occur in higher proportions within the volcaniclastic breccia.

Important micronutrients were solubilized by the volcanic rocks. In comparison to the MB4, all three rock types solubilized more Zn and Se, similar amounts of B and Cu, and smaller values for S, Cr, and Ni. Boron and zinc were micronutrients that were efficiently solubilized by all three studied rock types. Boron concentration in all three rock types was below the detection limit of 10 mg kg^{-1} and solubilized from 5 to 8 mg kg^{-1}. Zinc was also effectively solubilized with values up to 94% of the original rock content. Copper concentration in the solutions for experiments using all rock types and the MB4 yielded similar results (3–5 mg kg^{-1}), which is about half of the concentration measured in experiments using the acid volcanic waste studied by Ramos et al. (2017) and the basalts wastes studies by Nunes et al. (2014).

The solubilization of potentially toxic elements was below the values specified by MAPA (2016). As and Cd were below the detection limit of 1 mg kg^{-1} within solutions of all three rock types. The values of solubilized Pb range between 1 and 2.0 mg kg^{-1}, while the Hg concentration

Table 6
Levels and standard deviation of micronutrients and potentially toxic elements available in the soil after the incubation experiments using treatments with different rates and rock-types.

	Rate	Fe	± Fe	Mn	± Mn	Zn	± Zn	Cu	± Cu	Ni	± Ni	Cr	± Cr	Hg	As	± As	Cd	± Cd	Pb	± Pb
	t ha^{-1}	mg dm^{-3}																		
Volcaniclastic breccia	0	105	1.8	3.5	0.12	1.3	0.1	0.46	0.20	0.08	0.01	0.02	0.00	<0.1	0.055	0.01	0.02	0.00	1.1	0.02
	2	117	7.1	4.0	0.36	1.3	0.1	0.36	0.07	0.08	0.00	0.02	0.00	<0.1	0.050	0.01	0.02	0.00	1.1	0.09
	4	130	7.9	4.3	0.30	1.2	0.1	0.27	0.05	0.08	0.00	0.02	0.00	<0.1	0.055	0.01	0.02	0.00	1.1	0.07
	6	130	1.1	4.0	0.46	1.2	0.2	0.28	0.06	0.08	0.00	0.02	0.00	<0.1	0.050	0.02	0.02	0.00	1.0	0.12
	8	148	3.1	4.5	0.15	1.3	0.1	0.33	0.03	0.09	0.01	0.02	0.00	<0.1	0.050	0.01	0.02	0.00	1.1	0.03
	10	142	12.8	4.4	0.60	1.2	0.1	0.29	0.06	0.08	0.00	0.02	0.01	<0.1	0.045	0.01	0.02	0.00	1.1	0.09
Nepheline syenite	0	105	1.8	3.5	0.12	1.3	0.1	0.46	0.20	0.08	0.01	0.02	0.00	<0.1	0.055	0.01	0.02	0.00	1.1	0.02
	2	107	2.8	3.8	0.15	1.7	0.2	0.64	0.22	0.08	0.00	0.02	0.00	<0.1	0.045	0.01	0.02	0.00	1.2	0.04
	4	103	4.4	3.8	0.21	1.7	0.1	0.73	0.10	0.08	0.01	0.02	0.00	<0.1	0.045	0.01	0.02	0.00	1.2	0.07
	6	103	9.5	3.8	0.41	1.6	0.2	0.81	0.22	0.08	0.01	0.02	0.00	<0.1	0.050	0.01	0.02	0.01	1.2	0.13
	8	98	0.9	3.7	0.13	1.5	0.1	0.81	0.16	0.08	0.01	0.02	0.00	<0.1	0.045	0.01	0.02	0.00	1.2	0.06
	10	96	4.6	3.5	0.12	1.5	0.1	0.91	0.07	0.07	0.01	0.02	0.00	<0.1	0.055	0.01	0.02	0.00	1.2	0.03
Limestone	2.1	74	1.6	1.4	0.20	0.8	0.1	0.32	0.06	0.05	0.01	0.00	0.00	<0.1	0.05	0.02	0.00	0.00	1.1	0.04
MB4	2	101	6.3	2.5	0.27	1.2	0.3	0.31	0.06	0.17	0.02	0.02	0.00	<0.1	0.05	0.01	0.02	0.01	1.1	0.10

in the solutions was 1 mg kg^{-1} for all samples.

3.4. Incubation experiments

The availability of elements for plants in soil is determined by precipitation, dissolution, adsorption, and redox reactions (Ferreira et al., 2001). The results of the incubation experiments provide information on the variability of the pH, availability of micronutrients and macronutrients, and the occurrence of potentially toxic elements. Tables 5 and 6 summarize soil's content of major cations and micronutrients and potentially toxic elements that would be available in the soil for the different amendments. The results are better visualized by converting the units of exchangeable cations and micronutrients to kg ha^{-1} and subtract the values prior to the treatment application (Table 7). Fig. 3 shows the effect of each treatment for the macronutrients and micronutrients in the soil after the three-month incubation period.

3.4.1. Soil acidity

Both nepheline syenite and the volcaniclastic breccia promoted an increase of the soil's pH (Fig. 3A), but at lower rates when compared to the MB4 and the limestone treatments. The pH increase using the MB4 was approximately 0.5 units while using the limestone the pH increase was 1.5 units using the recommended application rate. Using the same rate of the nepheline syenite and the volcaniclastic breccia, the pH increase was of less than 0.1 pH unit, and only at higher application rates (10 t ha^{-1}), the pH increase was comparable to the MB4. There is a linear increase in pH with increasing the rate for both nepheline syenite and volcaniclastic breccia. The slightly higher pH increase promoted by the volcaniclastic breccia is probably due to the occurrence of calcite, which occurs as a secondary mineral filling cracks and vugs and its dissolution would release, Ca^{2+}, carbon dioxide, and hydroxide. The hydroxide will then combine to H^{+} to cause the observed rise in pH (Ritchey et al., 2016; Raij, 2017).

3.4.2. Available phosphorous

There was an increase in phosphorous availability with an increasing rate of the treatment, with the higher values and rate of increase registered by the amendment with nepheline syenite (Fig. 3B). In comparison with the MB4, almost four times more phosphorous was made available with treatment using the nepheline syenite, both at a rate of 2 t ha^{-1}, corresponding to an increase of about 28% or an equivalent of 3.2 kg ha^{-1} added (Table 7). The available phosphorous with the treatment using the volcaniclastic breccia and the MB4 yielded similar results, with an increase of 10% and 7%, respectively, for the rate of 2 t ha^{-1}. The available phosphorus concentration in soils for the different rock types corroborates with the nutrient's dissolution in aqueous media experiment, where the nepheline syenite was able to solubilize almost the double concentration of phosphorous (863 mg kg^{-1}) in comparison to the volcaniclastic breccia.

3.4.3. Exchangeable cations, macronutrients, and micronutrients

The potassium concentration in the soils was influenced by rocks application (Fig. 3C), but only nepheline syenite registered a linear increase with the increase of the rates, with an added potassium amount up to 25.4 kg ha^{-1} at the highest application rates (Table 7). This result agrees with Gautneb and Bakken (2013), which observed a higher supply for potassium in barley plants for treatments using nepheline-rich rocks in comparison to rocks where alkali feldspar was the main K-bearing phase.

There was also an increase of calcium and magnesium with increasing the rates for both rock treatment, but values for calcium and magnesium were smaller than what observed for the MB4 (Fig. 3D and E).

Even though the nepheline syenite presented high levels of Al on the dissolution experiment, it did not result in elevated aluminum values in soil, with values close to the volcaniclastic breccia (Fig. 3F, Table 7).

Table 7
Additional amounts of basic cations and micronutrients available in the soil, expressed in kg ha^{-1} for the different treatments using alkaline rocks.

Rock Type	Rate	Ca	Mg	K	Na	Al	P	Fe	Mn	Zn	Cu
	t ha^{-1}	Kg ha^{-1}[a]									
Volcaniclastic breccia	**2**	32	11	−0.4	1.1	−2.3	1.1	12	0.9	−0.08	−0.20
	4	87	25	−0.2	1.2	−10	2.5	25	1.4	−0.30	−0.38
	6	128	36	−6.3	0.8	−19	1.8	24	0.8	−0.29	−0.37
	8	174	49	−1.0	1.1	−20	2.1	42	1.9	−0.07	−0.27
	10	253	69	4.9	1.5	−27	3.5	37	1.6	−0.23	−0.34
Nepheline syenite	**2**	116	−1	4.3	10	−1.3	3.2	2	0.5	0.69	0.35
	4	43	2	8.2	18	−5	9.4	−3	0.5	0.79	0.53
	6	85	7	15.2	26	−9	11.0	−3	0.6	0.65	0.70
	8	129	12	25.4	33	−14	14.6	−7	0.4	0.40	0.69
	10	95	8	16.5	1	5	19.8	−9	−0.1	0.29	0.90
Limestone	**2.1**	1090	36	−51	0	−47	−0.7	−32	−7.1	−2.64	−0.92
MB4	**2**	146	203	−50	0	9	0.7	−4	−4.2	−1.11	−0.28

[a] Values for converted to kg ha^{-1} considering a soil layer with 20 cm depth and density of 1000 mg dm-3.

This was a well-known effect for soils with pH higher than 5.8 (Faquin, 2005), and this effect can be enhanced by the interaction with available P at pH conditions close to neutrality (Ferreira et al., 2001).

The sodium concentration showed an increase with increasing of the rates for the nepheline syenite, while the treatment using volcaniclastic breccia did not alter the sodium concentration in the soil (Fig. 3G, Table 7). These results are directly related to what was observe on the dissolution experiments, where the nepheline syenite solubilized up 30 times more sodium than the volcaniclastic breccia.

Halomorphic soils are common in northeast Brazil and more in-depth agronomic testing is recommended to better evaluate the effect of sodium availability with the treatment of using the proposed rocks, such as greenhouse and field trials using crop. There are about 9 million hectares of salt-affected soils in Brazil (Medeiros et al., 2016), predominantly found in the semiarid region of the northeast (Pessoa et al., 2019; Ribeiro et al., 2003), where up to 25% of the irrigated lands were salinized (Gheyi, 2000). Within northeast Brazil, the state of Ceará is the second most affected state by salt accumulation in the soil (Gheyi and Fageira, 1997), with approximately 14,8% of its land is covered by soils associated with problems of sodicity (Pereira, 1983; Mota and Oliveira, 1999). Ribeiro et al. (2010) obtained lower exchangeable sodium rates when applied rock powders of ultramafic alkaline rock and pyroclastic breccia in combination with liming materials. This suggests the need to test different mixtures between nepheline syenite and volcaniclastic breccia with lime as an acidity neutralizer.

Despite the different concentrations of the available bases in soil with treatments using the nepheline syenite and the volcaniclastic breccia, the sum of the total bases is similar for each rock type (Fig. 3H). The main differences are on the availability of Mg^{2+}, which is higher in the volcaniclastic breccia, and Na^{2+}, with higher concentrations on treatments using the nepheline syenite.

Treatments using nepheline syenite and volcaniclastic breccia yield higher concentration in soils for iron when compared to the treatment using the MB4 at the same rate. For the treatment using the volcaniclastic breccia, the iron content in soil increased 11%, the nepheline syenite showed a smaller increase (2% increase), while the MB4 registered a decrease (4%) on iron availability in soil (Fig. 3I, Table 7). Manganese availability increased with treatments using both nepheline syenite (9%) and volcaniclastic breccia (14%), while the treatment MB4 registered a decrease in manganese availably in soils (Fig. 3J). These results agree with the solubility tests where the experiment with the volcaniclastic breccia was able to solubilize higher amounts of iron and manganese when compared to the nepheline syenite and the volcaniclastic breccia. The amount of added manganese in soils was up to 1.0 kg ha^{-1} (Table 7) for amendments using the breccia, highlighting the mineralizing potential of this rock-type.

The pH increase will decrease the availability of copper and zinc in soils (Ferreira et al., 2001). When the soil's pH increases up to 5.5, zinc is adsorbed by hydroxides of aluminum (Kalbasi et al., 1978), iron (Kinniburgh and Jackson, 1982; Pombo and Klamt, 1986) and manganese (Loganathan et al., 1977). This was probably the main cause for the slight decrease in the amount of available zinc for treatments using the volcaniclastic breccia, despite the high dissolution (94%) observed on the nutrient dissolution in aqueous media experiment. On the other hand, the treatment using the nepheline syenite registered an increase of zinc availability for treatments using rates up to 6 t ha^{-1}, which decreased for treatments at moderate rates (Fig. 3K, Table 7). This is an indication that the reduction of zinc levels in the soil might be related to the interaction with the available phosphorous, present in higher concentrations on the nepheline syenite and highlights the importance of the incubation experiments. Saeed and Fox (1979) report that the application of phosphorous in soil increase zinc adsorption, mainly in hydrated aluminum and iron oxides. Copper probably does not suffer from the same effects of interaction with phosphorous as it shows a constant increase in soil availability with the increase of the rate for the nepheline syenite, while the volcaniclastic breccia records a decrease in copper values in soil with increasing the rate of application (Fig. 3L, Table 7).

The concentration of the potentially toxic elements (As, Cd, Hg, and Pb) were not altered with any of the treatments, regardless of rock type or rate (Table 6).

Further tests are required to better evaluate the agronomic potential of the nepheline syenite and volcaniclastic breccia in natural conditions. These would involve long term experiments to assess the effects of soil's improvement with time, as well as define which crops will have the highest yield and growth response from the treatment using the powders from the studied rocks. Future studies may include blends of both rock powders in different ratios and complemented with liming and organic fertilizers. Several researchers obtained positive results including lime materials or biofertilizers in stonemeal treatments as an alternative of soil-friendly farming practices (e.g. Montenegro, 2018; Sékula, 2011; Ribeiro et al., 2010). Thus, the combination of rock and organic fertilizers may satisfy the needs of macro and micronutrients of soil fertility (Theodoro and Leonardos, 2006). Recently, the use of rock powder in combination with wastewater derived from cassava (*Manihot esculentum Crantz*) processing has promoted effective improvements in the chemical quality of the studied soils and plants (Montenegro, 2018). Cassava is one of the major cultures grown in the region, which its residue shows great agronomic potential due to its composition rich in micronutrients and macronutrients, mainly K (Aragão and Ponte, 1995). Therefore, further agronomical testing using such organic wastes would be able to evaluate how much the agronomic effect of the nepheline syenite and

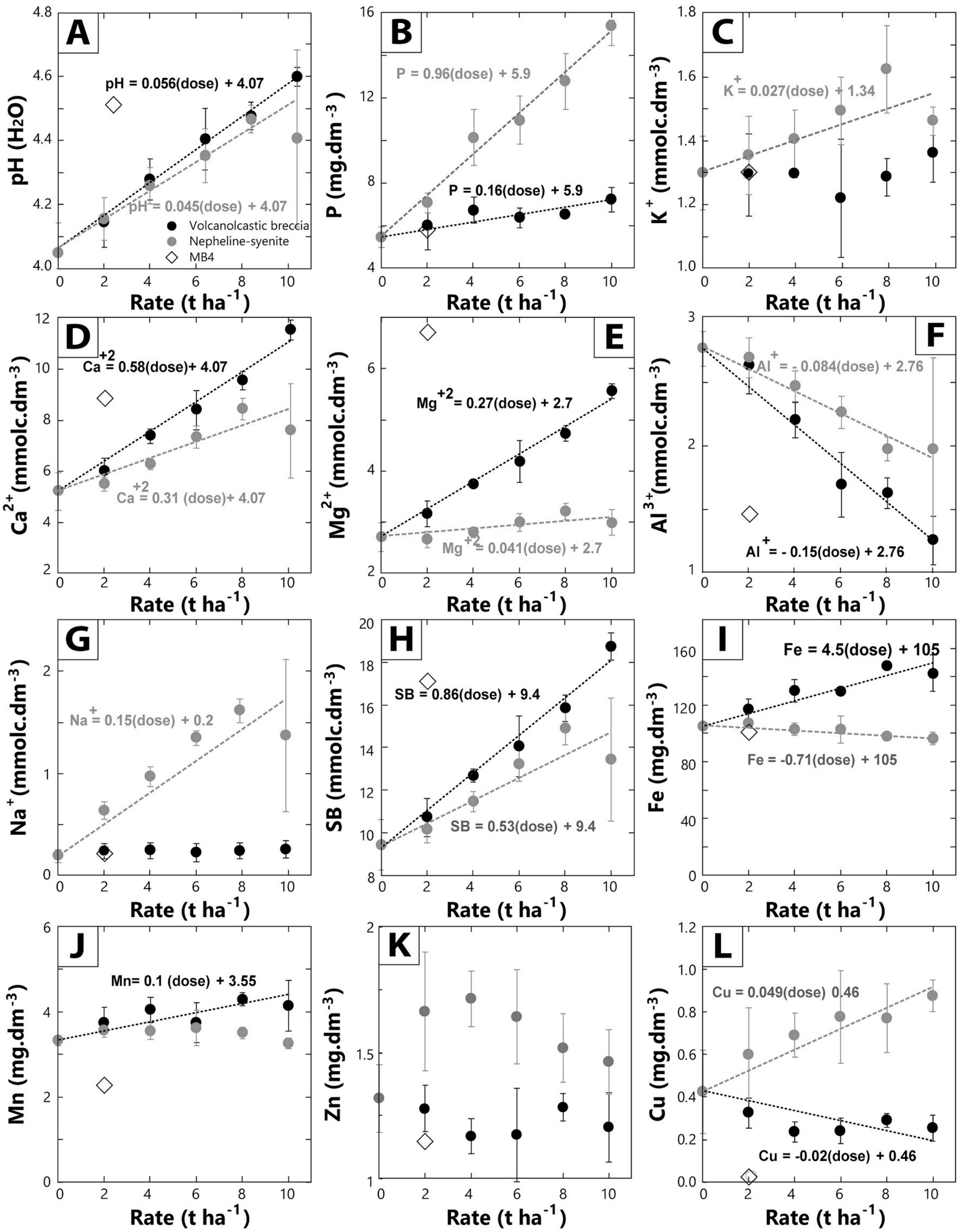

Fig. 3. Effect of the rates on treatments using the volcaniclastic breccia, the nepheline syenite, and the commercial product on pH, macronutrients, and micronutrients concentrations in the soil after the incubation experiments. (A) pH. (B) P. (C) K^{+}. (D) Ca^{2+}. (E) Mg^{2+}. (F) Al^{3+}. (G) Na^{+}. (H) Sum of bases (SB). (I) Fe^{2+}. (J) Mn^{2+}. (K) Zn^{2+} (L) Cu^{2+}. Five measurements were made for each data point and the standard deviation (1 σ) is shown as thin error bars.

the volcaniclastic breccia can be improved.

4. Conclusions

- All three studied rock types from the Fortaleza volcanic province do not contain potentially toxic elements (As, Cd, Hg, and Pb) at high levels and will not liberate harmful concentrations when applied to soils.
- The volcaniclastic breccia and the nepheline syenite are not as effective for pH correction when compared to limestone treatments.
- The volcaniclastic breccia yields promising results when compared to the MB4 product for soil remineralization of P, Fe, and Mn, while the nepheline syenite has good potential when compared to the MB4 for soil remineralization of K, P, and Cu.

- Aqueous media nutrient dissolution experiments do not always reflect results from the incubation experiments and should be carefully evaluated.
- Alkaline rocks from the Fortaleza volcanic province may act as efficient natural fertilizers and provide a local and sustainable source for this resource.

Author statement

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Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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