Characterization of verdete rock as a potential source of potassium[1]

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ABSTRACT

Potassium is a nutrient found at low levels in Brazilian soils, requiring large inputs of fertilizers to achieve satisfactory crop yields. Brazil has high external dependence and limited reserves of soluble K mineral, which is traditionally exploited for the production of fertilizers. On the other hand, it is common the occurrence in the country of potassium-rich silicate minerals which are not commercially exploited. This study aimed to characterize mineralogically and chemically samples of verdete rock separated into size fractions and evaluate its potential as potassium fertilizer. The mineral composition of verdete rock is based on glauconite, quartz and feldspar. The total K_2O content in verdete rock ranged from 5.18 to 9.0 dag/kg. The K content extracted in water or 2% citric acid was 2.4% below the total of K, indicating low reactivity of verdete rock and limitations for direct use as K source. The processes of physical fractionation and sedimentation in water are inefficient to promote the concentration of K in the different verdete rock fractions. The total K content in some samples are considerable and may enable the use of this rock as raw material for production of more reactive potassium fertilizers.

Key words: glauconite, K, fertilizer.

RESUMO

Caracterização do verdete como fonte potencial de potássio

O K é um nutriente encontrado em baixos teores em solos brasileiros, sendo necessário grande aporte via fertilização para alcançar produtividades satisfatórias das culturas. O Brasil apresenta elevada dependência externa e possui limitadas reservas de minerais solúveis de K, tradicionalmente explorados para a produção de fertilizantes. Por outro lado, é comum a ocorrência no país de minerais silicatados ricos em K não explorados comercialmente. Neste trabalho, objetivou-se caracterizar mineralógica e quimicamente amostras de verdete, separadas em frações granulométricas e avaliar seu potencial de uso com fertilizante potássico.A composição mineral do verdete é à base de glauconita, quartzo e feldspatos. Os teores totais de K_2O no verdete variaram de 5,18 a 9,0 dag/kg. O teor de K extraído em água ou em ácido cítrico a 2% foi inferior a 2,4% do K total, indicando baixa reatividade do verdete e limitações para seu uso direto como fonte de K. O fracionamento físico e a sedimentação em água são processos ineficientes para promover a concentração de K nas diferentes frações do verdete. Os teores totais de K em algumas amostras de verdete são consideráveis, podendo viabilizar o uso desta rocha como matéria prima para produção de fertilizantes potássicos mais reativos.

Palavras- chave: glauconita, K, fertilizante.

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INTRODUCTION

The limited supply of soluble K minerals in the world and the growing consumption of this nutrient have been constant concerns for importing countries of potash fertilizers and agricultural importance such as Brazil, USA, China and India. The exploitation of evaporite deposits, especially salts of K (sylvite), K and Na (sylvite) or K and Mg (carnallite), is the main source of potassium fertilizers, as salts derived from these deposits are water soluble and can be mined and processed more easily. These deposits occur primarily in Canada, Russia, Belarus and Germany, which together account for about 85% of world exports of K (Fertecon, 2013; Oliveira, 2012).

Potash fertilizer import was higher than 90% of Brazil's domestic consumption in 2011 (Oliveira, 2012), and the country was the world's third largest consumer of these products (IFA, 2013). Low domestic production of these fertilizers is mainly due to the limited occurrence of reserves of soluble K salts, besides the high cost, operational difficulties and environmental risks of exploitation. This is the case of carnallite and sylvite in the states of Sergipe and Amazonas, respectively (Oliveira & Sousa, 2001). These facts hinder the K commercial production in the country, which is unique in South America, with only one mine in Sergipe exploiting sylvite.

The offer and the price of potash fertilizers, as occurred in 2008, could jeopardize the expansion of Brazilian agriculture more than any other nutrient. This has stimulated the search for unconventional sources of K such as potassium silicate. In India, the nonoccurrence of commercially exploitable soluble K sources have led to exploration and research into rocks consisting mainly of glauconite (Rawlley, 1994; Rao *et al*., 1993), with reserves estimated at 940 Mt (million metric ton) (Mazumder *et al*., 1993). In New Zealand, the occurrence of glauconite in underwater platform, with reserves estimated at 2 Gt (billion metric ton), has raised the interest in the exploitation for the production of potassium fertilizer (Lawless, 2012).

In Brazil, alternative sources of K have been studied to be used as potassium fertilizers or as raw materials for their production, including nepheline syenite and feldspar (Faquin, 1982; Siqueira & Guedes, 1986; Nascimento, 2004; Orioli Junior & Coutinho, 2009), granite (Piza *et al*., 2010) and verdete rock (Lopes *et al*., 1972; Eichler, 1983; Santos, 1984; Leite, 1985; Piza *et al*., 2010, 2011).

Verdete rock, a sedimentary rock, stands out among the potential K sources, with variable composition and K_2O content ranging from 5 to 15 dag/kg (Loureiro *et al*., 2010). It occurs in Serra da Saudade, Alto Paranaíba Region (MG), geologically located in the San Francisco craton (Valarelli *et al*., 1993). According to Eichler (1983), assessments made by METAMIG indicated, only in the municipality of Cedro do Abaeté, MG, a total of 57 Mt of verdete rock, with an average content of 11.4 dag/kg K_2O.

Glauconite is the main potassium mineral present in verdete rock (Piza *et al*., 2010). According to Fassbender (1975), it is a mica of the illite group, which is characterized by having greater isomorphous substitution of Al^{3+} by Fe^{2+} in octahedral structures. This mineral is formed by a process called glauconitization, which occurs in a marine environment of slow sedimentation under reducing conditions. During this process, in low dentritic input conditions, there is loss of alumina and silica along with Fe and K enrichment (Pettijohn, 1963; Fassbender, 1975; Lima *et al*., 2007).

In the Bambui formation, the site for verdete rock in Brazil, glauconite gives the green color to this rock, usually with a particle size less than 3 μm. Stratigraphically, glauconite occurs in areas below the sedimentary input, and its genesis occurred slow halmirolysis within a reducing microenvironment (Guimarães, 1997). In the glauconitization process, the starting material resembles an iron aluminosilicate subsaturated with alkali, similar to smectites (Lima *et al*., 2007).

The exploitation and regional use of less reactive potassium minerals found in the country can benefit the agricultural sector with an essential nutrient input and promote the development of local mining industries. In this sense, the characterization of rocks containing K and knowledge of the variability of the nutrient in these materials are critical to planning processes and fertilizer production routes. Thus, this study aimed to characterize mineralogically and chemically samples of verdete rock and evaluate a physical method for concentrating K.

MATERIAL AND METHODS

This study was conducted in laboratory conditions, at the Department of Soil of the Federal University of Viçosa, using verdete rock as a potassium source.

Sample collection and preparation

Fourteen (14) samples of verdete rock were collected in the Central Region of Minas Gerais (MG), in the municipalities of Cedro do Abaeté and Quartel Geral. Sample collection was performed randomly in the landscape, from outcrops of this rock. Picks were used to break the rock, and approximately 10 kg of verdete rock was collected from different sites, which were georeferenced. The location of the collection points and their coordinates are shown in Table 1 and Figure 1, respectively.

Chemical Characterization

The chemical characterization of verdete rock samples was carried out with 0.300 g of rock with particle size smaller than 0.074 mm. The samples were transferred to microwave tubes and added of 4.0 mL of HCl, 9.0 mL of HNO_3 and 4.0 mL of HF. Next, 2.0 ml of saturated solution of H_3BO_3 (100 g/L) was added. Samples were taken to the microwave, which carried out the digestion by the EPA method 3052 (1996). The extract was filtered through quantitative filter paper by rapid filtration. Fe, Al, Ca, Mg, Na, P, Ti, Mn, Cr, Ba, Sr, Zn, Cu, Ni and Pb were determined using optical emission spectrometry with inductively coupled plasma (ICP OES) (8300-PerkinElmer) and Si by X-ray fluorescence (Medx1300-Shimadzu). The K content was determined by flame emission spectrophotometry (B462-Micronal).

K soluble in water was determined in 1.0 g sample with particle size <150 µm, which was transferred to a 125-ml Erlenmeyer flask and added of 50 mL of distilled water. The solution was boiled for 10 min in a heater plate at 180 °C. After cooling, the extract was filtered in slow quantitative filter paper (> 28 µm). The flasks were weighed before and after the boiling in order to correct the volume.

K-soluble in 2% citric acid was determined in 0.50 g of sample with particle size <150 µm, which was transferred to a 125-ml Erlenmeyer flask and added of 50 mL of the solution of 2% monohydrate citric acid. The solution was stirred in a circular horizontal shaking table at 160 rpm for 30 min. The extract was filtered in slow quantitative filter paper (> 28 µm). The K content of the aqueous extract and citric acid was determined by flame emission spectrophotometry.

Table 1. Coordinate locations of the collection sites of the verdete rock samples, in the municipalities of Quartel Geral and Cedro do Abaeté, in Minas Gerais, Brazil

Collection site	Coordinate UTM (Fuse UTM 23 S)	
	X	Y
1	420763.1	7871570.4
2	420763.1	7871570.4
3	419654.6	7871575.9
4	421120.1	7871937.6
5	421136.3	7871929.0
6	421142.7	7871878.0
7	421843.8	7872863.6
8	421873.2	7872788.0
9	422032.9	7872736.5
10	423725.6	7874215.9
11	423738.8	7874334.6
12	426090.9	7882653.2
13	425888.3	7882636.0
14	426728.6	7881766.5

Fractionation and sedimentation in water

The relationship between particle size and the total concentration of K in the rock was evaluated in verdete rock samples chemically characterized with K content above 8.4 dag/kg of K_2O. A sample of 250 g of rock was milled and passed through a 2.0 mm sieve and separated into three size classes (0.2-2.0; 0.15-0.2 and < 0,15 mm). Sedimented and suspended samples were also obtained in the < 0.15 mm fraction, in an aqueous medium. Twenty-five grams (25 g) of this fraction were transferred to 1.0-L beakers and stirred at intervals of 120 min for a total of five cycles, when the suspended fraction was collected using a siphon. Finally, the material was dried in a forced air oven at 105 °C. The trial was conducted in a completely randomized design with three replications.

A rock sample was ground to the size < 0.15 mm and used as a reference for the total concentration of K.

Mineralogical characterization

The mineralogical analysis was carried out using X-ray diffractometry (XRD). The samples were ground to a particle size < 0.15 mm and placed in excavated blade. A PHNalytical diffractometer model X' PertPRO, using CoKα radiation (1.7889 nm) with sample scanning in the range of 4 to 80 degrees 2θ, with intervals from 0.02 degrees 2θ to 1 step.s^{-1}; with 40 kV voltage and 30 mA current.

RESULTS

Verdete rock occurs in the municipalities of Cedro do Abaeté and Quartel Geral between 880 and 940 m altitude. The samples showed intense color variation and hardness, indicating the different degrees of weathering. The K contents varied randomly in the landscape and were higher in the hardest rocks of more intense green, tending to blue (Figure 2).

The total K content in the rock varied between 5.18 and 9.0 dag/kg, and the average solubility in water and 2% citric acid were 0.61 and 1.54% of the total K, respectively (Table 2).

Table 3 shows the total content of some elements found in verdete rock. In addition to K, the average contents of 62.64 dag/kg of SiO_2, 5.81 dag/kg of Fe_2O_3 and 14.43 dag/kg of Al_2O_3 also stand out. The rock is poor in P, Ca, Mg and S and has low levels of potentially toxic elements (Brasil, 2006). The contents of micronutrients Zn, Cu and Mn in the rock are very low or zero.

The mineralogical characterization using diffraction with peaks at 1.0, 0.5, 0.453, 0.363, 0.333, 2.396 and 1.511 nm indicates the occurrence of glauconite in all samples. The presence of quartz was diagnosed by peaks at 0.425, 0.335, 0.228 and 0.214 nm. Feldspars were recorded by the peaks at 0.574, 0.426, 0.404, 0.379, 0.348, 0.335, 0.324 and 0.30 nm (Figure 3).

Physical fractionation of verdete rock, in different grain sizes, caused no significant changes in total K contents in relation to the content obtained from the reference sample (Figure 4).

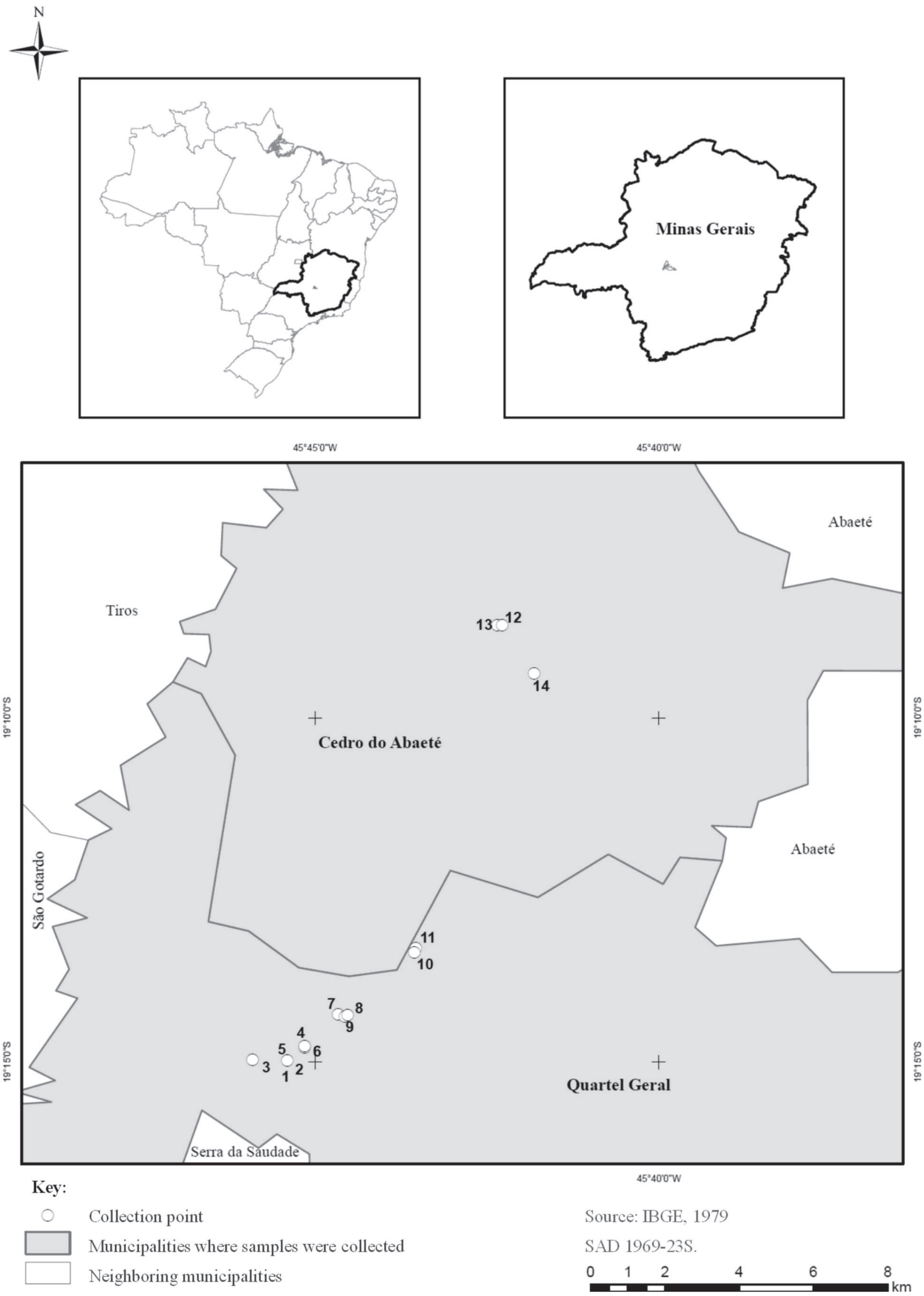

Figure 1. Location map of the collection sites of the verdete rock samples in the municipalities of Quartel Geral and Cedro do Abaeté, in Minas Gerais, Brazil.

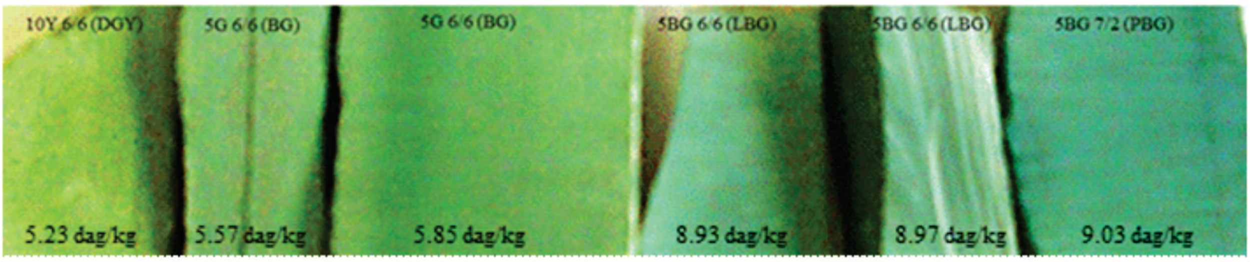

Figure 2. Photograph showing the variation in K_2O content (values at the bottom) in verdete rock samples and changes in the rock color. Color classification according to Munsell Color (2009) (top). DGY (Dark Greenish Yellow), BG (Brilliant Green), LBG (Light Blue Green) and PBG (Pale Blue Green).

Table 2. K total contents and soluble in water and 2% citric acid in samples of verdete rock

Samples	Total K_2O content	Solubility of verdete rock for K	
		water	2% citric acid
	dag/kg	%	
1	5.23	0.26	1.22
2	5.86	0.64	1.75
3	8.43	0.56	1.96
4	6.33	0.63	1.44
5	5.57	1.37	1.61
6	5.42	0.93	1.59
7	8.11	1.00	2.29
8	8.08	0.50	1.12
9	8.93	0.25	1.00
10	8.29	0.12	0.81
11	7.52	0.15	0.67
12	8.67	0.92	2.33
13	9.04	0.34	1.13
14	9.00	0.85	1.49
Lower	5.22	0.12	0.67
Medium	7.46	0.61	1.54
Higher	9.00	1.37	2.33
D-standard	1.45	0.37	0.58

DISCUSSION

The variation in K contents of the verdete rock in relation to the green intensity may be due to glauconite weathering. This micaceous mineral rich in K (Maraschin & Mizusaki, 2008), when weathered, undergoes initial changes which result in reduced load and loss of structural K (Curi *et al*., 2005), decreasing the K content in rock with the advance of weathering processes.

Variations in the green color of verdete rock may be related to the change in the relationship between Fe^{2+} and Fe^{3+}, which participate in the rock formation. This element is in the octahedral layer of glauconite, replacing Al^{3+} isomorphically. The weathering and oxidation of Fe^{2+} cause the ratio Fe^{2+}/Fe^{3+} to decrease, which could be an influential factor in defining the verdete rock color, which is independent of the total Fe content (Chiodi Filho *et al*., 2003), as confirmed by the results . The small changes in the levels of Fe and independence with the K content and the rock color confirm the statement by Chiodi Filho *et al*. (2003). Certainly the formation of minerals such as iron oxides during weathering of verdete rock immobilize the element in the rock. Data reported by Piza *et al*. (2011) confirm the presence of iron oxides in verdete rock samples. The same assumptions apply to Al, which must remain in the system during the weathering of verdete minerals due to the formation of Al oxy-hydroxides.

According to Lima (2007), the green color of the verdete rock may vary with the particle size of the rock, and as particle size decreases the green color becomes more intense, indicating that the chromophore mineral is concentrated in the clay fractions.

The variation in total contents of K in verdete rock has also been observed in other studies. Eichler (1983)

Table 3. Concentration of some elements in samples of verdete rock

Sample	Element concentration																	
	K_2O	SiO_2	Fe_2O_3	Al_2O_3	CaO	MgO	Na_2O	P_2O_5	TiO_2	MnO	Cr_2O_3	BaO	SrO	S	Zn	Cu	Ni	Pb
	% w/w													mg/kg				
1	5.23	61.13	6.70	16.25	0.62	1.13	0.21	0.00	0.74	0.00	0.01	0.07	0.01	400	90	91	10	42
2	5.86	63.68	6.00	14.40	0.03	1.10	0.16	0.00	0.35	0.00	0.01	0.00	0.00	200	95	85	2	54
3	8.43	62.56	5.46	14.42	0.35	1.08	0.18	0.00	0.57	0.01	0.01	0.02	0.00	400	94	88	3	47
4	6.33	58.37	6.57	12.02	0.80	1.27	0.21	0.00	0.61	0.01	0.02	0.01	0.01	400	131	116	12	43
5	5.57	65.61	5.63	14.96	0.00	1.20	0.14	0.00	0.70	0.00	0.02	0.03	0.00	200	87	190	7	29
6	5.42	59.09	5.83	14.25	0.00	1.10	0.12	0.00	0.62	0.00	0.01	0.04	0.00	200	89	88	2	50
7	8.11	62.42	5.67	14.62	0.38	1.20	0.22	0.05	0.57	0.00	0.02	0.05	0.00	300	117	92	7	35
8	8.08	60.83	5.77	15.09	0.38	1.10	0.25	0.05	0.62	0.00	0.01	0.01	0.00	300	100	88	3	43
9	8.93	65.63	5.74	15.06	0.01	1.22	0.20	0.07	0.63	0.00	0.01	0.01	0.01	200	100	94	7	39
10	8.29	64.34	4.84	13.43	0.00	1.12	0.11	0.07	0.64	0.00	0.01	0.01	0.01	200	92	75	3	47
11	7.52	60.91	6.20	15.30	0.76	1.22	0.23	0.07	0.64	0.01	0.01	0.00	0.02	300	123	100	11	48
12	8.67	60.83	5.06	14.49	0.43	1.12	0.24	0.05	0.53	0.00	0.01	0.01	0.00	200	106	82	3	41
13	9.04	65.25	6.13	14.02	0.04	1.25	0.15	0.02	0.60	0.01	0.01	0.00	0.00	200	133	102	14	39
14	9.00	66.32	5.49	13.74	0.14	1.25	0.19	0.05	0.46	0.00	0.01	0.04	0.00	200	111	83	5	42
Lower	5.23	58.37	4.84	3.43	0.00	1.08	0.11	0.00	0.35	0.00	0.01	0.01	0.00	200	87	75	2	29
Medium	7.46	62.64	5.81	14.43	0.28	1.17	0.18	0.03	0.57	0.00	0.01	0.02	0.00	264	105	98	6	43
Higher	9.04	66.32	6.70	16.25	0.80	1.27	0.25	0.07	0.74	0.01	0.02	0.05	0.02	400	133	190	14	54
D_standard	1.50	2.60	0.50	1.00	0.30	0.10	0.00	0.00	0.10	0.00	0.00	0.00	0.00	84	16	28	4	6

reported mean content of the element in rock of 11.4 da/kg, whereas Piza *et al*. (2010) found K_2O ranging between 6.09 and 7.33 dag/kg.

Verdete rock has low content of K soluble in water or 2% citric acid, indicating a certain limitation to the use as potassium fertilizer in its natural state, especially for short cycle crops. However, the total K content, of up to 9.0 dag/kg K_2O, indicate the potential use of the rock as raw material for the production of potash fertilizer.

Low concentration of other macronutrients of agronomic interest in verdete rock such as P, Ca, S, Mg and the micronutrients, Zn, Cu and Mn limit the use of this rock for exploitation of these elements or as fertilizer, since abundant and more concentrated sources are found in the country (Betekhine, 1968; Sampaio *et al*., 2005; CETEM, 2009).

The mineralogical characteristics of the verdete rock samples show similar composition of those reported by Piza *et al*. (2011). According to these authors, this rock

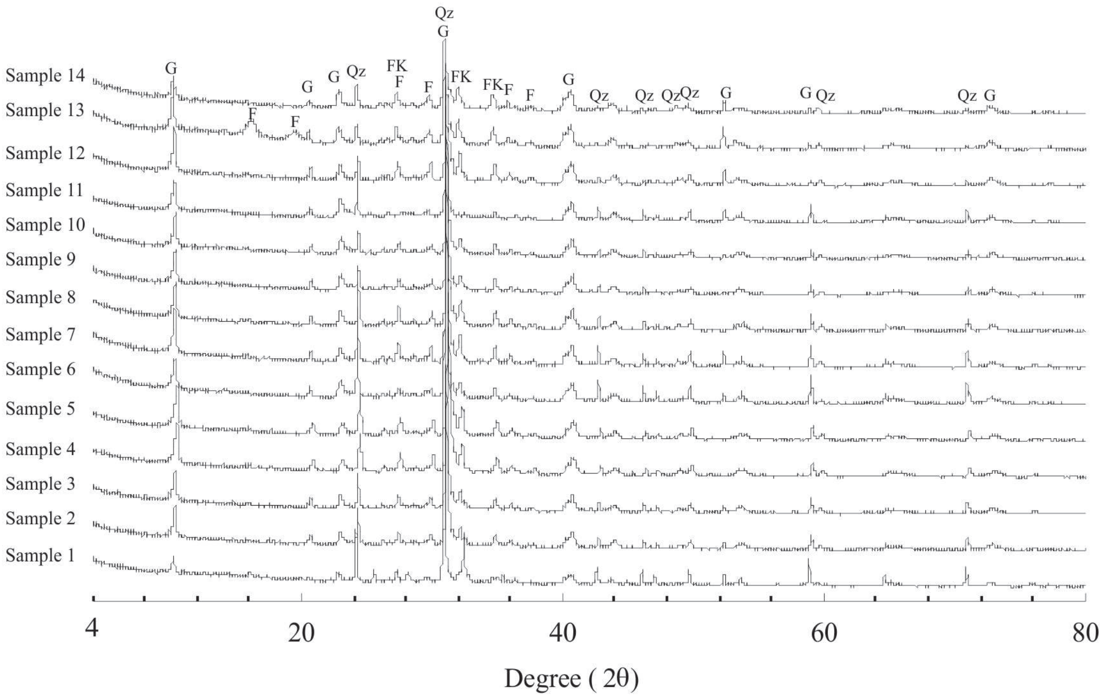

Figure 3. X-ray diffractograms of verdete rock samples, with cobalt radiation (1.7889 nm), scanning at 4-80 degrees (2θ). G: glauconite; Qz: quartz; F: feldspar; and FK: potassium feldspar.

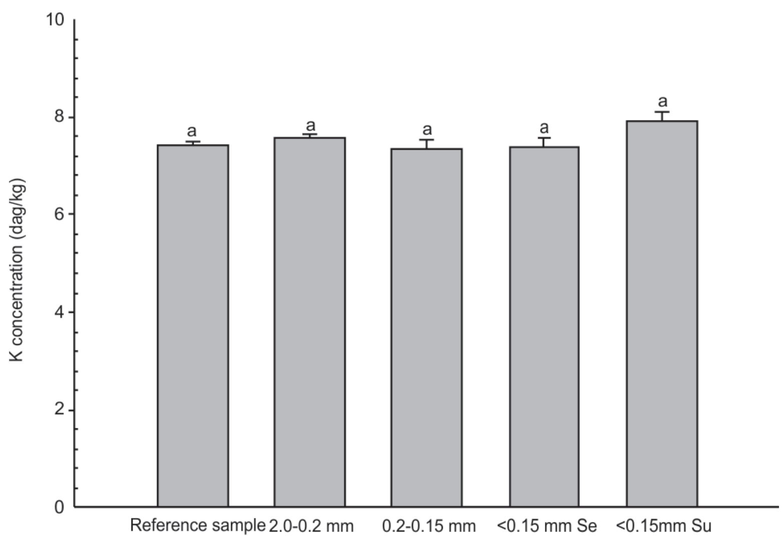

Figure 4. Total K contents in verdete rock (reference), in the size fractions between 2.0 and 0.2 mm; between 0.2 and 0.15 mm; less than 0.15 mm sedimented (Se) and less than 0.15 mm suspended (Su). Means followed by the same letter in the columns are not significantly different by the Tukey test at 5% probability. (I) represents the standard error of the mean.

contains on average 37% of glauconite, 24% quartz, 14% of light-brown clay matrix, 11% kaolinite, 7% iron oxides, 7% of muscovite and occurrence of feldspar. The occurrence of other potassic minerals in, such as vermiculite, chlorite and illite was also reported (Silva *et al*., 2012; Piza *et al*., 2010).

The concentration of K through physical fractionation was not achieved, certainly because the process was not able to separate quartz from glauconite. It is believed that the rock has great homogeneity in the distribution of minerals, thus these are broken in the same size by grinding and the separation by sieving or sedimentation is not possible. These results are in line with the study by Soni (1990), who tested a similar process to concentrate K from glauconitic sandstones. This author argues that the physical separation of glauconite, mainly from quartz, the second most abundant mineral in verdete rock (Piza *et al*., 2011), is not achievable through gravimetric processes because of the large variation in glauconite density, between 2.4 and 2.9 g/cm^3, coinciding with the quartz density, around 2.65 g/cm^3. However, Piza *et al*. (2011), after the granulometric analysis of verdete rock, observed with a binocular microscope that the quartz is separated from glauconite starting from the granulometric fraction below 0.105 mm. However, these authors found low concentrations of K in the fractions. Mazumder *et al*. (1993) state that glauconite is completely separated from other minerals in glauconitic sandstones starting from fractions with particle size < 0.15 mm.

CONCLUSIONS

Potassium contents vary markedly between verdete rock samples and are higher in rocks less weathered, harder and of more intense green, tending to blue.

Verdete rock has low reactivity in water or 2% citric acid.

Glauconite and feldspars are the crystalline mineral sources of K in verdete rock.

Although physical fractionation and sedimentation in water have proven ineffective processes for the concentration of K from verdete rock, their total K contents are promising for the development processes of solubilizing or concentration of K.

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