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Effects of biotite syenite on the nutrient levels and electrical charges in a Brazilian Savanna Ferralsol

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INTRODUCTION

The agricultural production in Brazil has increased, driven by technological advances for cultivation in the central region of the country called Cerrado (Brazilian Savanna). In this area, approximately 46 % of the soils are classified as Oxisols, i.e., highly weathered soils with low fertility (Lopes & Guilherme 2016). In order to overcome this limitation, several procedures are employed, including the massive addition of soluble fertilizers. However, the excessive use of soluble fertilizers has drawn attention, due to its environmental impacts.

ABSTRACT

In the Brazilian Savanna, most of the soils have a low natural fertility. In order to overcome this limitation, farmers use large amounts of soluble fertilizers, causing environmental impacts. In the present study, finely ground biotite syenite (0; 600; 1,200; 1,800; and 2,400 kg ha\(^{-1}\)) was evaluated as an alternative source of nutrients and electrical charges in a weathered Ferralsol from the Brazilian Savanna. Additionally, the development and nutrition of maize plants were analyzed. The values of Ca + Mg, T, t, Zn, Cu, Fe and Mn in the soil were not affected by the addition of biotite syenite, but the available P and K and the permanent charges were increased. The biotite did not increase the contents of P, Ca and Cu in the maize leaves, but it increased the contents of N and K. Increases of 103, 125 and 128 % were also detected, respectively, for plant height, stem diameter and leaf dry matter. The biotite syenite was able to increase the soil P, K and permanent charges, as well as the maize uptake of N and K, thus helping in the plant growth.

KEYWORDS: Plant nutrients, remineralization, rock powder, alternative source of nutrients.

RESUMO

No Cerrado, a maior parte dos solos possui baixa fertilidade natural. Para superar esta limitação, os agricultores empregam grandes quantidades de fertilizantes solúveis, gerando impactos ambientais. Objetivou-se avaliar a utilização de biotita sienito finamente moída (0, 600, 1,200, 1,800 e 2.400 kg ha\(^{-1}\)) como fonte alternativa de nutrientes e cargas elétricas em Latossolo intemperizado do Cerrado. Adicionalmente, o desenvolvimento e nutrição de plantas de milho foram avaliados. Os valores de Ca + Mg, T, t, Zn, Cu, Fe e Mn no solo não foram afetados pela adição da biotita sienito, mas houve aumento no P e K disponíveis e nas cargas permanentes. A biotita não elevou os teores de P, Ca e Cu nas folhas do milho, mas aumentou os teores de N e K. Aumentos de 103, 125 e 128 % também foram detectados, respectivamente, na altura da planta, diâmetro do caule e matéria seca das folhas. A biotita sienito foi capaz de aumentar o P, K e as cargas permanentes do solo, bem como a absorção de N e K pelo milho, auxiliando no crescimento da planta.

PALAVRAS-CHAVE: Nutrientes vegetais, remineralização, pó de rocha, fonte alternativa de nutrientes.

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supply the domestic demand for fertilizers, especially in the case of fertilizers containing potassium (K). The main form of K used in agriculture is muriate of potash, and 96 % of the amount used in Brazil are imported (AMA Brasil 2020), thus increasing production costs and making the use of synthetic fertilizers unfeasible for many undercapitalized and family-based farmers.

Certain silicate rocks existing in Brazil have been considered as an alternative source of K, especially at the regional level (Manning 2018), but their use is not widespread yet. This may be attributed to the fact that the highest K content of these rocks is approximately 15 % of K$_2$O, being very low, when compared to that of muriate of potash.

The estimated distribution of K in continental crust minerals indicates that 75 % of it is in K-feldspar and 21 % in mica, leaving 4 % of the total K$_2$O for other silicates which are common in alkaline rocks, such as nepheline, leucite and kalsilite. This estimate considers an average proportion in the continental crust of 2.4 % of K$_2$O, 12 % of K-feldspar and 5 % of micas (Ronov & Yaroshevsky 1969). Muriate of potash deposits reach only 0.01 % of the total K in the continental crust (Ciceri et al. 2017).

Biotite syenite is a rock that brings together two major K-bearing silicate rock minerals: biotite and K-feldspar. Biotite is a faster-release K silicate source than K-feldspar (Mohammed et al. 2014), and this may be strategic in the management of these sources, in relation to the residual effect. Rocks with these minerals are common in many countries and can be used as a model for studying the behavior of silicate minerals with potential to provide K for agriculture (Basak et al. 2020).

There are numerous studies on the use of thermal, chemical or hydrothermal processes to increase the K availability from rocks (Ciceri et al. 2017, Tanvar & Dhawan 2019). These transformation processes, however, present limited applicability, due to their high-energy consumption. Brazil is a pioneer in the regulation of silicate agrominerals, and farmers have already started using them.

Finely ground rocks may also increase the number of electrical charges on soils. Crushed basalt increased the cation exchange capacity in Oxisols from Australia (Gillman 1980), while Anda et al. (2015) reported an increase of net negative charges after the finely ground basalt application. In most of the studies on soil electrical charges, the analytical methods are based on indirect measures of exchangeable cations, such as Mg, Ca and K. Anderson & Sposito (1991) proposed a method based on Cs adsorption, which allows the combined and direct quantification of permanent and variable charges. This method presents a high detectability, making it possible to quantify small amounts of permanent charges, even in weathered soils (Fontes et al. 2001).

In the present study, finely ground biotite syenite was evaluated as an alternative source of nutrient and electrical charges in a weathered Ferralsol from the Brazilian Savanna. Additionally, the maize development and nutrition were analyzed. Maize was chosen because of its economic importance for the Brazilian Savanna, one of the world’s largest grain producers (FAO 2019).

**MATERIAL AND METHODS**

The experiments were conducted between May and December of 2015. Samples from the B horizon (1.2 m deep) of a Latossolo Vermelho-Amarelo Distrófico (Embrapa 2013), or Ferralsol (IUSS Working Group WRB 2014), were collected in Planaltina, in the Federal District of Brazil (15°36'25"S, 47°44'49"W and 1,014 m of altitude). The samples were air-dried, passed through a 2-mm sieve and were chemically analyzed according to Embrapa (2009). Briefly, the pH was measured in a soil:water suspension, using a pH-meter equipped with a glass electrode. Ca + Mg and Al were extracted with a 1 mol L$^{-1}$ of KCl solution, while H + Al was extracted using a 0.5 mol L$^{-1}$ of Ca(C$_2$H$_3$O$_2$)$_2$ solution at pH 7. The available P, K, Zn, Cu, Fe and Mn were obtained after extraction with a 0.0125 mol L$^{-1}$ of H$_2$SO$_4$ + 0.05 mol L$^{-1}$ of HCl solution. The Ca + Mg content was determined by titration with 0.0125 mol L$^{-1}$ of EDTA; Al and H + Al with 0.025 mol L$^{-1}$ of NaOH; K was determined by flame photometry and P by spectrophotometry at 660 nm, using the molybdenum blue method. Zn, Cu, Fe and Mn were determined by microwave-induced plasma atomic emission spectrometry. The total organic carbon was determined by the modified Walkley-Black method (Yeomans & Bremner 1988).

The biotite syenite rock was obtained in Guanambi, Bahia state, Brazil. Previous X-ray diffraction and petrographic analyzes showed that the material is composed of microcline (77 %),...
biotite (11 %) and clinopyroxene (11 %), and it was classified as biotite syenite (Rosa et al. 2000). The rocks were crushed in a ball mill and passed through a 0.100-mm mesh sieve to obtain a finely ground biotite syenite. The samples were digested using HNO₃ and HCl at 180 °C, for 4 h, and the resulting solution was diluted with ultrapure water. The contents of Si, Al, Fe, Ti, Ca, Mg, Ba, Mn, K and Na were assessed by microwave-induced plasma atomic emission spectrometry (Embrapa 2009). The P content was obtained after digestion with H₂SO₄, followed by dilution with ultrapure water and quantification by the molybdenum blue method (Murphy & Riley 1962).

The finely ground biotite syenite was incorporated into soil samples in doses equivalent to 0; 600; 1,200; 1,800; and 2,400 kg ha⁻¹, on a dry-weight basis. These doses correspond to 0, 75, 150, 225 and 300 kg ha⁻¹ of K₂O, which represent the range of K fertilizations most recommended for maize in the Brazilian Savanna region. The soil samples were transferred to 14 dm³ capacity pots and kept in a greenhouse for 60 days, with the soil moisture maintained at 60-70 % of the water holding capacity. After 60 days, samples were collected and analyzed, using the procedure aforementioned.

After 60 days of soil incubation, permanent and variable negative charges were determined by the Cs method (Anderson & Sposito 1991). Briefly, the soil samples (2.0 g) were mixed with a 0.01 mol L⁻¹ CsCl solution, to prepare a Cs-saturated sorbent, that was then heated to 65 °C, for 48 h, to enhance the Cs sorption on permanent negative charges by inner-sphere complexation. The displacement of Cs from the variable charge was achieved using 0.01 mol L⁻¹ of a LiCl solution, while, for the Cs from the permanent charges, it was carried out using 1.0 mol L⁻¹ of an NH₄OAc solution. Both the LiCl and NH₄OAc extracts were analyzed for Cs at 697 nm.

After the 60-day period, maize was cultivated in the pots previously incubated with soil and biotite syenite. The maize seeds were aseptically placed on germination paper and germinated in a dark chamber, at 20 °C. The seedlings were transferred to the pots and grown for 45 days, in a greenhouse, receiving an equivalent dose of 100 kg ha⁻¹ of N [(NH₄)₂SO₄]. After 45 days, the plant height and stem diameter were determined using a digital caliper. The plants were collected and heat-treated at 65 °C, to constant weight, and ground into a fine powder (2-mm mesh screen) using a knife mill. The N content was determined using a micro-Kjeldahl procedure after digestion with H₂SO₄·H₂O₂. For P, the same digestion with H₂SO₄·H₂O₂ was used and the concentration was determined by the molybdenum blue method. The levels of Ca, Mg, K, Zn, Fe, Cu, Ni and Mn were obtained after digestion with HNO₃·HCl and determination using microwave-induced plasma atomic emission spectrometry (Embrapa 2009).

The treatments were organized in a completely randomized design, with three replicates. The data were submitted to regression analysis, and the significance of the models was evaluated by the F-test. The significance of the model (p-value) and the coefficient of determination (R²) were adopted as criteria to choose the best regression model. To calculate the optimum biotite syenite dose in the quadratic model, the rule to find the first derivative was applied. The x-value was defined when the derivative was equal to zero and the maximum point was obtained. Univariate observations were made after the Anova by means comparison tests (Tukey; p < 0.05).

RESULTS AND DISCUSSION

The biotite syenite consisted mainly of SiO₂ (539.0 mg g⁻¹), Al₂O₃ (179.0 mg g⁻¹), K₂O (125.0 mg g⁻¹) and Fe₂O₃ (70.0 mg g⁻¹). CaO, MgO and Na₂O were also present at considerable concentrations (34.0, 19.0 and 18.0 mg g⁻¹, respectively), while TiO₂, P₂O₅ and BaO were present at intermediate concentrations (7.6, 6.7 and 6.0 mg g⁻¹, respectively). The contents of ZnO and MnO were low, about 2.1 and 0.6 mg g⁻¹, respectively.

Soil acidity characteristics, i.e., pH, H + Al and Al, remained unchanged at 60 days after the addition, regardless of the applied dose (Table 1). Also, the total organic carbon, Ca + Mg, Zn, Cu, Fe, Mn, T and t remained unchanged. These results disagree with previous studies that also investigated the effect of finely ground rocks on soil acidity. For example, Silva et al. (2012) observed that silicate rocks added to an Oxisol increased the soil pH after an addition period of 100 days, especially when ultramafic rock was used. Melo et al. (2012) described the neutralization of acidic soils after the application of 50 t ha⁻¹ of finely ground basalt rock, but it is important to observe that the authors used a dose significantly higher than the one used in this study.
than the maximum amount applied in this study (2,400 kg ha$^{-1}$). The application of lower doses and the relatively low concentration of CaO and MgO in the biotite syenite may explain, at least in part, why the soil acidity remained unchanged in their study. It is also important to mention that many reactions on the surface layer varied with time and it is thus possible that the 60-day period was not enough to change the soil acidity indicators.

The biotite syenite added significant amounts of P to the soil, and a quadratic model (Figure 1a) was observed as the applied doses were increased. A theoretical optimum biotite syenite dose of 1,562 kg ha$^{-1}$ was estimated by equating the derivative of the regression equation to zero. With the dose of 1,800 kg ha$^{-1}$, discounting the value of the control, approximately 4.4 mg dm$^{-3}$ of P were added to the soil, showing that the biotite syenite can contribute to increase the levels of this nutrient in the soil. The observed quadratic responses were similar to the ones reported by Silva et al. (2012), who evaluated the efficiency of brechia, biotite-schist, ultramafic rock and phlogopite schist in supplying P to an Oxisol. As previously mentioned, the soil acidity did not change with the addition of biotite syenite rock powder. Thus, it is possible that the soil acidity may have contributed

| Dose kg ha$^{-1}$ | pH      | TOC g kg$^{-1}$ | P mg dm$^{-3}$ | K | Ca + Mg | H + Al cmol dm$^{-3}$ | Al cmol dm$^{-3}$ |
|-------------------|---------|----------------|--------------|---|---------|-----------------------|---------------|
| 0                 | 5.56 ± 0.08 | 17.18 ± 0.21   | 0.96 ± 0.13 e* | 32.35 ± 1.23 d | 0.87 ± 0.02 | 2.60 ± 0.09 | 0.06 ± 0.00 |
| 600               | 5.77 ± 0.25 | 16.84 ± 0.57   | 3.18 ± 0.36 b | 39.29 ± 4.95 d | 0.70 ± 0.01 | 2.60 ± 0.09 | 0.11 ± 0.08 |
| 1,200             | 5.57 ± 0.15 | 16.74 ± 2.70   | 3.86 ± 0.33 b | 45.85 ± 7.55 e | 0.73 ± 0.15 | 2.35 ± 0.02 | 0.05 ± 0.02 |
| 1,800             | 5.30 ± 0.03 | 16.93 ± 0.44   | 5.39 ± 0.43 a | 62.25 ± 2.49 b | 0.92 ± 0.10 | 2.35 ± 0.21 | 0.05 ± 0.00 |
| 2,400             | 5.51 ± 0.40 | 16.55 ± 0.15   | 3.13 ± 0.30 b | 70.93 ± 7.55 a | 0.85 ± 0.05 | 2.22 ± 0.19 | 0.05 ± 0.01 |

* Means followed by different letters in the column differ significantly by the Tukey test ($p < 0.05$). The absence of letters in the same column represents no difference by the Tukey test. Values refer to the mean ± standard deviation. TOC: total organic carbon; T: cation exchange capacity [(Ca + Mg + K + (H + Al)]; t: effective cation exchange capacity (Ca + Mg + K + Al).

Table 1. Chemical characteristics of a Ferralsol from the Brazilian Savanna after incubation with doses of finely ground biotite syenite.
to release P from the rock powder. A possible explanation for the slight decrease in the P levels at the highest dose of the biotite syenite might be the immobilization of P in iron oxihydroxides caused by the weathering of biotite syenite, resulting in iron oxidation (Kraal et al. 2019). Additionally, the 1,200; 1,800; and 2,400 kg ha\(^{-1}\) doses presented a slightly lower Al content than that of the 600 kg ha\(^{-1}\) dose. However, due to the very complex dynamic of P in highly weathered soils, there is no doubt that further investigations would be necessary to elucidate the effect of higher doses of biotite syenite on available P.

The available K increased in a linear behavior after the addition of biotite syenite (Figure 1b), resulting in increases of 17.2 mg dm\(^{-3}\) for each ton of biotite syenite. At the highest dose, when compared to the control, an increase of 136 % in the available K was observed. The biotite syenite presented a large amount of K\(_2\)O (125.0 mg g\(^{-1}\)), favoring the increase of K in the soil. Gudbrandsson et al. (2011) observed that some groups of minerals found in basalt present dissolution rates that constantly decrease as the pH increases, and are slightly inhibited by a higher concentration of bivalent metallic cation. In this study, no increases in the pH and in the Ca and Mg levels were observed after the addition of rock powder, what may have favored the dissolution of K-containing minerals in the applied biotite syenite.

Variable charges were predominant in the Ferralsol (Table 2) and represented approximately 70 % of the total soil charges (from 53 to 79 %). Regardless of the biotite syenite dose, the amount of variable charges was not changed. Inversely, the permanent charges increased by 152 %, when compared to the highest dose of applied biotite syenite and to the control. The permanent charges in the control (5.6 mmol kg\(^{-1}\)) are close to those obtained in previous studies using the same Cs method for soil charges quantification in tropical soils. For example, Busato et al. (2012) reported 5.0 mmol kg\(^{-1}\) of permanent charges in a typical Oxisol, while Weber et al. (2005) obtained a value of 5.8 mmol kg\(^{-1}\) in an Oxisol. Higher levels of permanent charges may be a consequence of the rock milling, in which minerals in the biotite syenite structure may undergo physical deformation, increasing their specific surface (Quirk et al. 2012). X-ray diffraction and petrographic analyzes showed that the employed biotite syenite is formed by alkaline feldspar microcline (77 %), biotite (11 %) and clinopyroxene (11 %) (Rosa et al. 2000), which have high surface-charge density, reaching up to 2.7 µeq m\(^{-2}\) (Sollins et al. 1988). Therefore, the fine grinding of the biotite syenite served to increase the participation of these less weathered minerals, what resulted in increases in the permanent charges.

The predominance of positive charges in highly weathered tropical soils may reduce the efficiency of fertilizers. In tropical soils, the most active cation exchange sites are attributed to the organic matter content, making these soils dependent of carbon accumulation (Marchi et al. 2015). Contrary to expectations, in the present study, no changes in the cation exchange capacity (t or T), obtained by an indirect method, were observed (Table 1). In all likelihood, permanent charge sites, evidenced by the Cs method, were less available to ions present in the soil solution, since tropical soils present an ionic strength of about 0.05 mol L\(^{-1}\) (Alleoni et al. 2009).

The maize grew higher in biotite syenite-amended soil, when compared to the control, for all the doses considered in this study (Table 3). In the highest dose, the plant height was 103 % higher than the control. The stem diameter and dry leaf matter also increased with the highest dose (123 and 128 %, respectively, when compared to the control), while the dry root matter was not affected. The P, Ca and Cu levels in the maize leaves were unaffected by the addition of biotite syenite (Table 4). However, the concentrations of N, K, Mg, Fe, Zn and Mn were changed, with different responses, according to the biotite syenite dose. For instance, at the highest dose, the content of K increased by 6.7 %, when compared to the control, while N was increased by 11.3 %.

### Table 2. Variable and permanent charges analyzed by the cesium adsorption method in a Ferralsol from the Brazilian Savanna after addition of finely ground biotite syenite.

| Dose kg ha\(^{-1}\) | Variable charges mmol kg\(^{-1}\) of soil | Permanent charges mmol kg\(^{-1}\) of soil |
|---------------------|------------------------------------------|------------------------------------------|
| 0                   | 20.50 ± 1.90 aA* (79 %)                  | 05.60 ± 0.30 bB (21 %)                  |
| 600                 | 21.00 ± 1.20 aA (79 %)                   | 05.50 ± 0.20 bB (21 %)                  |
| 1,200               | 15.50 ± 1.40 aA (79 %)                   | 06.80 ± 0.30 bB (30 %)                  |
| 1,800               | 16.90 ± 1.30 aA (70 %)                   | 07.20 ± 0.30 bB (30 %)                  |
| 2,400               | 16.10 ± 1.00 aA (53 %)                   | 14.10 ± 0.40 aA (47 %)                  |

* Means followed by the same lower-case letter within a column and capital letter on the row do not differ statistically by the Tukey test (p < 0.05). Values refer to the mean ± standard deviation. Numbers in parentheses refer to the percentage, in relation to the total charges.
the other hand, the same dose reduced the content of Mg (32.9 %), Fe (23.2 %) and Zn (52.9 %), when compared to the control (Table 4), possibly due to an antagonism between K and other cations, such as Mg, Fe and Zn, as reported by Fageria (2001). While the concentration of K in the soil at the highest dose of syenite was approximately 100 % higher than for the control, the contents of Ca + Mg, Fe and Zn in the soil were not affected by the addition of syenite (Table 1).

In general, N, K and P are the nutrients most required by crops, and a reduced supply of these elements may negatively affect yield. N affects the dry matter production, due to its role in the photosynthetic process and in the development of the leaf area (Schlüter et al. 2012). Therefore, a low availability of N is the main cause of declining crop yield and quality. K has an essential role in maintaining the internal positive turgor potential of plants, as well as in the photosynthetic activity and tolerance to water deficit (Robin et al. 1989, Behboudian & Anderson 1990). Thus, the higher contents of N and K in the leaves are in accordance with the increased values of plant height, stem diameter and dry leaf matter (Table 3).

The increase in the P availability in the soil may have reduced the availability of Zn through the formation of P-Zn precipitates, leading to a lower absorption of Zn by plants (Table 4). The lack of response in the uptake of P at the highest dose of the biotite syenite supports this hypothesis. In addition, in the case of Zn, it is possible that an inverse relationship between plant growth and mineral contents in leaves has occurred, resulting in a dilution effect that occurs when the dry weight accumulation increases faster than the mineral nutrient accumulation (Riedell 2010).

Despite the absence of statistical difference for the P content in the leaves, higher doses of biotite syenite increased the P content by 7 %. This is promising, since P is a critical element in weathered soils of tropical regions.

| Dose kg ha⁻¹ | Plant height cm | Stem diameter mm | Root dry matter g | Leaf dry matter g |
|--------------|----------------|----------------|------------------|------------------|
| 0            | 11.33 ± 1.76 c* | 15.67 ± 4.04 ba | 0.15 ± 0.03      | 0.25 ± 0.03 b    |
| 600          | 16.86 ± 1.26 ba | 25.00 ± 5.00 ab | 0.33 ± 0.02      | 0.35 ± 0.05 b    |
| 1,200        | 19.00 ± 1.32 ab | 28.33 ± 4.72 ab | 0.31 ± 0.03      | 0.38 ± 0.04 b    |
| 1,800        | 17.50 ± 1.32 ab | 25.33 ± 2.16 ab | 0.11 ± 0.02      | 0.32 ± 0.03 b    |
| 2,400        | 23.02 ± 2.18 aa | 35.02 ± 4.66 aa | 0.19 ± 0.05      | 0.57 ± 0.05 a    |

* Means followed by different letters in the column differ significantly by the Tukey test (p < 0.05). Values indicate the mean ± standard deviation.

Table 3. Plant height, stem diameter, root and shoot dry matter of maize plants cultivated in soil samples after incubation with doses of finely ground biotite syenite.

| Dose kg ha⁻¹ | N  g kg⁻¹ | K  g kg⁻¹ | P  g kg⁻¹ | Ca  mg kg⁻¹ | Mg  mg kg⁻¹ |
|--------------|-----------|-----------|-----------|-------------|-------------|
| 0            | 28.66 ± 0.56 b* | 30.45 ± 0.03 bc | 3.73 ± 0.10 | 9.52 ± 1.21 | 4.96 ± 0.48 a |
| 600          | 30.51 ± 0.36 a  | 30.72 ± 0.54 b  | 3.82 ± 0.15 | 9.89 ± 1.36 | 4.00 ± 0.00 b |
| 1,200        | 31.74 ± 0.31 a  | 29.13 ± 0.86 ca | 3.81 ± 0.40 | 7.50 ± 1.85 | 3.32 ± 0.18 c |
| 1,800        | 31.54 ± 0.45 a  | 29.33 ± 0.62 bc | 4.01 ± 0.12 | 9.34 ± 1.62 | 3.30 ± 0.12 c |
| 2,400        | 31.91 ± 0.95 a  | 32.45 ± 0.39 aa | 4.00 ± 0.13 | 10.13 ± 1.90 | 3.33 ± 0.22 c |

* Means followed by different letters in the column differ significantly by the Tukey test (p < 0.05). Values indicate the mean ± standard deviation.

Table 4. Macro and micronutrients in maize leaves cultivated in a Ferralsol from the Brazilian Savanna after the addition of finely ground biotite syenite.
CONCLUSIONS

1. The values of pH, H + Al, total organic carbon, Ca + Mg, T, t, Zn, Cu, Fe and Mn in the soil were not affected by the addition of biotite syenite;
2. The available P and K in the soil and the net permanent charge increased, even at low doses of biotite syenite;
3. The biotite syenite did not increase the contents of P, Ca and Cu in the maize leaves, but it increased the contents of N (11.3 %) and K (6.7 %);
4. Increases in the uptake of N and K by maize, as well as in plant height, stem diameter and dry leaf matter, were detected.

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