01 - EVALUATION OF POTENTIALLY TOXIC ELEMENTS IN MINING TAILINGS COMING FROM BENEFICIATION OF VOLCANIC ROCK IN SOUTHERN BRAZIL

http://gmga.com.br/01-evaluation-of-potentially-toxic-elements-in-mining-tailings-coming-from-beneficiation-of-volcanic-rock-in-southern-brazil/

10.31419/ISSN.2594-942X.v62019i3a1CGR

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Abstract

Mining tailings characterization for use in agriculture should consider, besides macro-and micronutrients, the potentially toxic elements presents in these materials. This work characterized and evaluated the nutrients and potentially toxic elements contents of seven samples of volcanic rock mining tailings from southern Brazilian region. The chemical composition of SiO_2, TiO_2, Al_2O_3, Fe_2O_3, MnO, MgO, CaO, Na_2O, K_2O, and P_2O_5 was analyzed by X-ray fluorescence, and potentially toxic elements As, Cd, Cr, Cu, Hg, Pb, Se and Zn by inductively coupled plasma mass spectrometry. The bioavailability of potentially toxic elements was evaluated through leaching tests in Milli-Q water. X-ray diffraction was employed to identify the mineralogical phases of the rocks. Additional mineralogical characterization was done by using field emission scanning electron microscopy equipped with a energy dispersive X-ray spectroscopy. The results point to its potential use as a fertilizer, because mining tailings contain macronutrients such as Ca, K and Mg, together with micronutrients like Cu, Fe and Zn. In addition, the potentially toxic elements presented concentrations well below the limits allowed by US legislation that are 5 (As), 1 (Cd), 5 (Cr), 5 (Pb), 0.2 (Hg) and 1(Se) mg L^{-1}; by Brazilian regulation are 15 (As), 10 (Cd), 0.1 (Hg) and 200 (Pb) ppm, and by international literature are As (15-20), Cd (1-5), Cr (50-200), Cu (60-150), Hg (0.5-5), Pb (20-300) and Zn (100-300) mg kg^{-1}, for their application in agricultural soil. This indicates that the mining tailing application would not affect the environment and human health.

Keywords: Mining tailings, Soil remineralizer, Potentially toxic elements, Environmental safety.

Introduction

The increasing awareness of society, that the major cause of the various environmental impacts is a consequence of anthropic activity, and the proof of global warming, has been implicated in the growing interest in environmental preservation and sustainable development (Ali et al. 2015). Thus, special attention has been given to the use and destination of tailings from industrial activities, including mining. Reuse and value added to industrial waste, constitute a relevant approach for the construction of sustainable alternatives in the various industrial sectors, as well as to increase initiatives to preserve the environment (Manhães and Holanda 2008). However, for volcanic rock mining tailings to be viable alternatives for use in agriculture, it is essential that they are agronomically efficient and are environmentally and food-producing safe (Doumer 2011). In this sense, it is important to know the potentially toxic elements (PTEs such as As, Cd, Cr, Cu, Hg, Pb, Se and Zn) present in the rocks, their concentration and their bioavailability, since many are directly associated with environmental problems and plant, animal and human health (Sun and Chen 2018).

In the process of rock extraction and processing considerable amounts of tailings are generated in the form of powder (Toscan et al. 2007). In the process of rock extraction and processing considerable amounts of tailings are generated in the form of powder (Toscan et al. 2007). According to Korchagin et al. (2019) in Brazil, these tailings are accumulated alongside different quarries, resulting in environmental impacts that may compromise the ecosystem, ultimately needing an environmentally appropriate final disposal. In this context, remineralization of soils is included. The remineralization of soils is the main assumption of stone meal technology, which consists in the replacement of macro-and micronutrients to the soil by the application of ground rocks (Theodoro et al. 2010). As they are available locally or regionally, their costs are lower, which will reduce application costs (Van Straaten 2006) as well as the impacts generated by the excessive consumption of such fertilizers.

Several researches have been carried out on the incorporation of tailings of ground rock to agricultural soils and/or in the recovery of degraded areas (Manning 2010; Ciceri et al. 2015; Myrvang et al. 2016). This technique has been used for a short time and its results are shown in Brazil and other countries, such as Indonesia, Sweden, Uganda, France, South Africa and also for the developed world (Van Straaten 2016). In general, these geological materials present chemical and mineralogical compositions that, when added to the soils in adequate quantities, can amplify the remineralization process (Ramos et al. 2015). In addition to the presence of nutrients, it is also discussed the presence of heavy metal concentrations in the composition of the volcanic rocks. According to Oliveira et al. (2015), the harmful effect of these chemical elements depends, basically, on the amount present in the material and the chemical forms in which they appear in the soil. The PTEs occurrence in soluble, exchangeable, occluded, precipitated or complex forms are the main factors which defines the polluting potential.

In this context, Brazilian law nº 12,890/2013 included remineralizers as an input category for agriculture for a way of making domestic and external market requirements compatible with aspects related to the safety of inputs, in order to reduce the risks of contamination of manufactured foods, regulating the production and use of fertilizers in order to preserve the environment (Brazil 2013).

Given this and considering the great amounts of tailings produced in studied region, the present work is focused on the evaluation of the potential use in agriculture of generated tailings at seven volcanic rock mines in the southern Brazilian.

Materials and methods

The research was conducted by using samples of tailings from mining of volcanic rocks (VRMT) were collected directly from the tailing piles of seven mining companies operating in the southern Brazilian region (Figure 1).

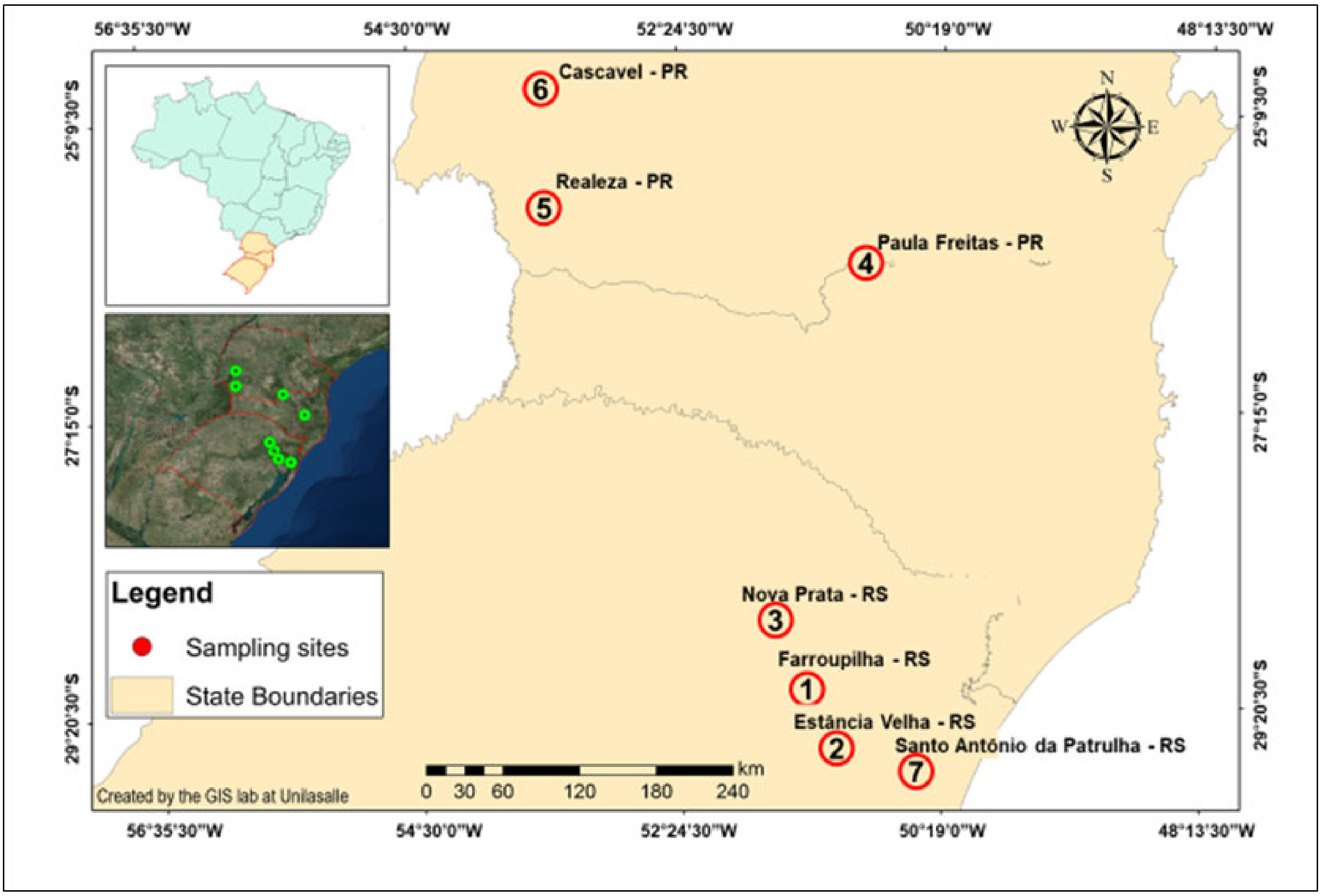

Figure 1. Sample collection points.

The study region is recognized for the large volume generated (52,400 m^3 annually) of mining tailings (Toscan et al. 2007). According to Nardy et al. (2014) in the Serra Geral Group the dominant volcanic rocks are basalt, basaltic andesite and andesite, followed by riodacite and little rhyolite, composed of 30 to 50% of plagioclase, 20 to 35% of augite and pigeonite and 5 to 15% of opaque minerals.

The sampling procedure was described by Ramos et al. (2015) and the samples were identified with numbers from 1 to 7. Chemical composition in percentage weight of SiO_2, TiO_2, Al_2O_3, Fe_2O_3, MnO, MgO, CaO, Na_2O, K_2O and P_2O_5 was determined by X-ray fluorescence (XRF) analysis on a Philips model PW1480 spectrometer after fusion with lithium tetraborate. Potentially toxic elements were determined by inductively coupled plasma mass spectrometry (ICP-MS). To do this, VRMT samples were digested in HNO_3 hot of followed by HF:HNO_3: $HClO_4$ according to Querol et al. (1997), the resulting solution was then analysed.

For the leaching tests, the solid waste SW-846 method developed by United State Environmental Protection Agency (US EPA 1997) has been used. For determining the PTEs bioavailable (As, Cd, Cr, Cu, Hg, Pb, Se and Zn) in VRMT, 50.0 g of each mining tailings sample was placed in flaks of 1 L containing 800 mL of Milli-Q water, (liquid to solid (L/S) ratio of 16:1). The solutions were placed on a mechanical shaker and agitated for 24 h. During the experiment, the pH was adjusted and maintained in

5±0.2 by addition of 0.5 mol L^{-1} acetic acid. After 24 h, the liquid volume was adjusted to 1000 mL by the addition of Milli-Q water. The solutions were filtered, and aliquots of 20 mL were separated into polypropylene flaks and analysed ICP-MS in duplicate.

The mineralogical composition of the VRMT were evaluated by X-ray diffraction (XRD). The procedure and analytical conditions were described by Ramos et al. (2015). Polished blades of VRMT samples were made and the microstructural characterization was performed using a 15-keV EVO MA10 field emission scanning electron microscopy (FE-SEM) equipped with a Zeiss energy dispersive X-ray detector (EDS).

Results and discussions

The main peaks in most samples correspond to anorthite. Moreover, it was also possible to detect quartz and amorphous materials in all samples. The analysis of X-ray diffraction of the samples indicate that the mineralogical compositions consist mainly of minerals rich in Si, Al, Ca, Fe, Na, Ca, and amorphous material (Table 1). These results confirm the presence of the main constituents of volcanic rocks of the study area, which are rich in chemical compounds called silicates - composed of metals combined with silicon and oxygen (Gill 2010). Among them we can mention the anorthite that is a mineral of the plagioclase class with a high proportion of calcium in its structure. Because it is a calcic plagioclase its weathering resistance is low (Gill 2010) and, therefore, can release their nutrients more easily to the environment. The augite that is an iron-magnesium mineral of group of pyroxenes, made up of elements such as Si, Mg, Fe and Ca is a very unstable mineral against weathering and because of this, can release nutrients and form new minerals (Deer et al. 2013). Also, it contains a certain amount of sanidine ($KAlSi_2O_8$), which is an alkali feldspar rich in K (Deer et al. 2013), easily altered under exogenous conditions, has a high potential for K release (Gill 2010). The presence of these minerals makes the VRMT very attractive as a soil remineralizers.

Table 1. Semiquantitative mineralogical composition (Rietveld calculation) by X-ray diffraction of the VRMT samples.

Minerals	Samples Identification						
	1	2	3	4	5	6	7
Cristobalite	11	ND*	ND*	ND*	ND*	ND*	ND*
Anorthite	46	48	37	46	49	43	48
Sanidine	19	17	21	ND*	ND*	ND*	18
Quartz	7	7	12.1	5	11.9	3	5
Mordenite	1	ND*	ND*	ND*	ND*	ND*	ND*
Montmorillonite	ND*	2	<1	<1	1.7	<1	3
Augite	ND*	ND*	ND*	10	31	15	ND*
Amorphous materials	16	26	28.9	38	6.4	38	25

*Not detected.

The semi-quantitative chemical analyses confirm that the tailings of volcanic rocks mining are mainly composed by aluminosilicates of Ca, Na, K, Fe and Mg and some Tip-bearing minerals. The results show a high potential for application as a fertilizer (Figure 2).

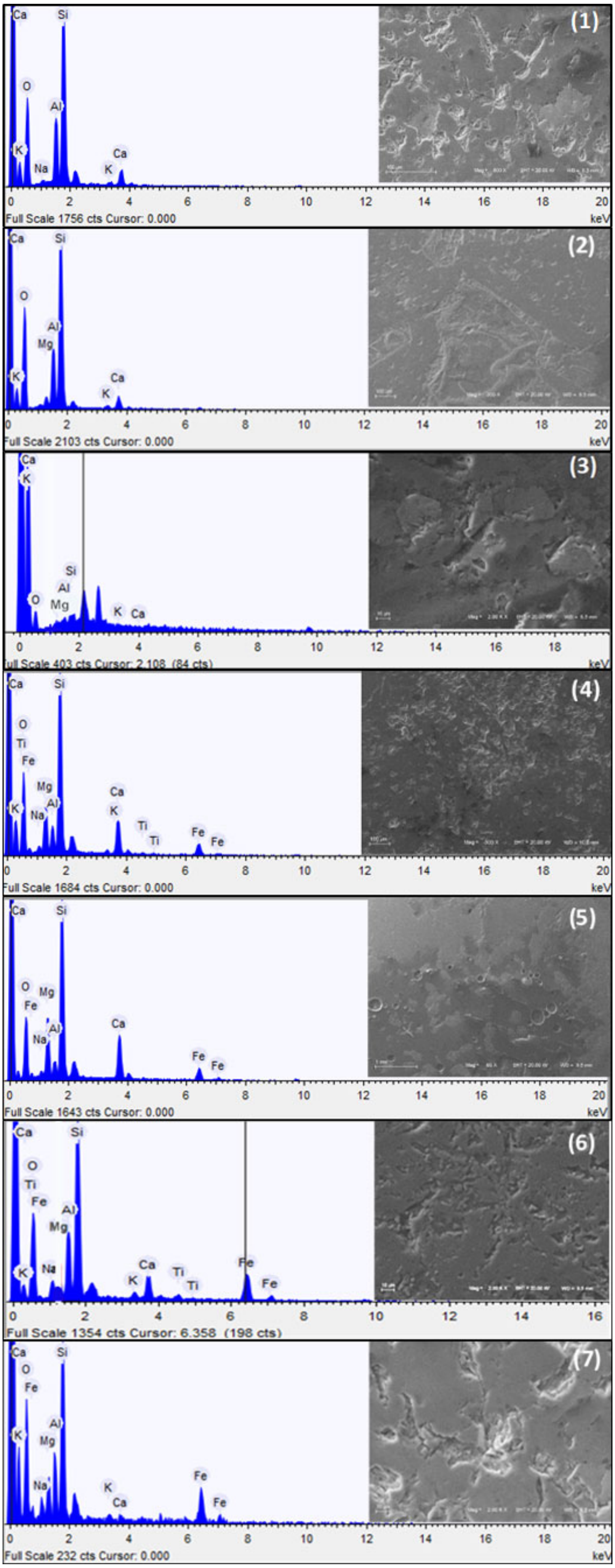

Figure 2. Field emission scanning electron

microscopy equipped with an energy dispersive X-ray spectroscopy of VRMT samples.

The analysis by FE-SEM/EDS proved to be not satisfactory for identification of PTEs in VRTM samples, which are possibly in concentration below the technique capacity.

The Normative Instructions nº 05 and nº 06 of Ministry of Agriculture, Livestock and Supply (MAPA) of Brazil, published on March 10, 2016, establish two sets of criteria for remineralizers. In this study, the first set of criteria will be considered, since it refers to the minimum specifications and guarantees that must be proven by geochemical and mineralogical analyses, such as sum of bases ($CaO + K_2O + MgO$) and maximum concentrations of potentially toxic elements (AS, Cd, Hg and Pb) for use as remineralizers for agriculture. The maximum levels are 15, 10, 0.1 and 200 ppm for As, Cd, Hg and Pb, respectively. The second set of criteria is related to agronomic efficiency, which should be verified in the case of Brazil by public research institutions and other entities accredited by MAPA (Brasil 2016a, b).

The high aluminum and silicon concentrations were expected because the rock samples are mainly composed of aluminosilicate minerals. High concentrations of Al^{3+} in soil are toxic to plants (Kabata-Pendias 2011), being considered the major limiting factor of agricultural productivity (Fageria et al. 2011). However, during weathering of aluminosilicate minerals the series of Al hydroxides of variable charge and composition, from $Al(OH)^{2+}$ to $Al(OH)_6^{3+}$, are formed and they become structural components of clay minerals. In general, the solubility of Al hydroxides is low, especially in the pH range of 5–8 (ideal for most crops), and solubility decreases over time (Kabata-Pendias 2011). Thus, toxic aluminum will hardly be made available to the soil.

As mentioned before, silicon, especially combinate with oxygen, is the basic nonmetallic component of all rocks (Kabata-Pendias 2011). Studies of Sahebi et al. (2015) confirmed that Si is an important element that increases the tolerance of plants against environmental stress. What makes Si necessary for plant growth, development, and structure strength.

It is important to note in Table 2 that all samples of this study meet the minimum guarantees in relation to the sum of bases (CaO, K_2O and MgO), which must be equal or higher than 9%, with a tolerance of up to 10% less and in relation to potassium oxide content (K_2O), which must be equal to or greater than 1%, established by IN nº 05/2016 (Brasil 2016a). This fact confirms the presence of minerals that make up the samples detected by mineralogical analyses (Table 1). Silva et al. (2012) studied six silicate rocks from the states of Goiás, Santa Catarina, Minas Gerais and Bahia (the central, southern, southeast and northeaster regions) of Brazil, that presented a sum of bases and K_2O levels above that established by Brazilian regulation, as well as the results obtained in the present study. From these results, the remineralization condition can be attributed to the volcanic rock mining tailings studied here.

Table 2. Concentration of the major and minor elements of VRMT samples (%) expressed by respective oxides.

Oxides	Samples

	1	2	3	4	5	6	7
SiO_2	62.4	55.3	59.5	55.2	55.4	54.8	57.5
TiO_2	1.02	1.18	1.15	1.22	1.45	1.44	1.05
Al_2O_3	14.1	13.8	13.7	13.5	13.1	13.2	15.1
Fe_2O_3	7.09	10.7	9.17	10.1	10.8	10.6	8.30
MnO	0.12	0.19	0.16	0.18	0.19	0.23	0.20
MgO	2.62	3.23	2.76	3.30	3.54	3.92	3.82
CaO	4.36	8.06	4.37	9.68	9.76	8.50	7.39
Na_2O	2.95	3.83	3.62	3.63	2.10	3.77	1.93
K_2O	3.36	1.67	3.25	1.18	1.39	1.55	1.65
P_2O_5	0.08	0.28	0.31	0.26	0.34	0.25	0.63

As the Brazilian regulation is restricted to PTEs As, Cd, Hg and Pb, were also used the Resolution 420 guideline values from Brazilian National Environmental Council (Conama, 2009) regarding chemical substances levels in order to protect the soil quality. The maximum levels allowed by Conama (2009) for As, Cd, Cr, Cu, Hg, Pb and Zn are 35, 3, 150, 200, 12, 180 and 300 mg kg^{-1}, respectively. Furthermore, the values reported commonly in the international literature of maximum allowable concentrations (MAC) for PTEs in agricultural soils, compiled by Kabata-Pendias (2011) from Kabata-Pendias and Sadurski (2004) and Chen (1999). According to Kabata-Pendias (2011) the ranges of MAC for PTEs in agricultural soils (mg kg^{-1}) are As (15-20), Cd (1-5), Cr (50-200), Cu (60-150), Hg (0.5-5), Pb (20-300) and Zn (100-300).

Table 3 show that the PTEs concentrations of all the studied samples presented levels well below the limits established in all regulations investigated here. Therefore, the rocks do not present risks of contamination of soils by PTEs under the conditions considered in this study. Similar results were previously reported by Ramos et al. (2015) and Ramos et al. (2017) which also found low concentrations of PTEs in the rock powder of the same region of this study. Bergmann et al. (2013) carried out analysis of PTEs such as As, Cd, Cr, Hg and Pb in 666 samples of volcanic rocks collected along the Serra Geral Group. These authors concluded that 629 these rocks presented characteristics suitable for agricultural use in the form of remineralizers. However, 33 samples were considered improper because of the Cr level, 3 because the As level, and 1 because the Pb level, all of them above the permissible limits in Brazilian regulation (Brasil, 2016a).

Table 3. Concentrations (mg kg^{-1}) of PTEs in the VRMT samples.

PTEs	Samples						
	1	2	3	4	5	6	7
As	0.07	0.06	0.07	0.06	0.02	0.06	0.11
Cd	<0.002	<0.002	<0.002	<0.002	<0.002	<0.001	<0.001
Cr	87.0	68.00	70.0	68.3	65.6	51.1	109.6
Cu	39.9	72.9	41.7	55.3	36.9	41.05	31.1

Hg	<0.02	<0.02	<0.01	<0.02	<0.01	<0.02	<0.02
Pb	0.03	0.01	0.02	<0.01	<0.01	<0.01	0.01
Se	<0.2	<0.2	<0.2	<0.2	<0.2	2.07	0.43
Zn	99.1	139.1	109.4	132.4	145.2	157.89	98.9

The data in Table 4 show that the PTEs concentration were below the limits established by US legislation (U.S. Government Publishing Office, 2011). In accordance with such legislation, a solid waste exhibits toxicity if the solution contains PTEs concentrations equal to or greater than the standard value: 5, 1, 5, 5, 0.2 and 1 mg l^{-1} for As, Cd, Cr, Pb, Hg, and Se respectively. Similar results were obtained by Inui et al. (2012) who studied five rock samples (one volcanic rock) from Japan to assess their leaching of lead and arsenic. These authors verified that As and Pb leaching were negligible for andesite (volcanic rock) sample. Manhães and Holanda (2008) also found concentrations of As (<0.01), Cd (<0.001) e Pb (0.09) mg l^{-1} in silicate rock, well below the limits allowed by US legislation.

Table 4. PTEs concentration in (mg L^{-1}) leachates of VRMT samples.

PTEs	Samples						
	1	2	3	4	5	6	7
As	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
Cd	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002
Cr	<0.002	<0.002	<0.002	<0.002	<0.002	0.04	0.05
Hg	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001
Pb	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002
Se	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002	<0.002
Zn	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001	< 0.001

Although the results of this study are limited to a few samples, it should be an indicator for more comprehensive studies and surveys that consider similar rocks belonging to Serra Geral Group.

Conclusions

The use of rock powder in agriculture is based on the assumption that maintaining soil fertility is part of a strategy that involves the conservation of natural resources, the supply of nutrients in adequate quantities and the attainment of sovereignty, with a reduction in dependence on external inputs. In spite of this, it is necessary to have an adequate control of its quality, especially regarding the concentration of K, Ca and Mg oxides, as well as the presence of PTEs. It is essential that constant monitoring of macro-and micronutrient contents be carried out on rock samples used for this purpose as a way of ensuring the quality of the products added to the soil, which will result in better quality foods and, in the end, the quality increase nutritional and environmental protection.

The purpose was achieved by using the FE-SEM, XRD and XRF for determining the mineralogical and chemical composition of tailings of volcanic rock mining. Was evidenced these techniques was shown to be very useful in identifying the minerals present in the studied VRMT enabling the comparison of the chemical composition and certifying those results.

According to the results obtained in this study, it can be concluded that volcanic rock mining tailings samples contain important macro and micronutrients, such as Ca, K, Mg, Cu, Fe and Zn, which indicate a good potential to be used as fertilizer. In addition, the chemical analysis revealed that the PTEs were below the limits established in the studied regulations. The safe use of this waste requires an evaluation of the composition of the PTEs, to guarantee the quality of the inputs added to the soil, which will result in better nutritional quality of the food and environmental protection.

Several factors restrict the use of rock powder in agricultural soils and are the main challenges of the technique. We can highlight the complex composition of the rocks; location of the deposits of these rocks in relation to the place of application; the supposed low-cost grinding methods; and the behavior of these rocks in interaction with the environment in which they will be applied. The use of volcanic rock mining tailings in agriculture may be appropriate to address the problem of mine off-site by-products and to reduce the consumption of highly soluble fertilizers. Another possible use to these materials would be the physical-chemical wastewater treatment for particulate material removal.

Acknowledgments

The authors would like to thank to James Hower for editing and to CNPq (Grant Nº. 445435/2014-3) for the financial support.

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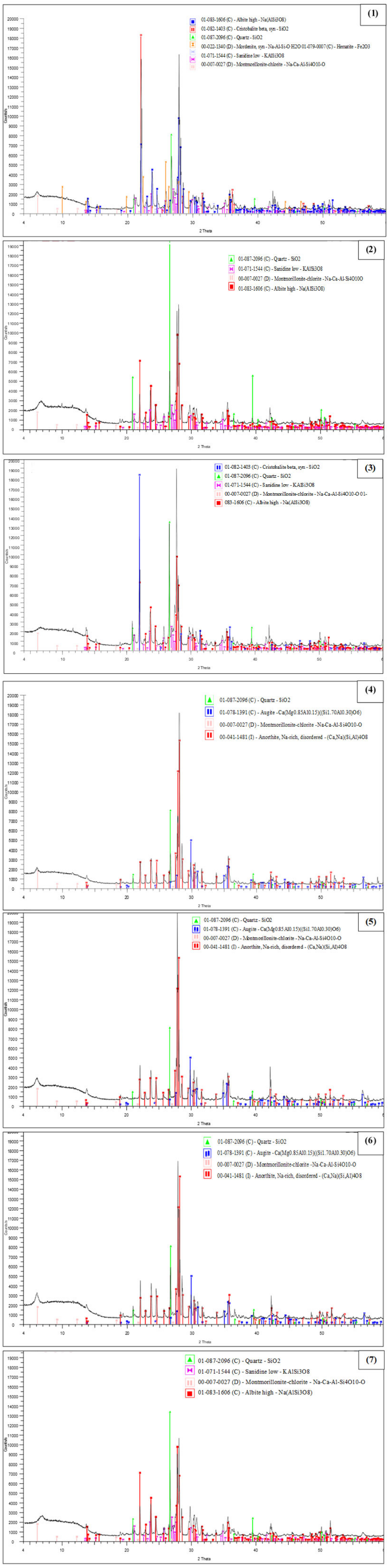
Supplementary Material.



X-ray diffraction spectrums of VRMT samples.

10.31419/ISSN.2594-942X.v62019i3a1CGR