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Glauconite as a potential source of potassium in Brazilian agriculture - a review[1]

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ABSTRACT - The high Brazilian dependence on potash fertilizers has encouraged the agricultural and forestry use of silicate rock powders. Dozens of rock powders, including glauconitic rocks produced in several states in Brazil are currently registered with the Ministry of Agriculture and Livestock (MAPA) as Soil Remineralizers, being generally known as alternative sources of K. The indication of these materials as potassium fertilizers is based on the high Brazilian dependence on K from abroad, which reached around 96% in the 2021 and 2022 harvests. Glauconitic rocks, such as verdetes, slates, and glauconitic siltstones occur in extensive areas in the State of Minas Gerais (Abaeté, Quartel Geral, Cedro do Abaeté, Sete Lagoas, Matutina, and São Gotardo), predominating in the Serra da Saudade formation as the sedimentary cover of the São Francisco Craton. In these rocks, K-K_2O contents generally vary in the range of 5-12% m/m. It is soluble in water (0.1-1.4%) and 2% citric acid (0.7-2.3%). In these rocks, the degree of glauconite crystallinity is variable due to different formation conditions and degree of alteration by weathering, especially in rocky outcrops. The use of natural glauconitic rocks as potassium fertilizers has shown low agronomic efficiency due to their low solubility and reactivity. Physical and thermal treatments in the presence of fluxing, chemical (acid or alkaline attacks), and biological (*Acidithiobacillus* sp.) agents have shown potential for dissolving glauconites. However, studies showing the financial viability of these materials are necessary for both such treatments and their direct use as fertilizers.

Key words: Verdetes. Glauconitic Siltstone. Potash Fertilization.

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INTRODUCTION

Potassium (K) is one of the nutrients most used as fertilizers in Brazilian agriculture due to the low natural fertility of soils and the high rates of absorption of this nutrient by crops (SCHUELER *et al*., 2021; SIPERT; COHIM; NASCIMENTO, 2020). Brazil is the world's second-largest consumer of potassium fertilizers, with a historical external dependence that reached around 10.5 Mt in 2019, representing 96.5% of internal KCl consumption that year (BRASIL, 2021). Russia, Canada, and Belarus are the main K exporters to Brazil (FARIAS *et al*., 2021). Thus, the great Brazilian dependence on K, associated with the prospect of exhaustion of internal reserves of sylvinite, recurrent increase in international KCl prices (due to fluctuations in exchange rates and sea freight prices), and decrease in supply due to sanctions to Eastern European countries (including Belarus), places K as a strategic issue for Brazil.

All this dependence related to international politics and trade has encouraged Brazilian researchers to investigate Brazilian sources of K, *e.g.*, sources of potassium aluminosilicates (RAMOS *et al*., 2017; ROSA-MAGRI *et al*., 2012; SANTOS *et al*., 2015; SILVA *et al*., 2012a, b). The effects of the 2008 global economic crisis included the rise in fertilizer prices to uncommon levels (GÓMEZ-OLIVER *et al*., 2008) and research into alternative K sources developed over the last five decades has been intensified. Since then, the use of silicate rock powders has been debated again in different forums. In the following years, this resulted in the creation of official measures to encourage and regulate the agricultural use of such materials, industrial by-products, or those with primary processing. Thus, the National Solid Waste Plan (BRASIL, 2010), the Normative Instruction (IN) 05/2016 (BRASIL, 2016) which regulates Soil Remineralizers, and the recent National Fertilizer Plan (BRASIL, 2021) emerged.

Currently, dozens of rock powders are registered as Remineralizers with the Ministry of Agriculture and Livestock (MAPA). Some of them contain the micaceous mineral glauconite as the main source of K found in rocks such as verdetes, slate, and glauconitic siltstone. The low solubility and natural reactivity of these minerals have encouraged the development of thermal (EICHLER, 1983; MAZUMDER *et al*., 1993; NASCIMENTO; LOUREIRO, 2004; SANTOS *et al*., 2015, 2016; VALARELLI; GUARDANI, 1981), chemical (NASCIMENTO; LOUREIRO, 2004; SANTOS *et al*., 2016, 2017) and biological (MATIAS *et al*., 2019) processes to increase the fertilizer value of glauconitic rocks and/or produce new soluble products.

Thus, this study investigated the use of natural glauconitic rocks as an alternative source of K and different treatments to increase the solubility of these materials to obtain K, as robust demonstrations of the financial viability and use of these materials in Brazilian agriculture are scarce. In addition, developing new extractors and characterization methods of these materials is necessary to predict their efficiency as K sources in the short, medium, and long terms. Therefore, the objective of the present review was to investigate the occurrence, characteristics, and agronomic potential of glauconitic rocks as alternative potash fertilizers in Brazil.

GLAUCONITE: CHARACTERISTICS OF THE MINERAL AND BRAZILIAN MINES

Definition and physicochemical properties of glauconite

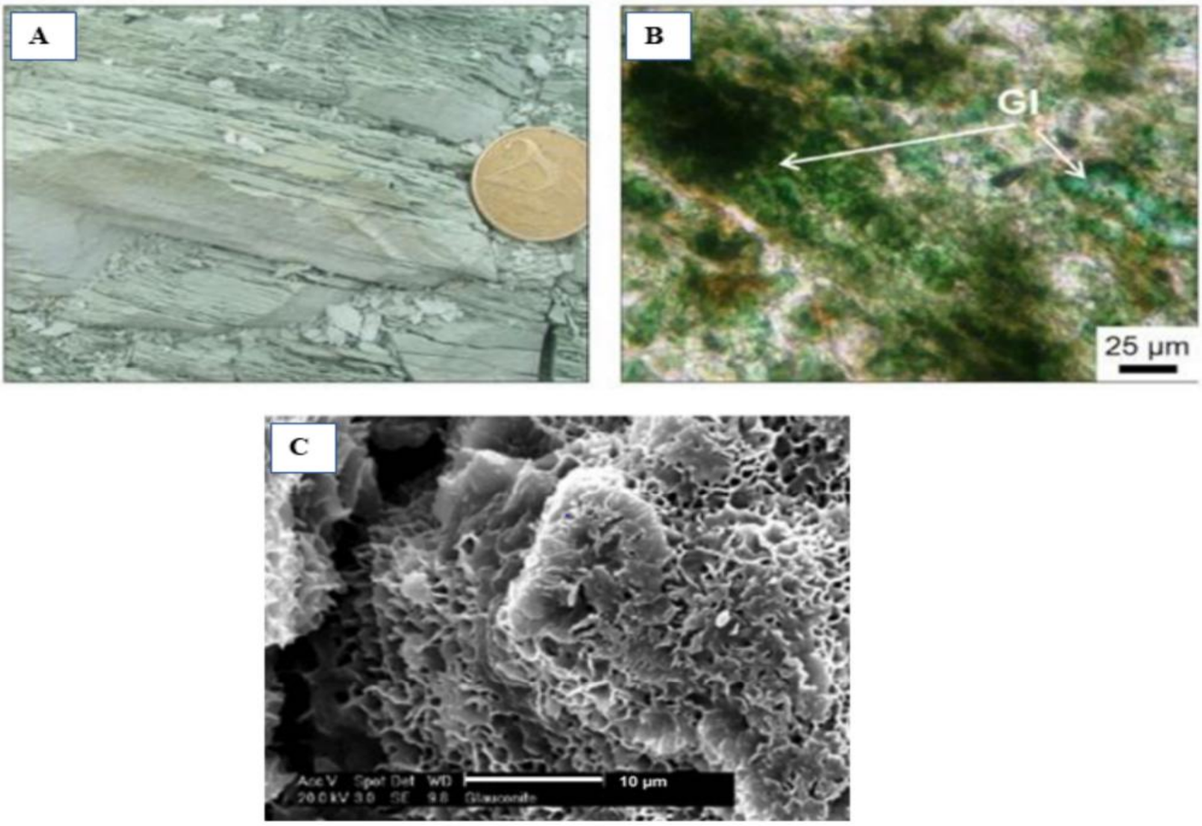
Glauconite is a mixture of minerals with a 2:1 dioctahedral structure, which is rich in K and Fe. It belongs to the monoclinic crystalline system whose origin is associated with clays of the illite group (LÓPEZ-QUIRÓS *et al*., 2020). The International Mineralogical Association (IMA) describes glauconite as a green-appearing mineral, which is a Fe-rich and charge-deficient phyllosilicate in the dioctahedral interlayer. Its simplified chemical structure is as follows: $K_{0.8}\,R^{3+}_{1.33}R^{2+}_{0.67}Al_{0.13}Si_{3.87}O_{10}\,(OH)_2$, which $^{VI}R^{2+}/(^{VI}R^{2+} + {}^{VI}R^{3+}) \geq 0.15$ and $^{VI}Al/(^{VI}Al + {}^{VI}Fe^{3+}) \leq 0.5$, where R is Fe (RIEDER *et al*., 1998).

We consider that the term glauconite was created by Brongniart (France; 1823) although older citations of its discovery and naming exist (TEDROW, 2002). Possibly, the word glauconite comes from *Glaukos* (Greek) meaning bluish green (MCRAE, 1972). According to Roe (2021), glauconite is a green mineral (Figure 1A and 1B). The internal structure of glauconite is similar to a honeycomb (Figure 1C) which allows the retention of water and nutrients inside (WILSON; WILSON; PATEY, 2014).

According to Tedrow (2002) glauconite is known by the following terms: I. *Glauconite* - generally greenish aggregates found in unconsolidated deposits and sedimentary rocks, containing high levels of Fe and minerals analogous to illite (minerals consisting mainly of hydrated Al, K, and Fe silicates); II. *Greensand* - generally unconsolidated sand-sized particles; practically all particles are green due to the high proportion of glauconite; III. *Greensand marl* - a term rarely used to describe unconsolidated glauconite-rich deposits; IV. *Lime marl* - this term may be used when earthy glauconite deposits contain a high proportion of calcium carbonate or dolomite.

The genesis of glauconite is described by the process called glauconization, which can be explained through the precipitation-dissolution-recrystallization theory (ODIN; FULLAGAR 1988; ODIN; MATTER, 1981). Glauconite formation theories have been addressed in

Figure 1 - **A**. Detail of glauconitic siltstone extracted in Serra da Saudade (MG, Brazil). **B**. Photomicrograph (200x, with parallel light) showing the siltstone matrix formed by the matrix with glauconitic grains (Gl) from Moreira *et al.* (2020). **C**. Internal structure of glauconite extracted on the Isle of Wight (England). Field of view ~44 µm wide (ROE, 2021)



recent studies such as that by López-Quirós *et al*. (2020) which can be summarized as a two-step glauconization: (I) formation of glauconitic smectite of fecal and sedimentary origin, which is poor in K and rich in Fe^{+3} in the octahedral layer and (II) replacement of Fe^{+3} by Fe^{+2}, with gradual enrichment of K^+ and forming glauconitic K-rich mica. As the glauconization process progresses, some chemical compounds are concentrated (K_2O: 3-8% or more; Fe_2O_3: 10-28%) whereas others are diluted (Al_2O_3: 20-0%).

For López-Quirós *et al*. (2020), an increase in negative charges occurs due to the high degree of isomorphic substitution (mainly of Fe^{+3} by Fe^{+2}) in the glauconization process, causing attraction of metals such as K into the structure of this mineral. In general, glauconitization is a low-temperature diagenetic process that can be classified based on the K_2O concentration (ODIN; FULLAGAR, 1988; ODIN; MATTER, 1981). The differences between glauconite and other green clay minerals, such as Fe-illite or celadonite, are unclear. Thus, knowledge about the nature of glauconite is still challenging.

Also, according to Odin and Matter (1981), different percentages of Fe_2O_3 are found in the average composition of illitic (<10%) and glauconitic (>15%) minerals. Therefore, glauconite is richer than illitic minerals in Fe. In general, the mineral glauconite shows great physicochemical variability. Analysis of glauconite and glauconitic siltstone (identified in Brazilian soils) indicates that these rocks have the supply potential the K needed for agriculture.

Occurrence of glauconite in the world and Brazil

Global glauconitic deposits date to the Precambrian period. In New Jersey (USA), they date from the Cretaceous and lower Tertiary periods. Although in New Jersey glauconite deposits are generally unconsolidated (sedimentary) rocks, in many parts of the planet glauconite is part of the composition of consolidated rocks (such as sandstone, shale, calcite, and dolomite), which are rich in fossil shells (TEDROW, 2002).

In Brazil, the main region with the incidence of glauconite comes from consolidated rocks in the Serra da Saudade Formation (State of Minas Gerais). They are part of the Ediacaran/Bambuí group (early Cambrian period), being a sedimentary cover of the Western São Francisco Craton (Figure 2A and 2B) (MOREIRA *et al*., 2020). The Serra da Saudade Formation is composed of sedimentary pelitic-sandy materials, and there are rocks (including rhythmite and pelitic-psammitic), sandstones with massive cross-stratification (green siltstone, glauconitic silt, phosphatic rhythmite, reworked carbonate), and lower occurrence of limestone (MOREIRA *et al*., 2021).

Studies by Moreira (2020) report differences between the glauconitic siltstone mentioned above and verdete, *e.g.*, glauconite concentration <37% and K_2O in the range of 6.0-7.3% in verdete, interspersed with rhythmites and sandstones and a platform environment shallower than that of glauconitic siltstone. Glauconitic siltstone has greater glauconization and this greater rock maturity provides a higher concentration of K_2O and a greater capacity for cation exchange. Verdete has a lesser degree of cation exchange capacity (rock with a lower degree of glauconization).

In Brazil, rocks as potential sources of K were mapped in a study by Kulaif and Góes (2016) and are shown in Table 1. The municipality of Matutina is highlighted by the total volume of verdete (green siltstone) in the order of 1.64 billion tons, with a K_2O content of around 8% (w/w).

The municipality of Quartel Geral is highlighted by the concentration of K_2O (9.4-10%), with a total reserve volume of around 0.5 billion Mg (Table 1). The measured reserve shows a high level of reliability and economic viability in its indicators because the drilling holes and other processes were carried out in a short spacing. The volumes determined in the indicated reserve are more imprecise because the spacing between the sampling of drilling holes and other processes is greater, and the geological indicators in these areas have few investigations works.

Glauconite-rich rocks were identified in the State of Minas Gerais (Serra da Saudade formation), with emphasis on the municipalities of São Gotardo, Matutina, Tiros, Cedro do Abaeté, Paineiras, Quartel Geral, and Santa Rosa da Serra (Figures 3A and 3B) (MOREIRA, 2015). Also, according to Moreira (2015), verdete and glauconitic siltstone occur in this region. In Brazil, these rock reserves are the most important in area, volume, and economic aspects due to the presence of K. The reserve layer has an average thickness of 70 m and there are frequent outcrops visible from a distance (Figure 3A-3C) even in satellite images (Figure 3D). In situations of weathering, rocks become white and fractured, or reddish due to the oxidation of Fe^{+2} (SANTOS *et al.*, 2015).

THE USE OF GLAUCONITE AS A POTASSIUM FERTILIZER IN BRAZILIAN AGRICULTURE

Contextualization of the problem of potassium fertilizers in Brazilian agriculture

Figure 2 - **A**. Region of glauconitic resources comprising the municipalities of São Gotardo, Matutina, Tiros, Cedro do Abaeté, Paineiras, Quartel Geral, and Santa Rosa da Serra (MG, Brazil) from Moreira (2015). **B**. Image (Google Earth) of the municipalities of São Gotardo, Matutina, Cedro do Abaeté, and Quartel Geral (Serra da Saudade Formation, MG, Brazil) where the glauconitic verdete resources are located

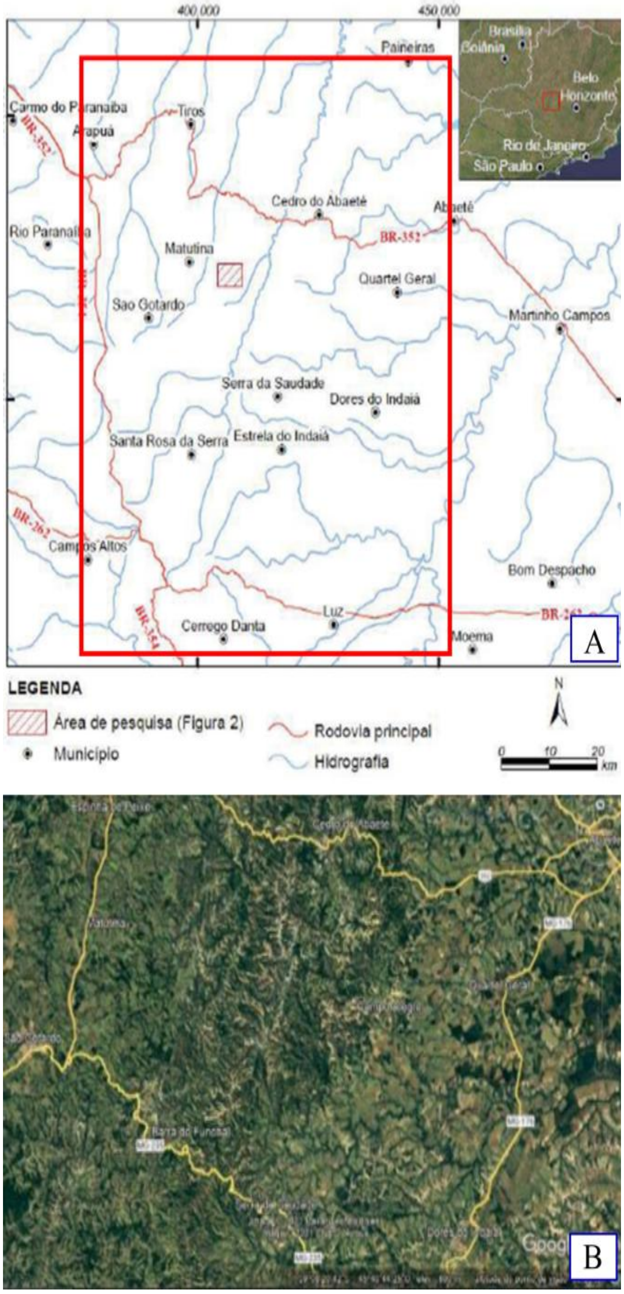


Table 1 - Official resources of K-K_2O in Brazil (in thousand Mg) in 2013 (KULAIF; GÓES, 2016)

Location (*)	Substance	Measure		Indicated		Inferred	
		Ore	K_2O (%)	Ore	K_2O (%)	Ore	K_2O (%)
AMAZONAS		939,305	n.p.	63,020	n.p.	150,220	n.p.
Itacoatiara	Evap - Sylvinite	446,300	18.32-21.57	63,020	20.47-20.48	150,220	16.41-20.19
Nova Olinda do Norte	Evap - Sylvinite	493,005	16.79				
MINAS GERAIS		1,915,741	n.p.	1,081,397	n.p.	261,421	n.p.
Andradas	RoSil - Alkaline	37,029	n.a.	20,412	n.a.		
Caldas	RoSil - Alkaline	9,521	n.a.	10,911	n.a.		
Matutina	RoSil - Verdete	1,100,132	8.18	537,728	8.09	5,714	7.5
Poços de Caldas	RoSil - Alkaline	28,677	n.a.	19,197	n.a.		
Quartel Geral	RoSil - Verdete	301,683	10.07	182,485	9.39	47,825	10.56
São Gotardo	RoSil - Verdete	438,699	7.5	310,664	7.5	207,882	7.5
SERGIPE		5,242,785	n.p.	5,379,000	n.p.	4,291,000	
Rosário do Catete	Evap - Silvinita	477,785	9.74				
Rosário do Catete	Evap - Carnalita	4,765,000	6.34	5,379,000	6.34	4,291,000	6.34
SÃO PAULO		38,782	n.p.	7,947	n.p.	585	n.p.
Águas da Prata	RoSil - Alkaline	38,782	n.a.	7,947	n.a.	585	n.a.
TOTAL		8,136,613	n.p.	6,531,364	n.p.	4,703,226	n.a

Notes: (*) When reserves or polygonal areas cover more than one UF and/or municipality, the location informed is the mine's base or that informed by the company; Evap: Evaporites; RoSil: Silicate rocks; n.a.: not available; n.p.: not pertinent. In the case of RoSil - Alkaline, as in many cases the reserves were saved to take advantage of more than one type of product, the potassium content was not determined in many of them, precluding to calculation of the mean content per municipality

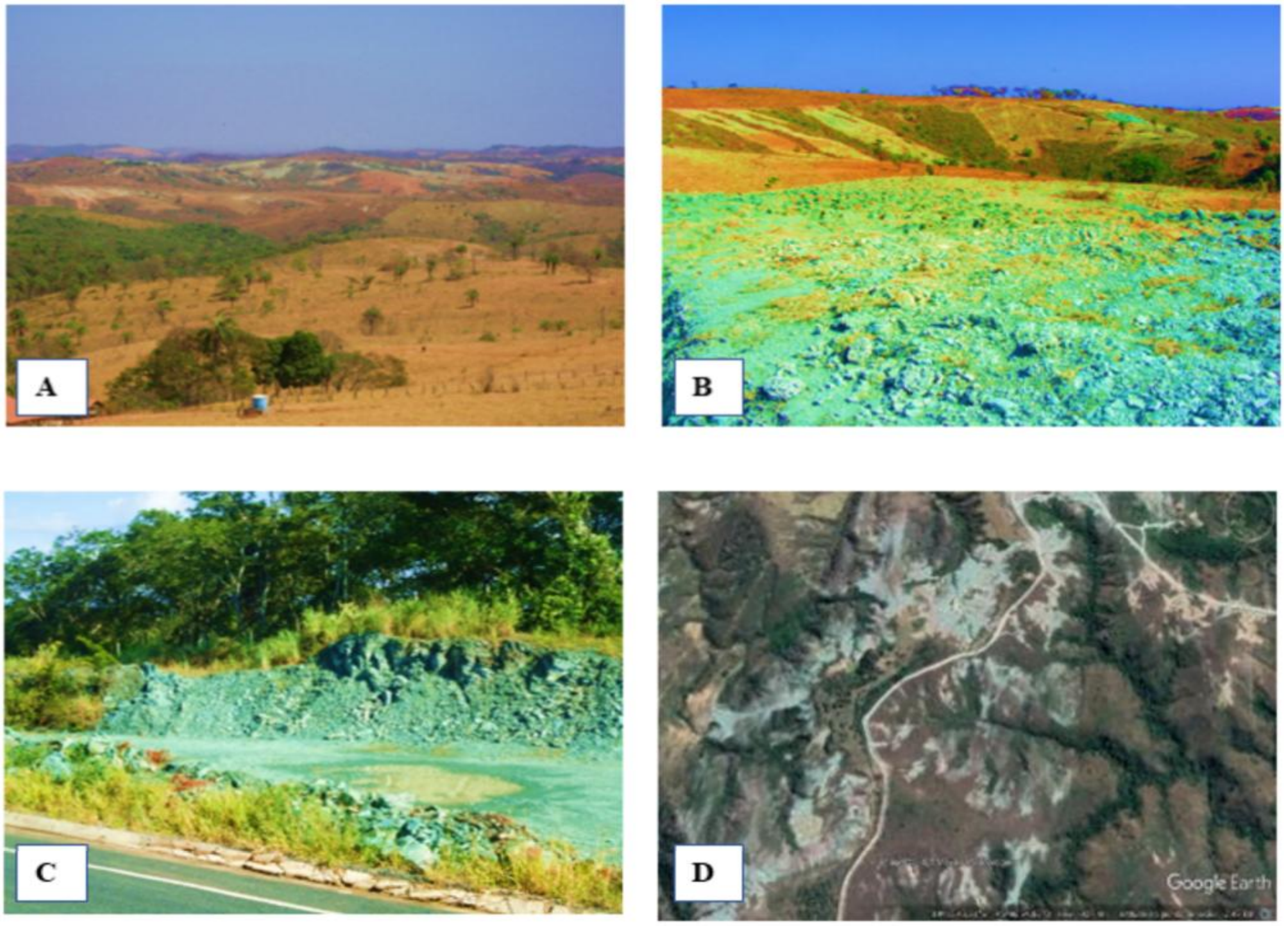
The global growth of agriculture has been highlighted in tropical areas, making Brazil a country with great potential to lead the world supply of agricultural *commodities in* a short time, which it already does for various products (ALVES *et al*., 2021). However, Brazil is highly dependent on external fertilizer supplies, and dependence on potassium fertilizers is the most critical. To meet this demand, Brazil currently imports volumes greater than 96% of the total K used as fertilizer (BRASIL, 2021).

As K is a strategic macronutrient in the agricultural production system, contextualizing traditional and alternative sources for Brazil is important. Several minerals and K salts are extracted and used as fertilizers in agriculture. They are also called potash, a term used since the 19th century when the K used as fertilizer came from potash (MIKKELSEN; ROBERTS, 2021). Potash is a term that covers many individual K fertilizers, *e.g*.: KCl (potassium chloride); K_2SO_4 (potassium sulfate); $K_2SO_4MgSO_4$ (potassium magnesium sulfate); KNO_3 (potassium nitrate; Chilean saltpeter), and $NaNO_3$ + KNO_3 (mixed sodium-potassium nitrate from Chilean saltpeter), which are essentially evaporites (this term is used to describe minerals formed by chemical precipitation in an aqueous environment). Despite the abundance of carnallite in the Sergipe-Alagoas sedimentary basin, the occurrence of this ore at depths greater than 600 m seems to be the biggest obstacle to its exploration.

In addition to the occurrence of K (sylvinite and carnallite) in the Sergipe-Alagoas sedimentary basin, the occurrence of a significant reserve of sylvinite was found in the Amazon region in 1957 (IBRAM, 2011). Thus, the discovery of new K deposits identified in the State of Amazonas (Autazes region) is highlighted. These deposits occur mainly in the Madeira River basin (Autazes and Fazendinha), and there is no prospect of exploration in the short-medium term due to issues related to impacts on the environment and local communities. These deposits are voluminous (with promising K content), occurring just 120 km from Manaus at depths in the range of 680-900 m (KIEFER; UHLEIN; FANTON, 2019).

Thus, alternative minerals also exist containing lower concentrations of K, with low solubility compared to evaporites (Table 2). Glauconite is included in those minerals where K is a silicate crystalline lattice. The process of its dissolution generally occurs by chemical attack, heat treatment, and/or mechanical

Figure 3 - A-C. Outcrop of glauconitic verdete in the Serra da Saudade Formation (Cedro do Abaeté, MG, Brazil). **D.** Image (Google Earth) of the verdete outcrop in the region of Quartel de São João (Municipality of Quartel Geral, MG, Brazil)



activation. However, the stability of minerals such as glauconite is related to their structural characteristics (hardness, solubility, surface area, etc.) and the degree of weathering in the environment where these minerals will be applied. This occurs because the environment, combined with other factors (such as specific area), accelerates the weathering of these minerals after a certain moment (KÄMPF; CURI; MARQUES, 2009). Thus, for more expressive and representative results regarding the application of glauconite and other remineralizers in the soil, experiments are necessary in Brazilian tropical soils. Thus, it is possible to know the behavior of these minerals in an environment where the chemical balance is subject to the characteristics of highly weathered soils.

Use of glauconitic rocks as a source of K in agriculture

The historical and growing Brazilian external dependence on K, which is associated with recurrent increases in the prices of these commodities, has stimulated the search for Brazilian sources of K since the 1970s, with emphasis on glauconitic silicate rocks (aluminosilicates) of great occurrence in the country. Research carried out in the period 1970-1980 (EICHLER *et al*., 1983; LEITE, 1985; LOPES *et al*., 1972) with the glauconite-rich verdete rock showed a low efficiency of this rock as a source of K for short-medium term effects when used *in natura*.

Thus, the number of studies and/or incentives for the use of these sources was minimal for around two decades. Due to the global economic crisis, however, a sharp increase occurred in fertilizer prices (including KCl) from 2008 onwards, and the topic “Use of silicate rocks as sources of K” was once again noticed. This resumption of the theme gained some support with the national solid waste policy (LAW 12,305/2010; BRASIL, 2010) which established the need to manage solid waste in Brazil, encouraging the reuse, recycling, and treatment of these materials. In addition, the

Table 2 - Some characteristics of the main minerals containing K

Mineral	Family	Formula	%K_2O	Relative Solubility
Silvite		KCl	60	High
Glauconite	Mica	$(K,Na)(Mg,Fe^{2+})(Fe^{3+},Al)(Si,Al)_4O_{10}(OH)_2$	10-15	Average
Phlogopite	Mica	$K_2Mg_6Si_6Al_2O_{20}(OH)_4$	11	Low
Biotite	Mica	$K_2Fe_6Si_6Al_2O_{20}(OH)_4$	9	Low
Muscovite	Mica	$KAl_3Si_3O_{10}(OH)_2$	10	Very low
Leucite	Feldspathoid	$KAlSi_2O_6$	21	Average
Nepheline	Feldspathoid	$(K,Na)AlSiO_4$	15	Average
K-feldspar	Feldspar	$KAlSi_3O_8$	16	Very low

Adapted from Moreira (2015) and Mikkelsen and Roberts (2021)

Fertilizers Law (6894/1980) (BRASIL, 1980) was amended by Law 12,890/2013 (DECREE 8,384/2014; BRASIL 2013), which included remineralizing agents in the category of agricultural inputs and defined it as a "*mineral material that suffered only reduction and size classification by mechanical processes and changes soil fertility indexes by adding macro and micronutrients for plants, as well as promoting an improvement in the physical or physicochemical properties or biological activity of the soil*".

The Normative Instruction (IN) 05/2016 (BRASIL, 2016) was another milestone that defined (as warranty items) that soil remineralizers must have contents of K-K_2O ≥1.0%, ∑K_2O, MgO, and CaO ≥9.0% and contents of As, Cd, Hg e Pb lower than 15, 10, 0,1 e 200 mg kg^{-1}, respectively. In addition, the IN has made it possible for these materials can be sold as *filler*, powder, or crumble. Recently, the National Fertilizer Plan (BRASIL, 2021) endorsed and encouraged the use of remineralizers as an alternative to decrease external dependence on K by 2050.

Regarding the use of glauconite directly in the soil after grinding, Rudmin *et al*. (2019a) evaluated wheat fertilization in Western Siberia (Tomsk Region) using this alternative product. In this research, Meso-Cenozoic glauconitic rock from Western Siberia (6.4% K_2O) was used in a dose corresponding to 128 kg ha^{-1} K_2O in a soil that previously had 224 mg kg^{-1} of K. The results were positive applying a grinding fraction of less than 2 mm in this soil whose natural fertility was high compared to a large part of Brazilian soils. Its productivity increased by 18.4% and plant height increased by 32.3%; the productivity of the area with glauconite was 1,613 kg ha^{-1} *versus* the control area (without fertilization) with 1,362 kg ha^{-1}. In this experiment, it is worth highlighting that the original content of K_2O in glauconite was reduced by 24% during the wheat crop cycle, indicating residual release for the next 2-3 crop cycles. These results are relevant, but the data could be better explored using a more robust statistical analysis. In tropical soils where there is generally a low concentration of K, sources with higher concentration and a rapid release of K are necessary. Thus, a change in its physical and/or chemical structure is necessary to solubilize the K contained in the rock.

The use of silicate rocks as remineralizers is closely related to the rate of weathering in tropical soils. In a panoramic approach to rock powders, Swoboda, Döring and Hamer (2022) pointed out that dissolution rates are generally related to: *i. types of rock and minerals*: less crystalline rocks have an easier availability of K; *ii. size of particles added to the soil*: *fillers*, powders, or crumbs are found on the market, but smaller particles (therefore with a larger surface) favor the dissolution of products; *iii. amount of material applied to the soil*: in the case of glauconite, the productivity obtained is proportional to the K_2O content of KCl; *iv. soil type*: tropical soils with low mineral content, easily weathered, and with a high leaching rate favor the dissolution of applied materials; *v. plant species*: plants with longer cycles or sequential crops can make better use of K from these sources, and higher dissolution rates can be provided by plants with biological N fixation, mycorrhizal associations, and/or fasciculated roots; *vi. climatic issues*: in areas with higher temperature and increased rainfall, the rate of dissolution of minerals increases; *viii. time and/or duration of application*: as silicate rocks are poorly soluble in water (Table 2), they require considerably longer time to dissolve; and *viii. modifications that occur in the soil after application*: aspects related to physical, chemical (mainly its chemical balance in the soil), and biological properties affect K availability.

Thus, the feasibility of applying glauconite is closely related to the behavior of this mineral after its application to the soil. The dissolution rates of glauconite obtained by modeling are in the range of -4.80 log mol·m^{-2} s^{-1}, *i.e.*, the dissolution of glauconite is 182,000 times faster than that of potassium feldspars (PALANDRI; KHARAKA, 2004), but the difference in crystallinity between them was not considered. Characteristics that affect the K availability of silicate rocks, such as verdete and glauconitic siltstone, are fundamental to positioning a product as a remineralizer on the market.

MANUFACTURING PROCESSES OF GLAUCONITE-BASED FERTILIZERS

The main advantages of Brazilian sedimentary rocks containing glauconite (such as verdete and glauconitic siltstone) for use in fertilizer production are the abundance of surface deposits and considerable levels of K_2O (8-12%). Despite these characteristics, the low solubility of these rocks requires treatment, *i.e.*, activation strategies so that K can be available to plants in the short term (SCHIMICOSCKI; OLIVEIRA; ÁVILA-NETO, 2020). There are several routes to obtain this activation. They can be physical, chemical, and/or biological, and treatments can be directly or indirectly combined leading to the activation of this mineral for agricultural use.

Activation of glauconite by physical processes

Calcination of glauconite rocks has shown efficiency in increasing the solubility of K in verdete or glauconitic siltstone. In the solubilization of nutrients in rocks, this technique is highlighted by its operational ease and possibility of association with other solubilization techniques. Santos *et al*. (2015) tested the efficiency of various salts as fluxes in the production of thermopotassium from verdete. The results of this study are simplified in Table 3, showing that $Ca_3(PO_4)_2$, $CaCO_3$, and $CaSO_4$ are little effective in the thermal interaction with verdete. On the other hand, this study made it possible to qualify the efficiencies of LiCl and $CaCl_2$ (high efficiency) and Na_2CO_3 and NaCl (medium efficiency) in this process. This study also made it possible to indicate that the temperature of 300 ºC is insufficient to promote satisfactory verdete -flux interactions.

To model the effect of heat treatment on the formation of soluble K species, Santos *et al*. (2017) selected $CaCl_2$ as flux and varied temperature and the rock-flux relationship. Analysis of soluble K content as a function of temperature and verdete/fondant ratio allowed adjusting the multiple regression model (Eq. 1) that describes the soluble K content (y) as a function of these variables, and made it possible to choose 1.7 and 850 ºC, as the optimum proportion (m/m) and temperature, respectively.

$$y = -412 + 43.16p + 1.12t - 21.09p^2 - 0.000699t^2 + 0.034pt \quad R^2 = 0.81 \quad (1)$$

y = soluble K content (%);

p = proportion w/w verdete/$CaCl_2.2H_2O$ and

t = temperature (ºC).

In the same study (SANTOS *et al*., 2017), mineralogy analyses (X-ray diffractometry) made it possible to verify the transformations of K species in verdete and the formation of new species in the calcination products (Figure 4).

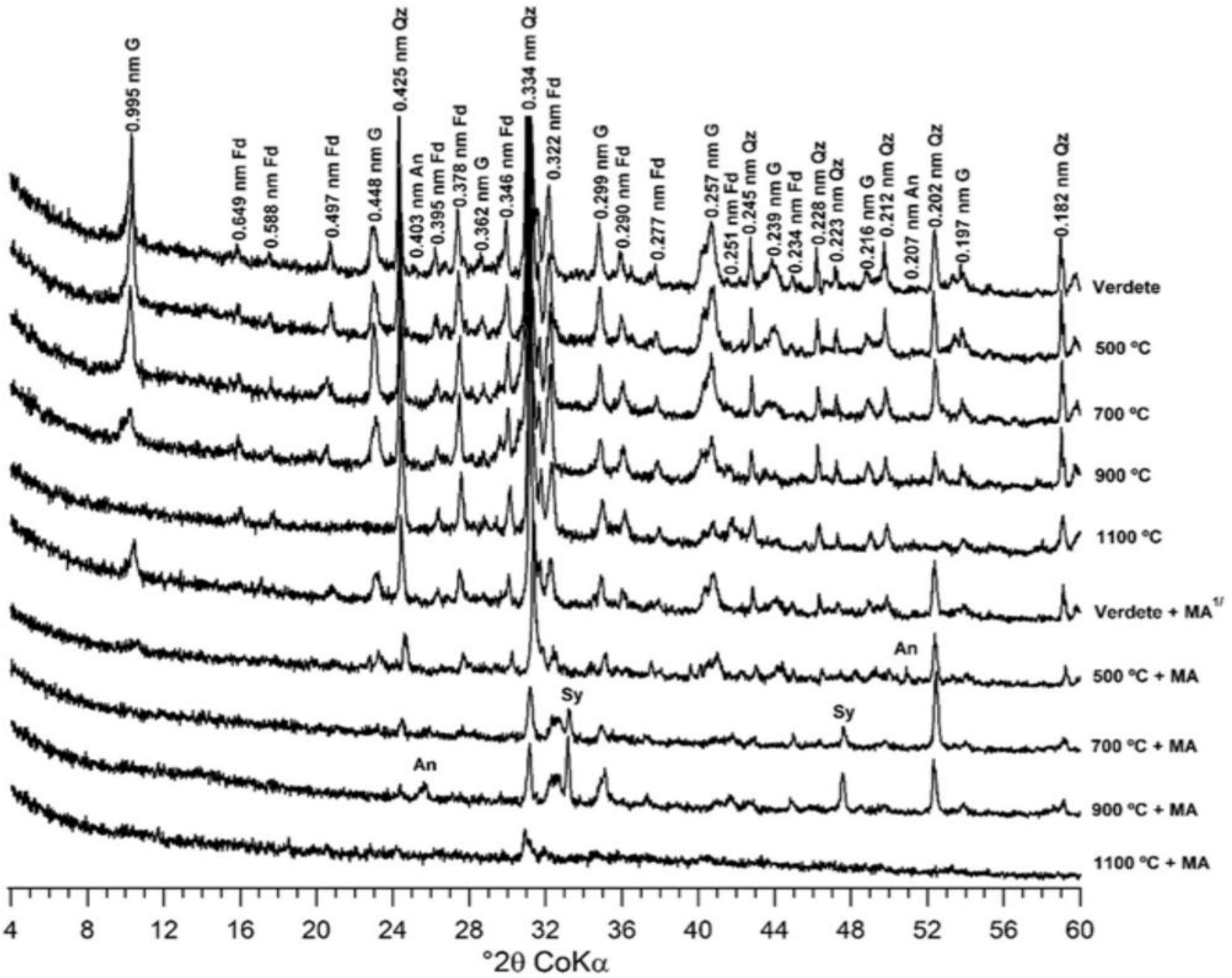
Dias *et al.* (2018) evaluated calcined glauconitic siltstone in the production of Arabica coffee (*Coffea arabica*). In this study, KCl was compared with calcined glauconitic siltstone (TK47; 7% K_2O, thermopotassium) and the product *Super Greensand* (glauconitic siltstone obtained by mechanical activation; 10% K_2O). Fertilization with 336 kg ha^{-1} of K_2O-TK47 was highlighted both in the sensorial analysis of the coffee drink (statistically

Table 3 - Percentages of K in water-soluble verdete for various fluxing agents, verdete/fluxant ratios, and calcination temperatures (SANTOS *et al*., 2015)

Fondant	Calcination temperatures (ºC)					
	300(1)	700	1100	300(2)	700	1.100
	%					
$CaCl_2.2H_2O$	0.98	39.79	30.09	1.09	70.41	100
$CaCO_3$	0.66	2.32	0.28	0.71	5.72	0.55
$Ca_3(PO_4)_2$	1.31	4.39	0.87	2.42	14.81	8.69
$CaSO_4$	1.41	3.60	0.30	1.67	4.37	1.44
LiCl	0.99	100	100	1.09	99.63	100
Na_2CO_3	1.01	14.30	1.72	1.85	38.33	41.91
NaCl	1.33	10.66	16.42	1.21	19.07	50.00

verdete/fondant ratio w/w 10/1; (2) verdete/fondant ratio w/w 10/2.5

Figure 4 - X-ray diffraction patterns of Verdete rock (VR) subjected to calcination in the presence of fluxing agent ($CaCl_2 \cdot 2H_2O$_MA) at various calcination temperatures. **G**: $[K_2(Mg,Fe)_2Al_6(Si_4O_{10})_3(OH)_{12}]$; **Qz**: quartz ($SiO_2$); **Fp**: potassium feldspar, microcline [$(KAlSi_3O_8)$]; **Sy**: sylvite (KCl), and **An**: anorthite ($CaAl_2Si_2O_8$). Data were collected using Co $K_{\alpha 1}$ radiation (λ =1.789 Å) (SANTOS *et al*., 2017)



* The samples used correspond to the ratio (ww^{-1}) MA/VR equal to 1/1

superior for this item), to the detriment of the activity of polyphenol oxidase (an enzyme extremely sensitive to the presence of chlorine). In addition, the production of grains the fertilization by 336 kg ha^{-1} of K_2O-TK47 was similar to split fertilization with 618 kg ha^{-1} of K_2O (KCl). This great efficiency of TK47 would be related to the fact that coffee is a long-cycle crop in which this fertilizer would have had good results concerning KCl.

In Brazil, the use of thermopotassium was also studied by Korndorfer *et al*. (2018) in sugarcane (*Saccharum* spp.) (Table 4). The study was conducted in the field during two harvests using two sources of K_2O, also comparing KCl with calcined glauconitic siltstone (TK47; 7% K_2O, thermopotassium). Regarding productivity, the TK47 fertilizer showed to be similar to KCl in the first harvest and superior in the second harvest, and the maximum productivity between both fertilizers was 133 Mg ha^{-1} (121 kg ha^{-1} of K_2O). Therefore, thermopotassium showed to be a viable fertilizer option in supplying K, still showing a residual effect over time.

In general, the studies differ regarding the addition of Ca in thermal treatment (calcination). Martins *et al.* (2015) evaluated calcination and alkaline solubilization using samples of verdete (Abaeté, MG, Brazil) with addition of limestone. In this study, the increase in the proportion of limestone *versus* verdete promoted a reduction in the value of K solubility indicators, as a greater volume of carbonate decreased the burning temperature. Thus, calcination was not sufficient for phase changes and K release. Then, the solubility of K in citric acid in verdete calcined alone increased concerning that which received limestone addition.

The use of nanotechnology is also an alternative to take advantage of K in glauconite. This can occur through: *i*. nanoparticle synthesis, *ii*. understanding of their physical, and *iii*. chemical properties, and organization of complex matter at the nanoscale using weak non-covalent interactions (ADHIKARI *et al*., 2015). In a study by Praveen *et al.* (2020), using corn (*Zea mays* L) in India, nanotechnology was used to prepare glauconite nanoparticles (GNP) and thus evaluate the release of K. A high-energy ball mill led to the change in the size of glauconite from the millimeter scale (mm) to the nanometer scale (nm). Five treatments and the absorption of K (by the aerial part of corn stalk) were evaluated during five collections of leaf samples, in each of the five successive cultivations, without addition of K. In the fifth leaf collection (dosage of 100 mg kg^{-1} K_2O), a statistical difference between GNP and KCl was not observed, *i.e.*, both sources had the same long-term effect.

Mechanical activation of glauconitic siltstone (MAGS) is also an important physical process for the use of glauconite in Brazil. Samples of Brazilian glauconitic siltstones show low dissolution, around 5% (PRATAP *et al.*, 2020). In this study, mechanical activation (8 h; 60% NaOH) before heat treatment (600 °C; 30 min) in recovering more than 95% K from glauconite was successful. The residue was composed mainly of quartz. This indicates the complete breakdown of the silicate matrix, which favors a greater solubility of this material.

The British company Verde Agritech explores glauconitic siltstone in São Gotardo (MG, Brazil) (Figure 5). It adopts mechanical activation as technology in its products, being currently the main Brazilian supplier of glauconite-based K. This mining company registered a patent application (BR 10 2017 019490 6) for this industrial process at the Brazilian Institute of Industrial Property (INPI). The text of this patent cites glauconitic siltstone as an innovative raw material, and the invention includes a process comprising the following steps: 1. selection of glauconitic siltstone, 2. rock fragmentation to specific parameters, 3. sieving, and 4. optional acidulation. In the end, materials with fertilizing and phytoprotective characteristics were obtained (INPI, 2019a, b).

Figures 5A and 5B show the initial and final characteristics of glauconitic siltstone, the mineral that originates the product “K Forte”, the main product sold by ‘Verde Agritech’. This product was registered with MAPA (MG 000662-9.000006; granted on 28/08/2020) as a simple mineral fertilizer, containing the following information: K_2O (4%) dissolved in tartaric acid (5%), NaF (0.5%), total K_2O (8%) and total Si (25%). This is the minimum guarantee.

Brasil *et al.* (2020) used the AMSG technique in their experiment (Quirinópolis, GO, Brazil) under experimental field conditions and successive cultivations of beans (*Phaseolus vulgaris* L.) and corn. A soil classified as Arenosol was used, with an initial K content of 30 mg dm^{-3} (Mehlich-1 extractor). Table 5 shows the productivity results for each treatment. The similar result (total dose of 222 kg ha^{-1} K_2O) is evident, comparing KCl *versus* glauconitic siltstone that received mechanical activation. Therefore, this technique tends to be consolidated on a large scale in Brazilian agriculture, mainly in Fertilization Systems,

Table 4 - Productivity (Mg ha^{-1}) of sugar cane (*Saccharum* spp.; variety SP832847) after adopting different sources and doses of K (harvests 2011/12 and 2012/13). Vale do São Simão sugar and alcohol plant (Chaveslândia, MG, Brazil) (KORNDORFER *et al*., 2018)

K_2O levels	1st harvest			2nd harvest		
	TK47	KCl	Mean	TK47	KCl	Mean
(kg ha^{-1})	Stem productivity (Mg ha^{-1})					
0	113	113.6	113.3	108.2	107.8	108
50	135.7	122.8	129.3	119.5	111.9	115.7
100	136.1	132.5	134.3	124.3	115.6	119.9
150	123.4	128.8	126.1	125.2	117.2	121.2
200	134.8	120.8	127.8	126.9	118.7	122.8
Mean	128.6 A	123.7 A		120.8 A	114.3 B	
	CV = 10.57% LSDsource = 7.65			CV = 7.79% LSDsource = 5.25		

[1] Means with the same letters do not differ significantly using the Tukey test (5% probability). TK47: thermopotassium and KCl: Potassium Chloride

i.e., in sequential annual crops (as is the case with soybeans) followed by corn in the 2nd harvest.

The use of mechanically activated glauconitic siltstone has been adopted by Brazilian farmers in perennial and annual crops, mainly due to the significant increase in the cost of imported K. However, the data from this study could be better explored using a more rigorous statistical analysis including regression with the applied doses of glauconitic siltstone.

As glauconite is a mineral with a slow release of K, large-scale annual crops in Brazil (such as soybeans and corn) are being fertilized with this alternative source of K, especially in successive crops. In the first and second harvests, the total dose of K_2O is being fully applied 30-90 days before the first cultivation based on soil fertility analysis.

Figure 5 - **A**. Exploration of glauconitic siltstone in São Gotardo (MG, Brazil); initial characteristic of the rock. **B**. Granulometric characteristic of the commercial product "K Forte", resulting from the mechanical activation of glauconitic siltstone (VERDE AGRITECH, 2022)

Table 5 - Comparison between the productivities (kg ha^{-1}) of beans and corn, using system fertilization in sequential crops with different doses and sources of K_2O

	Treatments	Fertilization System		Total K_2O	Productivity	
		1st Cultivation of Beans	2nd Cultivation of Corn		1st Cultivation of Beans	2nd Cultivation of Corn
		K_2O kg ha^{-1}			kg ha^{-1}	
1	Absolute control	0	0	0	2,824.50 c	6,994.00 b
2	Control with KCl	72	150	222	2,983.50 bc	7,286.00 b
3	Glauconitic siltstone 1x	222	0	222	3,035.25 bc	7,192.00 b
4	Glauconitic siltstone 2x	444	0	444	3,017.25 bc	7,488.00 ab
5	Glauconitic siltstone 4x	888	0	888	3,573.00 a	7,439.00 ab
6	Glauconitic siltstone 8x	1.776	0	1.776	3,233.25 b	7,854.00 a

Adapted from Brasil *et al.* (2020). Note: In the columns, the mean values with similar letters were not significantly different by Duncan's test ($p < 0.05$). Potassium from glauconitic siltstone was fully applied 30 days before sowing the beans. The letter *x* represents how many times the total dose of the conventional control with KCl was multiplied. The soil was amended with limestone to raise the base saturation to 70%; in addition, it received suitable fertilization with N and P for this productive ceiling of beans and corn

Studies by Rudmin *et al*. (2019b, 2020, 2022) evaluated the efficiency of mechanical intercalation of urea and glauconite using nanotechnology. *Slow-Release Fertilizers* (SRF) of this nitric-potassium complex were synthesized using a mechanochemical methodology, mixing glauconite with urea in different proportions. The objective of this work was to form urea and glauconite layers to minimize NH_3 volatilization and develop a slow-release fertilizer from K and N sources. The experimental production of a slow-release multifunctional fertilizer was the product with an interspersed urea and clay structure. The results obtained from the leaching experiment indicated a rapid increase in the leaching of K and N followed by a rate reduction after 6-7 days, possibly due to a reduction in the release of nitrate and ammonium provided by the fertilizer. In addition, germination tests were also carried out with oats (*Avena sativa*), and the synthesized fertilizer promoted greater seedling growth but with lower dry mass and germination rate relative to urea.

Activation by other treatments and other implications

Chemical treatments can be associated with a physical treatment to increase the potential of glauconite dissolution (DUARTE *et al., 2021*). The use of sulfuric acid (H_2SO_4) was addressed by Schimicoscki, Oliveira and Ávila-Neto (2020) to make K available. In this study, the efficiency of the hydrothermal methodology was evaluated by breaking glauconite structures in verdete at a low liquid/solid ratio to extract K. Potassium was released due to the structural damage caused by H^+ to the mica plates. The reaction occurred due to the replacement by H^+ in the K^+ interlayer, resulting in a partial release of Al^{3+}, Fe^{3+}, Fe^{2+}, and Mg^{2+} in the 2:1 layer. The results indicated that the release of K increased as the acid concentration increased, at temperatures in the range of 80-120 ºC, reaching 24% of K recovered.

Solubilization of glauconite from rocks such as verdete may also occur by adding organic acid. Duarte *et al*. (2021) verified K solubilization using oxalic (6% extracted K) and citric (2.3% extracted K) acids. In the extraction of K, oxalic acid was more efficient due to the formation of metal-oxalate complexes with the metals Al, Fe, Mg, Mn, Ni, and Zn in verdete.

Research carried out with Brazilian rhizospheric microorganisms indicated that this material could provide greater growth of microorganisms that produce siderophore chelating agents as glauconite contains significant levels of Fe. This material would also provide metal chelation, enhancing the solubilization of this potash mineral (CARA; RIZZO; CUNHA, 2010). In another study, Matias et *al*. (2019) used a bacteria that promotes sulfur oxidation (*Acidithiobacillus thiooxidans*) to reduce pH and solubilize verdete. The results indicated a significant reduction in pH (from 4.2 to 0.57) in 49 days of incubation, providing an increase in the solubility of K in this rock (6.6% of the total content). These results suggest another low-energy method to activate glauconitic rocks using a biological route.

Given the great diversity of materials in terms of chemical composition (content of nutrients and potentially beneficial elements), mineral characteristics (mineral composition, degree of crystallinity, solubility, reactivity, and granulometry), edaphoclimatic conditions of use (type of soil, content of clay, organic matter content, acidity, Si activity, microbial activity, water regime, temperature, leaching rates, etc.), and cultural (types of culture and/or nutrient acquisition mechanisms, and form of application), robust studies on the short to long term effects of these materials on soil are still needed despite legal advances on the use of aluminosilicates as soil remineralizers in Brazil.

Considering the significant variety of materials and conditions of more operational use of remineralizers, developing chemical extractors that enable a good prediction of the efficiency of these rock powders is necessary. Thus, we can say that the soluble contents in specific extractants are useful to better assess the feasibility of their use.

CONCLUSIONS

1. The search for food in the world is growing, and Brazil tends to expand its role as aprovider of agricultural *commodities*. The exploration of glauconitic deposits in Brazil is a potential to reduce Brazilian external dependence on K. This study raised information about glauconite deposits in Brazil and their potential for direct or indirect use as an alternative source of K for Brazilian agriculture;
2. The use of glauconitic rocks as raw material to produce fertilizers with medium-high solubility or reactivity has shown to be effective, but financial studies on the viability of these industrial routes are necessary;
3. Studies on activation and/or concentration of glauconitic rocks are necessary to increase the K content in the final product.

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