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## Growth of *Urochloa* grass in an oxisol treated with powdered silicate materials

### Crescimento de capim *Urochloa* em um latossolo tratado com materiais silicáticos em pó

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### ABSTRACT

An experiment was carried out in pots with a soil mixed with six powdered silicate materials originated from mining (kyanite, biotite schist, biotite syenite, basalt, vermiculite, or bentonite). These sources were mixed in 4.4 kg of soil (Anionic Acrustox) at the rates of 0 (control), or 440 g of ground silicate materials, in pots where *Urochloa decumbens* cultivar Basilisk was grown. The grass growth followed the relative total dry mass (%): 100 > 93.8 > 82.7 > 71.4 > 54.4 > 9.4 > 6.8 for biotite schist, bentonite, vermiculite, basalt, syenite, kyanite, and soil (control), respectively. Soil pH (6.5) ultimately decreased Mn availability to plants. Although some silicate materials increased Mn availability to plants, increasing treated plants growth, it was not possible to evaluate if plants were able to acquire more P when treated with silicate materials. Concentrations of Fe and P extracted from the soil and from shoots were highly correlated with dry mass. The reactivity of these materials in soils increasing soil pH may be an important parameter for ranking agrominerals, as the availability of nutrients was correlated to the amount of carbonate used to increase pH of the treated soil to 6.5.

**Keywords:** *Urochloa decumbens* cv. “Basilisk”; Biotite schist; Basalt; Biotite syenite.

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## RESUMO

Um experimento foi realizado em vasos com um solo misturado com seis materiais silicatados em pó originados da mineração (cianita, biotita xisto, biotita sienito, basalto, vermiculita ou bentonita). Essas fontes foram misturadas em 4,4 kg de solo nas taxas de 0 (controle) ou 440 g, em vasos onde *Urochloa decumbens* cultivar Basilisk foi cultivada. O crescimento do capim seguiu a ordem, segundo sua massa seca total relativa (%):  $100 > 93,8 > 82,7 > 71,4 > 54,4 > 9,4 > 6,8$  para biotita xisto, bentonita, vermiculita, basalto, biotita sienito, cianita e solo, respectivamente. O pH do solo (6,5) diminuiu a disponibilidade de Mn para as plantas. Embora alguns materiais silicatados tenham aumentado a disponibilidade de Mn para as plantas, aumentando o crescimento das plantas tratadas, não foi possível avaliar se as plantas adquiriram mais P quando tratadas com materiais silicatados. As concentrações de Fe e P extraídas do solo e da parte aérea das plantas foram correlacionadas com a massa seca. A reatividade desses materiais, aumentando o pH do solo, pode ser um parâmetro importante para classificar agrominerais, pois a disponibilidade de nutrientes foi correlacionada à quantidade de carbonato usada para aumentar o pH do solo tratado.

**Palavras-chave:** *Urochloa decumbens* cv. “Basilisk”; Biotita xisto; Basalto; Biotita sienito

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## INTRODUCTION

Organic acids released by roots react directly with soil components, forming complexes with iron and aluminum, and dissolving P from the surfaces of oxides (Barrow et al., 2018; Khan et al., 2019). The acidification of rhizosphere by P-deficient plants is well documented in many studies (Li et al., 2015; Shen et al., 2018). Root exudates, therefore, are regulated by the plant nutritional status. A Fe-deficient plant drives mineral weathering processes in the rhizosphere, also through exudation of organic ligands (Gattullo et al., 2016). On the other hand, the acquisition of poorly bioavailable Fe in the soil takes place by two strategies used by plants (graminaceous and non-graminaceous). In short, graminaceous plants use a chelation-based mechanism (strategy-II), releasing mugineic acid family phytosiderophores in the soil to chelate ferric iron (Kobayashi et al., 2019).

Phytosiderophores are secreted into the rhizosphere where they chelate and help to solubilize Fe(III). The Fe(III)–phytosiderophore complex is then taken up into root cells through the action of Yellow Stripe1 (YS1) proteins (Walker; Connolly, 2008). Tropical grasses also exude organic acids to acquire P from hematite and goethite, by inducing P desorption, and by complexing metal cations (Almeida et al., 2020).

In soil experiments in pots with powdered silicate material is preponderant that pH is stabilized, as some powdered rocks may increase pH substantially, over or sub estimating benefits that are being observed. The use of increasing rates of remineralizers

favor increases in soil pH (Alovisi et al., 2023). Unless the experiment objectives are linked to the pH control in soils, results under analysis will be impaired by differences in the soil pH.

At the same time, soil pH should be adequately adjusted to limit micronutrient deficiencies. Manganese fertilization is not recommended for crops grown in soils presenting values of Mehlich-1 extractable concentrations above  $5.0 \text{ mg dm}^{-3} \text{ Mn}$  (Galvão 1999, Lopes 1998), or DTPA extractable concentrations above  $12 \text{ mg dm}^{-3} \text{ Mn}$  (Lopes 1998). Despite the abundance of Mn in the soil, exceeding, by far,  $5.0 \text{ mg dm}^{-3} \text{ Mn}$ , its availability to plants is highly constrained when soils  $\text{pH}_{\text{water}}$  is above 6.5. Though, chemical methods used to estimate the Mn availability to plants, such as DTPA-TEA at pH 7.3, HCl  $0.1 \text{ mol L}^{-1}$ , Mehlich-1 and 3, might not reveal the real Mn availability status for plants.

Soils treated with powdered rocks could decrease P adsorption in (hydr)oxides of Fe and Al, increasing adsorption in other phases produced by rock powders, increasing P availability to plants. Thus, powder rocks could improve P uptake, and increase crops yield. The aim of this work was to evaluate the growth of *Urochloa decumbens* cv. “Basilisk” in greenhouse, using powder rocks and a soluble source of P in a Cerrado soil.

## MATERIAL AND METHODS

### Soil characterization

A composite soil sample (Anionic Acrustox, fine-loamy, mixed, isothermic (USDA, 2015) Latossolo Amarelo ácrico típico, textura média, A moderado, gibbsítico, aniônico, fase Cerrado tropical subcaducifólio relevo plano (Embrapa, 2013), located at S  $15^{\circ} 11' 20''$  W  $47^{\circ} 74' 27''$ , in Planaltina, DF, Brazil, from 0 to 20 cm depth was collected. The soil analysis presented the following characteristics (Embrapa, 2017): sand =  $498.2 \text{ g kg}^{-1}$ ; silt =  $107.5 \text{ g kg}^{-1}$ ; clay =  $394.3 \text{ g kg}^{-1}$ ; organic carbon = 1.9%; organic matter =  $32 \text{ g kg}^{-1}$ ; density =  $1.24 \text{ g cm}^{-3}$ ;  $\text{pH}_{\text{water}} = 5.8$ ;  $\text{pH}_{\text{CaCl}_2} = 5.2$ ; extracted by Mehlich-1 (HCl  $0.05 \text{ mol L}^{-1} + \text{H}_2\text{SO}_4 0.0125 \text{ mol L}^{-1}$ ), 1:10 soil:solution ratio (Mehlich, 1953): P =  $2.6 \text{ mg dm}^{-3}$  and K =  $55.6 \text{ mg dm}^{-3}$ ; S extracted by  $\text{Ca}(\text{H}_2\text{PO}_4)_2 0.01 \text{ mol L}^{-1}$ , 1:10 soil:solution ratio (Singh et al., 1995),  $1.5 \text{ mg dm}^{-3}$ ; by KCl ( $1 \text{ mol L}^{-1}$ ), 1:10 soil:solution ratio (Embrapa, 2017): Ca =  $2.1 \text{ cmol}_c \text{ dm}^{-3}$ , Mg =  $1.2 \text{ cmol}_c \text{ dm}^{-3}$  and Al <  $0.1 \text{ cmol}_c \text{ dm}^{-3}$ ; H+Al extracted by  $\text{Ca}(\text{OAc})_2 0.5 \text{ mol L}^{-1}$  at pH 7.0 (Embrapa, 2017) =

3.1 cmol<sub>c</sub> dm<sup>-3</sup>; CEC at pH 7.0 = 6.5 cmol<sub>c</sub> dm<sup>-3</sup>; B (hot water) = 0.3 mg dm<sup>-3</sup>; Si extracted by CaCl<sub>2</sub> 0.01 mol L<sup>-1</sup> (Korndörfer et al., 2004): 2.0 mg kg<sup>-1</sup>; micronutrients extracted by Mehlich-1: Fe = 61.9 mg dm<sup>-3</sup>, Cu = 0.3 mg dm<sup>-3</sup>, and Zn = 1.5 mg dm<sup>-3</sup>; Mn = 7.0 mg dm<sup>-3</sup>.

### Crushed rocks characterization

Crushed rock samples of kyanite, biotite schist, biotite syenite, basalt, vermiculite, bentonite were selected for the study. Kyanite was obtained from the Northwest of the Quadrilátero Ferrífero, province in the state of Minas Gerais (Evangelista and Delgado, 2007; Luz et al., 2008); Biotite schist was collected from residue piles in quarries located at the state of Goiás (Navarro et al., 2013) and biotite syenite was collected at the Southeastern of the state of Bahia (Cruz et al., 2016); basalt was collected in Formação Serra Geral, Araguari, state of Minas Gerais; vermiculite was obtained in São Luís dos Montes Belos, state of Goiás; bentonite from Quatro Barras, state of Paraná (Cruz et al., 2016). All crushed rocks were sieved to <2 mm fraction. Samples were air dried and homogenized using the cone-and-quartering reduction method. This procedure was repeated several times to ensure complete homogenization of material.

These samples were wet sieved to obtain the percentual of four size fractions: < 53 µm, 53 – 300 µm, 300 – 1000 µm and > 1000 µm (Table 1). The main chemical elements of crushed rocks were analyzed using the multi-acid solution method, where: 500 mg of sample was digested in 10:15:10:5 mL of HCl:HNO<sub>3</sub>:HF:HClO<sub>4</sub>, respectively, determined by ICP-OES (SGS Geosol Laboratórios Ltda). Major elements were determined by wavelength dispersive X-ray fluorescence (XRF) spectroscopy, on fused glass discs, 40 mm-diameter, prepared from 0.8 g of sample powder mixed with 4.5 g lithium tetraborate flux and fused in Pt-5% Au crucibles at 1120°C (SGS Geosol Laboratórios Ltda). The loss on ignition was determined after heating samples overnight at 105°C to remove water. The weight loss was measured after calcination of samples at 1000°C for approximately 2 hours. The results are presented in the Table 2.

The mineralogical composition of crushed rock fractions < 53 µm was analyzed by X-ray diffraction analysis (XRD) using a PANalytical Empyrean (PW3050/60) diffractometer, using the powder method in the range of 5° < 2θ < 75°. CoKα radiation (40 kV; 40 mA) was applied, and the 2θ scanning speed was set at 0.02° s<sup>-1</sup>. Data was acquired using the software X'Pert Data Collector 4.0 and the data were treated on X'Pert

HighScore 3.0 (PANalytical). Minerals were identified by comparing the obtained diffractogram with the ICDD-PDF (International Center for Diffraction Data) database.

**Table 1.** Particle size distribution of the rock powders used in the experiment.

Sample	Particle size distribution ( $\mu\text{m}$ )			
	< 53	53 - 300	300 - 1000	> 1000 - 2000
	-----%-----			
Kyanite	6.9	42.4	49.3	1.4
Biotite schist	21.1	17.4	2.4	59.1
Biotite syenite	11.9	23.2	29.6	35.3
Basalt	10.7	12.4	26.5	50.4
Vermiculite	38.1	39.0	22.5	0.5
Bentonite	88.0	11.6	0.2	0.2

The mineralogical composition in each rock size fraction was estimated by the stoichiometric method, also known as rational calculation, which is based on the relationship of the experimental chemical composition with the chemical formulas of the minerals, establishing logical considerations based on the qualitative (XRD) and quantitative (XRF) analytical data. The ModAn (Paktunk, 1998) software was used to perform the rational calculation.

### Soil treatments

Experimental design was completely at random factorial scheme, using two factors: phosphate presence or absence and crushed rock materials, in four replicates. Treatments were prepared as follows: control soil ( $4.4 \text{ kg pot}^{-1}$ ) and soils mixed with rock materials in a rate of  $4.4 \text{ kg}$  of soil and  $440 \text{ g}$  of rock material, totaling  $4.84 \text{ kg}$  of treated soil, were incubated in open pots for 30 days, irrigated until field capacity one time a week. After the mentioned period, the soil was allowed to dry for 7 days. To estimate the amount of  $\text{CaCO}_3$  needed to adjust pot soils to the pH 6.5, ten grams of soil of each pot ( $80 \text{ g}$  in total for each treatment) was obtained to perform neutralization curves in laboratory. Increasing rates (0, 3, 5, 10, and 20 mg) of calcium carbonate were added to 5 g of the collected soil and mixed in 12.5 mL of water, with 3 replicates. These soil samples were incubated for 72hs before pH measurements were taken. After the neutralization curves were determined, calculated rates of  $\text{CaCO}_3$  were mixed to the soil and rock materials in order to equalize soil pH at 6.5, and placed into the pots. Treated soils in pots were incubated again for 30 days, while irrigated until field capacity one time

a week. Values of pH were measured to ensure the pH 6.5 was attained. Thereafter, treated soils in pots were allowed to dry for 7 days (Table 2).

**Table 2.** Incubation-pH of soil, and soil mixed with 10% of crushed rock materials.<sup>#</sup>

Soil-mixed material	Equation	R <sup>2</sup>	CaCO <sub>3</sub> <sup>§</sup> g kg <sup>-1</sup> soil	Final pH <sup>¶</sup>	
				Water	CaCl <sub>2</sub>
Soil (control)	$y = -0.0023x^2 + 0.159x + 5.6222$	0.99	1.21		
Kyanite	$y = -0.0051x^2 + 0.2079x + 5.7833$	0.98	0.76		
Biotite schist	$y = -0.0046x^2 + 0.1852x + 6.1975$	0.98	0.34		
Syenite	$y = -0.0059x^2 + 0.2029x + 6.2672$	0.99	0.24	6.26	5.65
Basalt	$y = -0.0046x^2 + 0.1851x + 6.087$	0.99	0.47	s=0.16*	s=0.15
Vermiculite	$y = -0.005x^2 + 0.1821x + 6.27$	0.99	0.26		
Bentonite	$y = -0.0053x^2 + 0.2172x + 5.5306$	0.99	1.02		

<sup>#</sup>Soil and soil-mixed materials to the soil were treated with levels of CaCO<sub>3</sub> and incubated for 30 days prior to pH measurement (n = 3). <sup>§</sup>CaCO<sub>3</sub> added per pot to increase soil pH in water to 6.5. <sup>¶</sup>After plants were harvested, it was not found significant differences in the final pH in water and in CaCl<sub>2</sub> of control or treated soils. \* s = standard deviation (n = 56).

Soluble fertilizers in the powder form were added, mixing the soil and fertilizers with the use of plastic bags. Half of the pots received 67.7 mg kg<sup>-1</sup> N as NH<sub>4</sub>SO<sub>4</sub>, and 40 mg kg<sup>-1</sup> K as KCl, characterizing the treatments which did not receive phosphorus. The other half received 67.7 mg kg<sup>-1</sup> N, 150 mg kg<sup>-1</sup> P as NH<sub>4</sub>PO<sub>4</sub>, and 40 mg kg<sup>-1</sup> K as KCl, characterizing those treatments that received phosphorus. The treated soils were placed into containers 33 cm length x 66 cm height x 2.8 cm width, with 8 holes of 4 mm diameter at the bottom. The containers were translucent for root growth observation. The containers were kept in the dark during the growth of plants. Soils were, then, incubated for 45 days, being kept irrigated to field capacity one time a week. Ten seeds of *Urochloa decumbens* cv. “Basilisk” were sown on October 10th, 2017. One week later, plants in excess were cut off leaving only one plant per pot. Plants were grown for 50 days since seeds were sown.

Container positions were randomly switched 1 time per week. During the growth period the temperature inside the glasshouse varied from 18.3 to 46.4°C; Relative humidity varied from 31 to 92.6%. At the end of the experiment, plants were collected, separated into roots and shoots in paper bags and placed to dry in oven at 60 °C until constant weight. Dry matter weight was taken. Plants were chemically analyzed. After

the plants were harvested, samples of the treated soils in pots were collected and placed to dry for posterior analysis.

### Soil and plant chemical procedures and statistical analysis of the data

After harvesting, treated soils collected from pots were analyzed for the following characteristics:  $pH_{\text{water}}$  and  $pH_{\text{CaCl}_2}$ ; P, K Na, S, Ca, Mg, Al, and Si; micronutrients Cu, Fe, Mn, and Zn;  $H^+Al$ ; CEC at pH 7.0 was calculated as the sum of K, Ca, Mg, and  $H^+Al$ .

Chemical analyses of the plants were performed only for those treatments which received P, as the amount of dry mass produced in those which did not receive P was scant and did not allow analysis of plants using conventional methods. Plants grown in pots where P was supplied were analyzed for major (K, Ca, Mg, Al, Fe) and minor elements (B, Cu Mn e Zn), which were extracted by using  $HNO_3:HClO_4$  (3:1) in a digestion block (Bataglia et al., 1983), and determined by inductively coupled plasma - optical emission spectrometry (ICP-OES). The results are presented in the Table 3

Tests for normality and equal variance (Shapiro; Wilk, 1965) were used prior to the analysis of variance. The Tukey test was applied for multiple comparisons of groups in normally distributed data. Not normally distributed data was evaluated by Scheirer Ray Hare (Real Statistics for Excel; Zaiontz, C., 2018) and Kruskal-Wallis (Sigma Plot, version 12, Systat Software Inc., San Jose, CA, USA) analysis; The Tukey test was used for all pairwise comparison of the groups mean ranks.

Pearson's correlation analysis to relate the data from soil and plant chemical analysis was performed using the Sigma Plot, version 12.0, software. Due to the high mineralogical and chemical differences among rocks, the correlation of the complete set of treatments with crushed rocks did not was nonsignificant, therefore, smaller groups of treatments were correlated in a partial correlation (Marchi et al., 2020). Therefore, Cook's distance influential point (Di) analysis, to find influential points in a set of predictor variables, was performed using the R software (Team, 2017). Observations from selected treatments were kept aside from the main group based on its influence on the analysis. Results were correlated using all n observations for the results, except the ith observation. The intent was to find correlation of interest for each group of crushed rocks. The used guidelines were: if Di was greater than 0.5, then the ith data point was further investigated as it might be influential; and if Di was greater than 1, then the ith data point was quite likely to be influential.

## RESULTS AND DISCUSSION

### Rock powders characterization

The chemical composition (Tables 3 and 4) and the XRD from powder rocks samples (Figure 1) were used to determine their mineralogical composition (Table 5). Kyanite, a nesosilicate, Al-rich rock, contains mostly the mineral kyanite ( $\text{Al}_2\text{SiO}_5$ ; Table 4). The mineral kyanite consists of chains of edge-sharing Al–O octahedrons interlinked by Si–O tetrahedrons and Al–O tetrahedrons, presenting high stability in pH values from 5.5 – 7.5, at 0 – 22°C (Zhang et al., 2019). The presence of dolomite in this rock increased soil pH, decreasing the amount of  $\text{CaCO}_3$  needed to adjust soil pH to 6.5 when compared to the control.

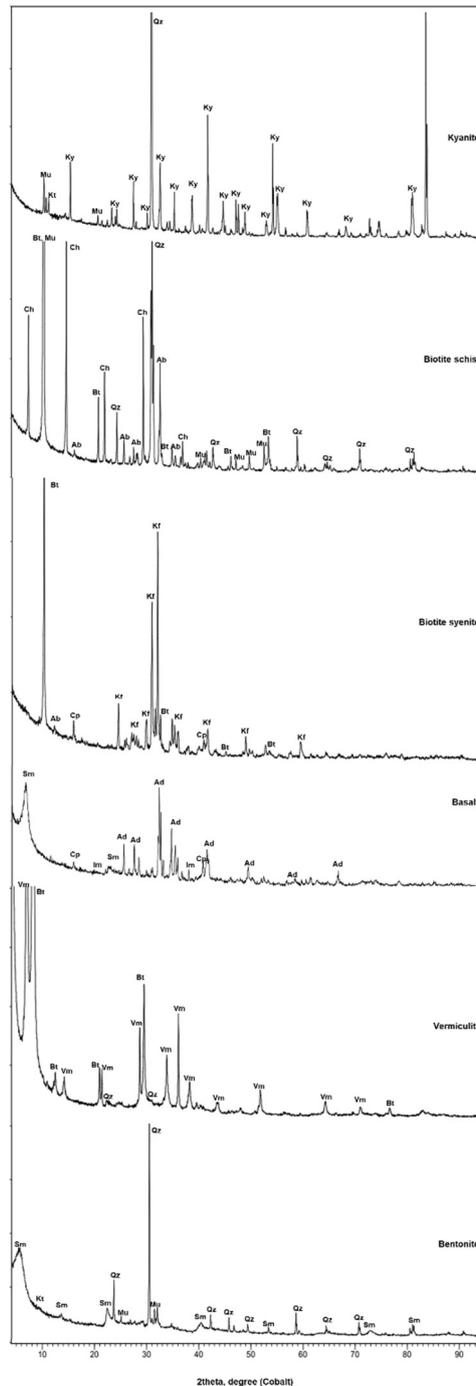
Minerals containing appreciable concentrations of  $\text{Fe}^{2+}$  and  $\text{Mn}^{2+}$  were found in the biotite schist, biotite syenite, and basalt, such as chlorite, which have a general chemical composition  $(\text{A,B})_{4-6}(\text{Si,Al})_4\text{O}_{10}(\text{OH,O})_8$  (where: A and B in the formula represent ions, which might include:  $\text{Fe}^{2+}$ ,  $\text{Fe}^{3+}$ ,  $\text{Mg}^{2+}$ ,  $\text{Mn}^{2+}$ ,  $\text{Ni}^{2+}$ ,  $\text{Zn}^{2+}$ ,  $\text{Al}^{3+}$ ,  $\text{Li}^+$ , or  $\text{Ti}^{4+}$ ; Malmström et al., 1995), found in biotite schist, and clinopyroxene, which is a mafic silicate of a subgroup of the pyroxenes, presenting a general chemical composition of  $\text{ABSi}_2\text{O}_6$  (where: A =  $\text{Ca}^{2+}$ ,  $\text{Na}^+$ , and  $\text{Li}^+$ ; and B =  $\text{Mg}^{2+}$ ,  $\text{Fe}^{2+}$ ,  $\text{Fe}^{3+}$ , and  $\text{Al}^{3+}$ ; Malmström et al., 1995), found in biotite syenite and basalt.

The mineral biotite was found in the composition of both biotite schist and biotite syenite, as well as in vermiculite (Table 5). Although in lower proportions than the other minerals, biotite is the main mineral prone to release nutrients, such as K, Ca, Mg, Fe and Mn, as well as other elements, such as Al and Na, from biotite schist and biotite syenite, by weathering, during plant growth (Krahl, 2020). Even though, variations in the chemical composition of biotite, lead to differences in its weathering rates and, therefore, in the availability of elements to plants (Basak, 2018; Krahl, 2020).

Basalt presents high content of  $\text{Fe}_2\text{O}_3$  that comes from ilmenite [ $\text{FeTiO}_3$ ]. The dissolution of small inclusions of ilmenite and diopside in basalt releases Fe (Krahl et al., 2020). Strains of Fe-oxidizing bacteria are able to grow using the FeII derived from ilmenite of basaltic rocks (Navarrete et al., 2012). This effect may be strongly influenced by the presence of organic acids released from the rhizosphere of plants (Dontsova et al.,

2014). Other important nutrient source for plant growth in basalt is andesine  $[(Ca,Na)(Al,Si)_4O_8]$ , a plagioclase feldspar (Krahl et al., 2020).

**Figure 1.** X-ray diffraction patterns of basalt, biotite syenite, biotite schist, vermiculite, bentonite, and kyanite. Keys: Cp (clinopyroxene); Ad (andesine); Bt (biotite); Mu (muscovite); Ch (chlorite); Ab (albite); Kf (K-feldspar); Ky (kyanite); Qz (quartz); Im (ilmenite); Vm (vermiculite); Sm (esmectite), Kt (kaolinite).



Source: Authors' elaboration (2023).

**Table 3.** Major elements in rock materials analyzed by x-ray fluorescence.<sup>1</sup>

Samples	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	TiO <sub>2</sub>	P <sub>2</sub> O <sub>5</sub>	Na <sub>2</sub> O	K <sub>2</sub> O	MnO	BaO	Cr <sub>2</sub> O <sub>3</sub>	LOI <sup>1</sup>	Total
	weight %													
Kyanite	39.2	54.2	3.4	0.1	0.3	0.2	0.04	<0.1	0.3	0.02	<0.01	0.43	1.18	99.37
Biotite syenite	55.9	16.0	8.2	2.0	1.7	1.5	0.24	1.2	11.3	0.11	0.57	<0,01	0.49	99.15
Biotite schist	62.6	16.8	7.8	1.4	3.2	0.9	0.20	1.9	3.2	0.12	0.07	0.02	2.17	100.38
Basalt	49.0	12.6	15.4	8.7	5.3	3.2	0.43	2.1	0.9	0.20	0.06	<0.01	1.32	99.21
Vermiculite	38.2	11.9	12.0	0.2	20.0	4.2	0.13	<0.1	3.6	0.11	0.12	0.12	7.78	98.36
Bentonite	65.7	15.7	5.4	0.4	1.5	0.8	0.05	0.6	2.1	0.05	0.09	<0.01	6.09	98.48

<sup>1</sup> LOI = Loss on ignition. Source: Authors' elaboration (2023)

**Table 4.** Elements of interest analyzed in multiacid extracts of rock materials by inductively coupled plasma atomic emission spectroscopy.

Rock materials	Ni	Cu	Zn	Sr	Co	V	La	Li	Pb	Zr	Sc	Y
	mg kg <sup>-1</sup>											
Kyanite	11	24	111	20	<8	354	<20	4	<8	23	12	6
Biotite Schist	51	49	133	140	18	103	24	67	10	58	13	22
Biotite syenite	23	31	89	420	13	81	20	12	<8	33	12	10
Basalt	49	208	139	491	37	221	26	13	<8	123	25	25
Vermiculite	320	30	121	33	63	66	86	20	<8	142	19	<3
Bentonite	10	25	98	102	<8	41	38	42	16	139	5	26

Source: Authors' elaboration (2023)

**Table 5.** Mineralogical composition of rock and mineral materials used in the experiment.

Samples	Minerals (weight %)																	
	Cp	Am	Ad	Bt	Ch	Do	Mu	Ab	Kf	Ap	Ky	Qz	Mt	Im	Hm	Vm	Sm	Kt
Kyanite						<1		-	-	-	>80	5-10		<1	1-5			
Biotite syenite <sup>4</sup>	10-20	1-5		10-20			-	5-10	40-60	<1								
Biotite schist <sup>4</sup>	1-5			10-20	10-20		10-20	20-40	-	<1		20-40						
Basalt <sup>3,4</sup>	20-40	-	40-60	-	-	-	-	-	-	<1	-	-	-	10-20				1-5
Vermiculite				10-20			-	-	-	<1		1-5				>80		
Bentonite			1-5				1-5	-	-	-		10-20	1-5				>80	1-5

<sup>1</sup> MoDan (Paktunk, 1998); <sup>2</sup> Keys: Cp (clinopyroxene); Am (amphibole); Ad (andesine); Bt (biotite); Mu (muscovite); Ch (chlorite); Do (dolomite); Ab (albite); Kf (K-feldspar); Ap (apatite); Ky (kyanite); Qz (quartz); Mt (magnetite); Im (ilmenite); Hm (hematite); Vm (vermiculite); Sm (esmectite), Kt (kaolinite); <sup>4</sup> Source: Krahl (2020).

The mineral vermiculite, found in vermiculite, presenting the empirical chemical composition  $Mg_{1.8}Fe^{2+}_{0.9}Al_{4.3}SiO_{10}(OH)_2 \cdot 4(H_2O)$  may be, along with the mineral biotite, an important source of Fe and Mg for plants.

The bentonite obtained for the study presented a very high amount of smectite. Although the bentonite was rich in  $SiO_2$  and  $AlO_2$ , and presented lower concentrations of plant nutrients when compared to the other agrominerals (Tables 3 and 4), nutrients, such as Fe, Mn, Ca, Mg and K present in the exchange sites from the interlayer space of clay minerals may be released for plant uptake (Cuadros, 2017).

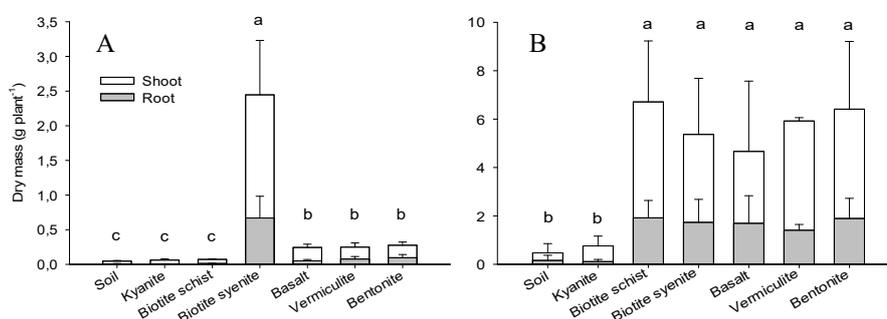
### **Urochloa grass dry matter production**

The increase (difference) in total dry mass yield between treatments which were fertilized with P, as opposed to the not fertilized (Figure 2), was 8.6, 8.0, 7.1, 6.1, 4.7, 0.8, and 0.6 g per plant for biotite schist, bentonite, vermiculite, basalt, kyanite, untreated soil (control), and biotite syenite respectively, corresponding to an increase of 11891, 2903, 2846, 2509, 1307, 1241, and 190%, respectively. Normalizing data, within the treatments treated with P, to the maximum yield, obtained in the biotite schist treatment, rendered the following relative total dry mass (%):  $100 > 93.8 > 82.7 > 71.4 > 54.4 > 9.4 > 6.8$  for biotite schist, bentonite, vermiculite, basalt, syenite, kyanite, and soil (control), respectively. The average dry mass yield of plants not fertilized with P was 0.48 g per plant, while for those fertilized with P was 5.6 g; representing an average increase of 11.6 times.

### **Influence of rock powders on the soil attributes and plant nutrition**

The reactivity of some crushed rock species in soils, along with the release/availability of nutrients, impacting in plant growth, could, in some extent, be related to the amount of  $CaCO_3$  added to adjust the treated soil to pH to 6.5. In fact, a partial Pearson's correlation, assisted by Di, of the amount of added  $CaCO_3$  ( $mg\ kg^{-1}$ ), and the mean dry mass ( $g\ pot^{-1}$ ) produced by plants in all treatments, except bentonite, in which  $150\ mg\ kg^{-1}$  P was supplied ( $n = 6$ ; Figure 2) showed correlation coefficients of -0,91 ( $P=0.01$ ), -0.93 ( $P<0.01$ ), and -0,88 ( $P=0.02$ ) for shoots, roots and the total dry mass yields, respectively, in plants fertilized with phosphorus.

**Figure 2.** Dry matter produced by *Urochloa decumbens* cv. “Basilisk” in treatments with rock powder materials and 0 mg kg<sup>-1</sup> P (A) or 150 mg kg<sup>-1</sup> P (B) provided. Error bars = Standard Deviation; Tukey Pairwise Grouping, indicated by letters a, b.



Source: Authors' elaboration (2023).

The reactivity of these powdered materials, increasing soil pH, may be an important parameter for selecting agrominerals. Bentonite, due to the low amount of both CaO and MgO, and probably by the nature of the silicate materials it contains, which presented lower reactivity in the soil than the other treatments, was excluded from the correlation. A partial correlation, excluding only bentonite and kyanite (n=5), returned better correlation coefficients: -0.96 (P=0.01), -0.92 (P=0.02), and -0.96 (P<0.01) for shoots, roots, and the total dry mass yields, respectively. Kyanite presents low amounts of both CaO and MgO, increasing soil pH, but did not result effectively in dry mass yield; therefore, kyanite, along with bentonite, decreased the correlation coefficients when the whole group was considered. When phosphorus was not supplied, considering the partial correlation methodology or not, the correlation with dry mass returned poor coefficients.

Some soil chemical attributes were excluded from discussion as statistical analysis showed that there were no significant differences among treatments, such as soil's organic matter content (4.59%; s = 0.41), extractable S (3.08 mg kg<sup>-1</sup>; s = 0.07) and Ca (3.55 mg kg<sup>-1</sup>; s = 0.12), as well as final pH<sub>water</sub> (6.28; s = 0.15); final pH<sub>CaCl2</sub> (5.66; s = 0.14). Other nutrients from soil, in the light of the used chemical extractants, such as P, K, Mg, Si, Fe, and Mn (Figure 3), as well as CEC at pH 7.0 (Figure 4), showed significant differences among treatments.

Vermiculite, as well as bentonite, present high density of permanent charges, therefore, soils and crops may benefit directly from the use of these materials, by significantly increasing its CEC (Figure 4). Syenite also presented a higher CEC than the control, indicating that, even in a short time (50 days), biotite minerals went through

bioweathering, increasing CEC. In syenite samples Khral et al (2020), in a successive growth of maize in pure powder rock, found that the oxidation of Fe(II) in minerals of biotite covered the rock particles, forming a stable weathering environment, preventing further K release and the increase in CEC. The authors found a constant CEC for syenite during the whole experiment ( $CEC = 3.73 \pm 0.93 \text{ cmol}_c \text{ kg}^{-1}$ ). This powdered rock, in the present experiment, mixed in soil, apparently did not present such stability which hindered weathering. Probably part of the released Fe(II) during biotite weathering was oxidized and deposited in soil particle surfaces as well, instead of just in the rock particle surfaces.

Biotite schist did not show increases in CEC as compared to the control at the end of the experiment (50 days after sowing, Figure 4). Meanwhile, Khral et al (2020) in a successive growth of maize in pure powder rock experiment, found that in pure biotite schist, the CEC on the fraction  $< 53 \mu\text{m}$  and the forming rate of hydrobiotite (a precursor of vermiculite) were positively correlated, with CEC increasing along the experiment (from 1.48 to  $8.41 \text{ cmol}_c \text{ kg}^{-1}$ ).

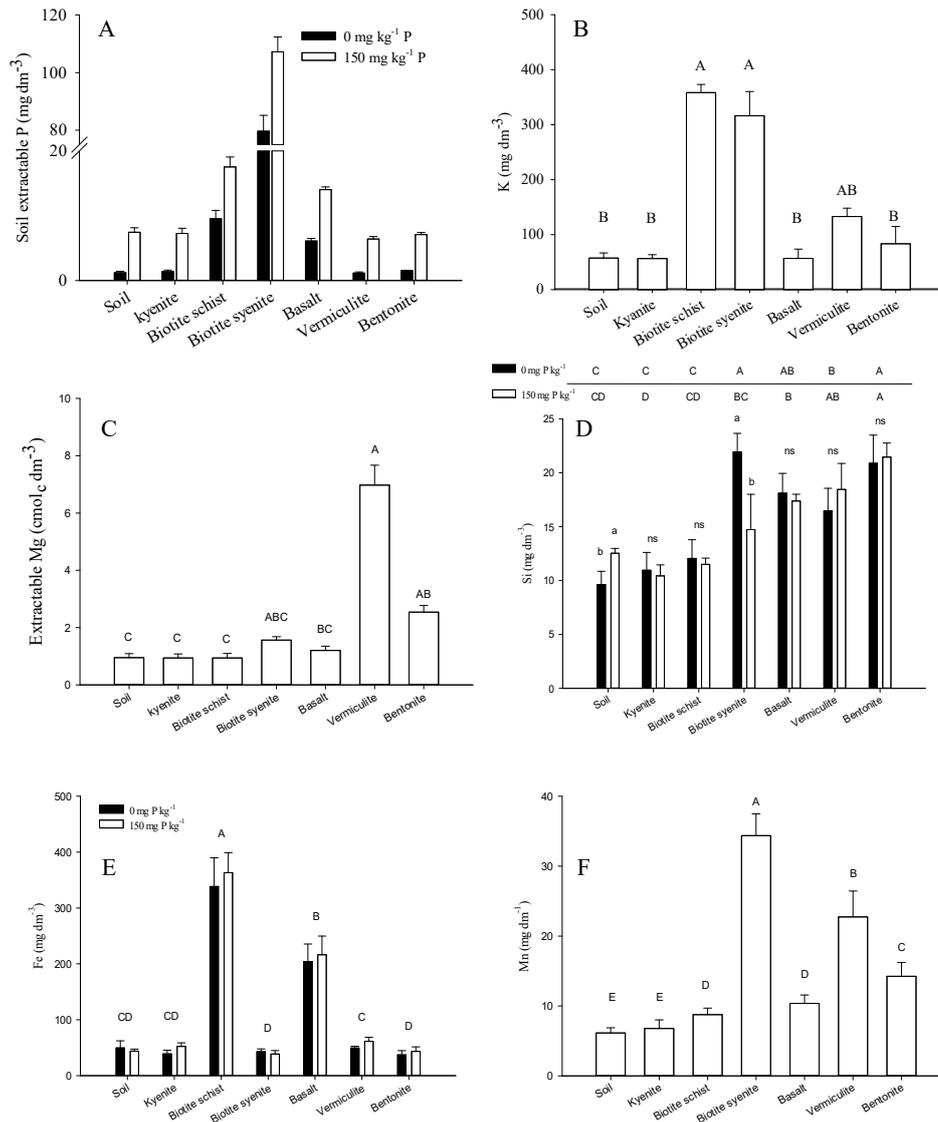
### Correlation analysis

The concentration of nutrients in plant tissues, such as P, Fe, Mn, K, Ca, Mg, S, Na and Zn, along with elements, such as Al (Figure 5), also showed statistical differences among treatments. The whole set of data from these nutrient extractions was confronted by using the Pearson's correlation, and a partial correlation assisted by Di (Table 6).

When plants were not fertilized with P, dry matter yield (DM) was strongly correlated with P extracted chemically from soil ( $r = 0.89$ ; Table 6). Biotite syenite showed the highest DM yield among the other treatments probably because part of the P was released from syenite minerals, promoting plant growth (Figure 2, left). When the syenite treatment was excluded from the group, the partial correlation between DM and P dropped to non-significant, and a high correlation between P and Fe extracted from soil, emerged (Table 6). However, Fe and P extracted by Mehlich-1 were not correlated to the plant uptake of P (Figure 5; Table 6). The extractant accessed P from rocks (mainly biotite schist and basalt; Figure 3) which was not proportional to that available to plants during its 50 days of growth (Figure 2A).

**Figure 3.** Extractable nutrients from soil treated with different crushed rocks and the growth of *Urochloa decumbens* for 50 days. Error bars = Standard Deviation; Tukey

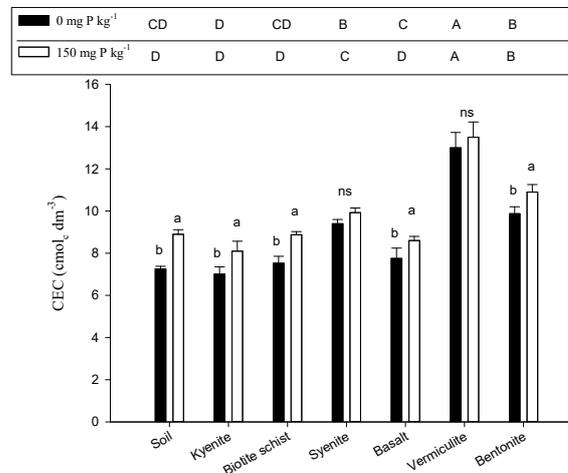
Pairwise Grouping on Mean Ranks for P, K, and Mg indicated that the 0 and 150 mg P kg<sup>-1</sup> rates among rock treatments are significantly different; lowercase within 0 mg P kg<sup>-1</sup> and uppercase within 150 mg P kg<sup>-1</sup>. For Mg, 0 and 150 mg P kg<sup>-1</sup> rates, even though different among P fertilization treatments, rendered equal letters when the test was applied within treatments. Tukey Pairwise Grouping for Si, Fe, and Mn indicated by a, b is significantly different.



Source: Authors' elaboration (2023).

**Figure 4.** Cation exchange capacity of soil at pH 7.0, measured by summation of exchangeable cations, after mixing different powder rock treatments followed by the

growth of *Urochloa decumbens*, for 50 days (indicated by lowercase letters for ln-transformed comparisons within materials, and uppercase, within rates of P). Error bars = Standard Deviation; Tukey Pairwise Grouping indicated by a, b.



Source: Authors' elaboration (2023).

The same pattern occurred in those treatments where P fertilization was provided (Table 6; Figure 2B). The P and Mn extracted from syenite treated soil were correlated (Table 6). However, in the partial correlation, without syenite, Fe and P extracted from the soil ( $r = 0.97$ ; Table 6) were highly correlated. Contents of P and Fe in shoots were correlated ( $r=0.80$ ; Table 6), and both P and Fe content in shoots correlated with the DM ( $r=0.9$  and  $0.72$ , respectively; Table 6) indicating that P and Fe absorption was probably promoted by the chelation-based mechanism (strategy-II; Kobayashi et al., 2019).

The P extracted from the soil by plants and by Mehlich-1, in all treatments (Figures 3 and 5), due to adsorption properties of the soil, was much lower than the phosphorus added ( $150 \text{ mg kg}^{-1}$ ). The concentrations of P extracted are consonant with those shown by Broggi et al. (2010), where  $\sim 90\%$  of P was strongly adsorbed in soil particles, and little was recovered by chemical extractants. The concentration of Fe and P in shoots from control and soil treated with kyanite were above the range defined as adequate (from  $50$  to  $250 \text{ mg kg}^{-1}$  of dry weight), by Werner et al. (1997). Therefore, P was supplied adequately for all treatments, and was accessible to plant uptake.

Iron in shoots correlated with DM, P, K, and S in shoots (Table 6). However, although biotite schist and basalt provided the highest amount of Fe chemically extracted from soil, by Mehlich-1, accounting for  $338.5$  ( $s = 51.4$ ), and  $204.1$  ( $s = 31.6$ )  $\text{mg dm}^{-3}$  Fe, respectively (Figure 3), it was not translated in highest amount of Fe accumulated in shoots (Figure 5). Both bentonite and vermiculite presented lower content of Fe extracted

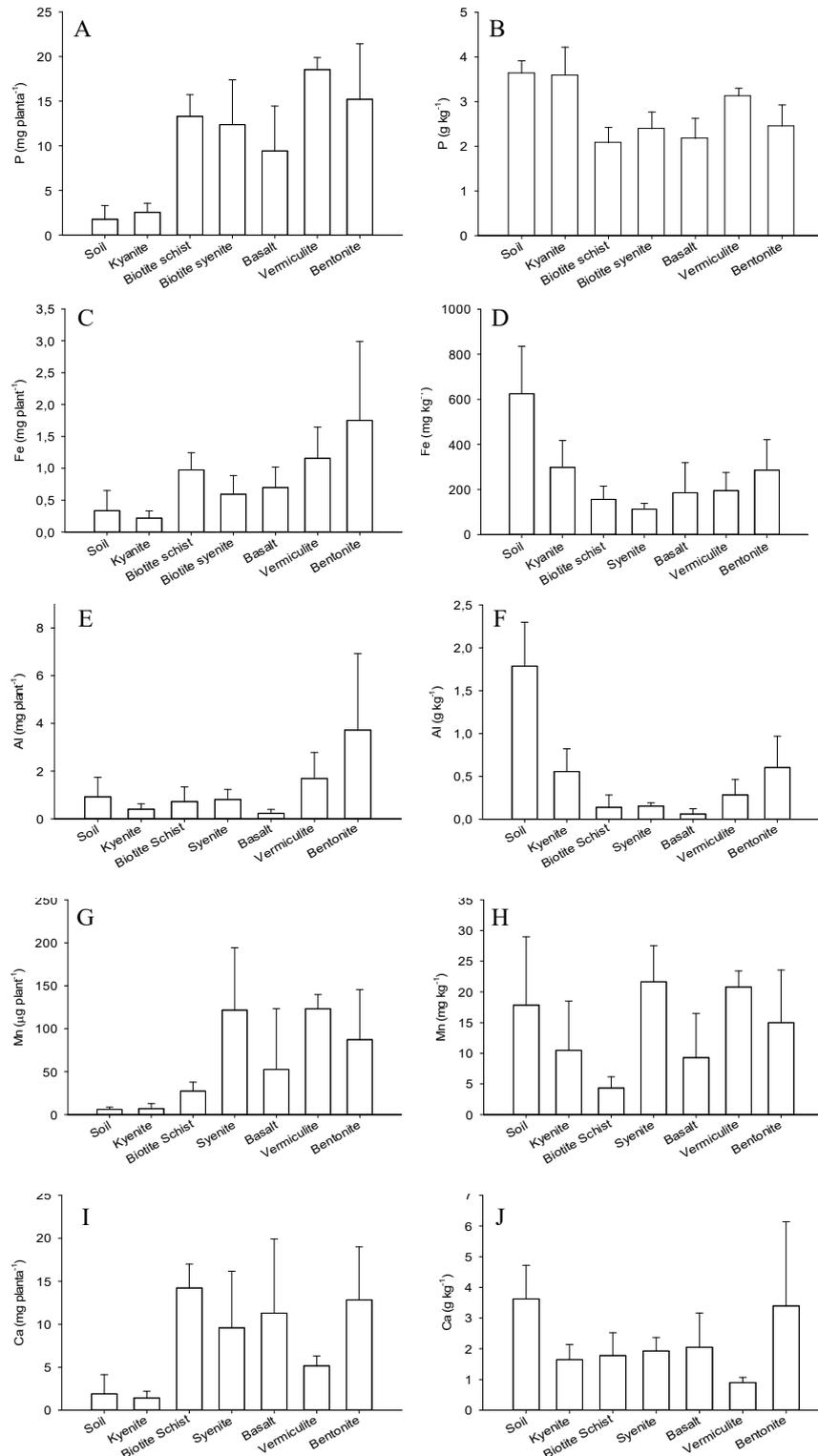
chemically from soil (47.8;  $s = 4.0$ , and 37.2;  $s = 7.5$ )  $\mu\text{g dm}^{-3}$  Fe, respectively) than biotite schist and basalt but reached higher content of Fe in shoots. Bentonite presented lower values of chemically extractable Fe in soil even than the untreated soil (49.7  $\text{mg dm}^{-3}$ ;  $s = 12.7$ ; Figure 4) but provided highest amount accumulated in shoots (1.14  $\text{mg plant}^{-1}$ ;  $s = 295.4$ ).

The DM correlation with Fe, P, K and S in shoots ( $r = 0.76, 0.90, 0.93$  and  $0.95$ , respectively; Table 6) imply, probably, that K and S covariate with P and Fe, and DM, promoting higher plant growth. Therefore, K and S were available in adequate concentration in all treatments, as they did not correlate with P or Fe extracted from soil. Potassium concentrations in shoots were above the adequate range (from 12 to 25  $\text{mg kg}^{-1}$ ; Werner et al., 1997) in all treatments.

Manganese concentration in the shoots of the fertilized plants from the treatments which received soluble P (Figure 5) were below the range defined as adequate (from 40 to 250  $\text{mg kg}^{-1}$ ; Werner et al., 1997). There is no indicative that there was a lack of any of the evaluated nutrients, except for Mn, in shoots in the treatments which received soluble P, in the lower yield treatments (soil and kyanite).

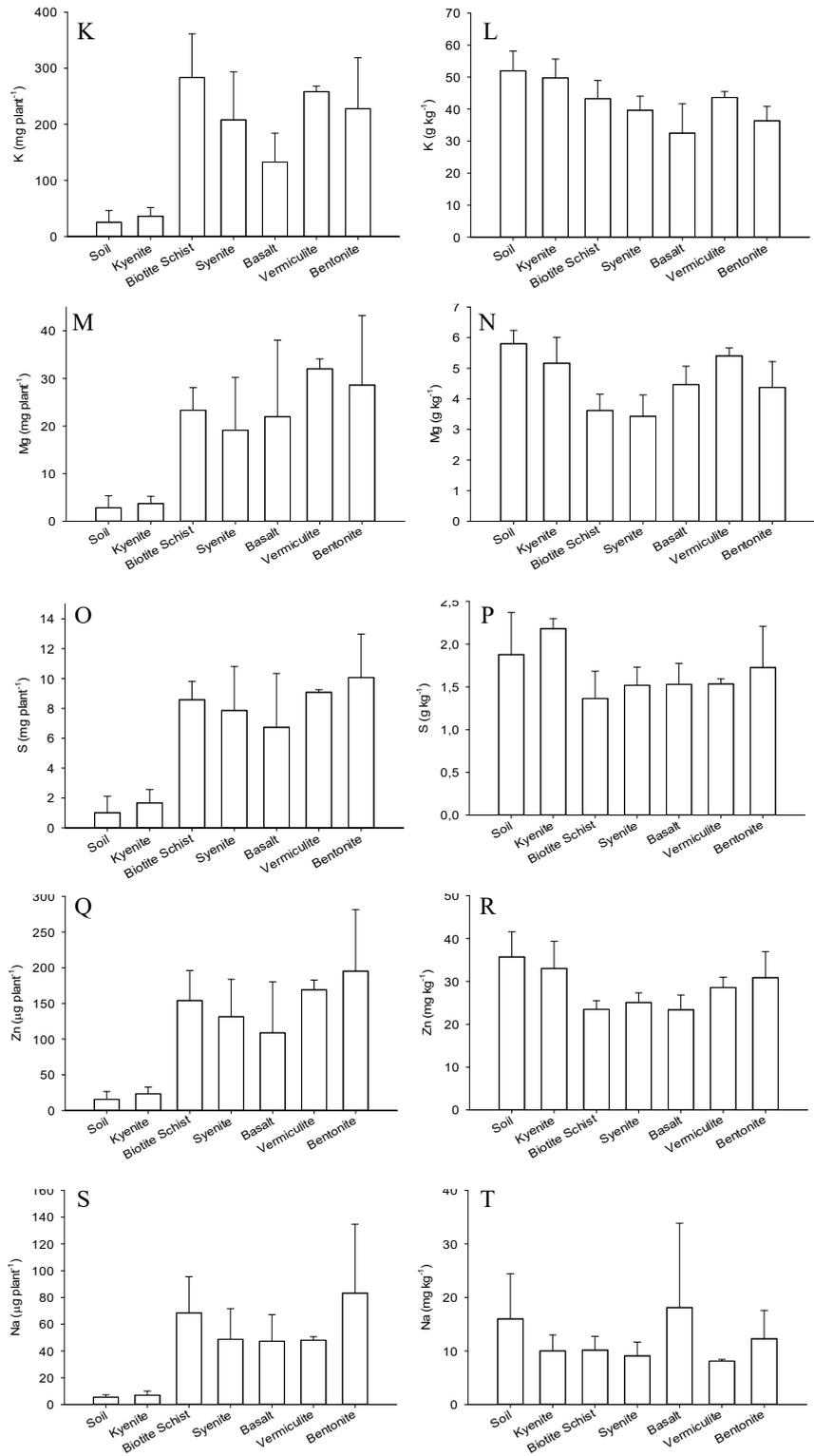
Values of Mehlich-1 extractable concentrations above 5.0  $\text{mg dm}^{-3}$  Mn in soils are considered high (van Raij, 1997). Values of Mn extracted from soil was above that limit (7.0  $\text{mg dm}^{-3}$ ), and after the growth of plants, in all treatments, were all above 5  $\text{mg dm}^{-3}$  Mn (Figure 5). Sienite treated soils were as high a 36.9  $\text{mg dm}^{-3}$ . Probably the soil pH was a limiting factor for Mn absorption by the grass in this soil, although the pH 6.5 is considered adequate for the growth of crops. Soils pH above 6.5 (Galrão, 1999) may present a decrease in the exchangeable  $\text{Mn}^{2+}$  and readily reducible Mn levels and, consequently, a reduction in the  $\text{Mn}^{2+}$  uptake by plants. Although the Mn content in shoots was lower than the adequate, no visual symptoms of deficiency in leaves were noticed during the experiment. Low manganese availability can be a serious limiting factor for plant growth, causing interveinal chlorosis, and low crop yield (Alejandro et al., 2020).

**Figure 5.** *Urochloa decumbens* offtake of elements from soil and soil mixed with powder rock materials produced with 150  $\text{mg kg}^{-1}$  P for 50 days in shoots and roots. Error bars = Standard Deviation.



Source: Authors' elaboration (2023).

**Figure 5.** *Urochloa decumbens* uptake of elements from soil and soil mixed with powder rock materials produced with 150 mg kg<sup>-1</sup> P for 50 days in shoots and roots. Error bars = Standard Deviation. (continuation).



Source: Authors' elaboration (2023).

**Table 6.** Pearson’s correlation analysis (within treatment groups) of grams of plant dry mass (DM), mg kg<sup>-1</sup> of soil P, Fe and Mn (Soil-P, Soil-Fe and Soil-Mn, respectively), and mg plant<sup>-1</sup> of P, Fe, Mn, K and S in shoots (Shoots-P, Shoots-Fe, Shoots-Mn, Shoots-K, Shoots-S, respectively) of *Urochloa decumbens* cv. “Basilisk”.

	No P fertilization				With P fertilization										
	----- Soil -----				----- Soil -----				----- Shoots -----						
	P	Fe	Mn	Si	P	Fe	Mn	Si	P	Fe	Mn	K	S	Al	
	Complete set of treatments (n = 56)				Complete set of treatments (n = 56)										
DM	<b>0.89**</b>	-0.23	<b>0.80**</b>	0.52**	0.21	0.38*	0.36	0.38*	<b>0.90**</b>	<b>0.72**</b>	0.40*	<b>0.93**</b>	<b>0.95**</b>	<b>-0.66**</b>	
Soil-P		-0.13	<b>0.85**</b>	0.53**		-0.16	<b>0.77**</b>	-0.08	0.15	-0.09	0.36	0.2	0.19	-0.29	
Soil-Fe			-0.28	-0.16			-0.36	-0.25	0.16	0.25	0.48*	0.38	0.26	-0.41*	
Soil-Mn				<b>0.65**</b>				0.34	0.44*	0.22	0.10	0.41	0.44	0.29	
Soil-Si									0.51**	0.45*	-0.27	0.35	0.52**	-0.25	
Shoots-P										<b>0.80**</b>	0.27	<b>0.93**</b>	<b>0.94**</b>	<b>-0.64**</b>	
Shoots-Fe											0.29	<b>0.73**</b>	<b>0.79**</b>	-0.37	
Shoots-Mn												0.33	0.32	-0.33	
Shoots-K													<b>0.92**</b>	<b>-0.64**</b>	
	Partial correlation (excluding syenite; n = 48)				Partial correlation (excluding syenite; n = 48)										
DM	-0.13	-0.14	<b>0.66**</b>	<b>0.66**</b>	0.40	0.46	0.40	0.44*	<b>0.90**</b>	<b>0.76**</b>	0.35	<b>0.92**</b>	<b>0.95**</b>	<b>-0.67**</b>	
Soil-P		<b>0.98**</b>	-0.18	0.00		<b>0.97**</b>	-0.32	-0.27	0.12	0.14	<b>0.68**</b>	0.35	0.24	-0.48*	
Soil-Fe			-0.15	-0.04			-0.24	0.17	0.21	0.23	<b>0.68**</b>	0.45*	0.32	-0.52*	
Soil-Mn				0.50*				<b>0.69**</b>	<b>0.66**</b>	0.53*	-0.20	0.50*	<b>0.54**</b>	-0.23	
Soil-Si									<b>0.56**</b>	0.47*	-0.33	0.39	<b>0.59**</b>	-0.28	
Shoots-P										<b>0.82**</b>	0.20	<b>0.92**</b>	<b>0.94**</b>	<b>-0.65**</b>	
Shoots-Fe											0.32	<b>0.76**</b>	<b>0.83**</b>	<b>-0.44*</b>	
Shoots-Mn												0.26	0.27	-0.33	
Shoots-K													<b>0.91**</b>	<b>-0.65**</b>	

<sup>(1)</sup>Groups selection was assisted by Cook’s distance influential point analysis; <sup>(2)</sup> DM = plant dry mass yield, Soil-P = P extracted from soil by Mehlich 1 method (Mehlich, 1953), Soil-Fe and Soil-Mn = Fe and Mn extracted from soil by DTPA 0.005 mol L<sup>-1</sup> method (Lindsay; Norvell, 1978), Shoots-P, Shoots-Fe, Shoots-Mn, Shoots-K, Shoots-S, and Shoots-Al = P, Fe, Mn, K, S, and Al content (mg plant<sup>-1</sup>) in plants extracted by HNO<sub>3</sub>:HNO method (Bataglia et al., 1983); <sup>(3)</sup> \* p<0.05; \*\* p<0.01. Source: Authors' elaboration (2023).

The highest shoot yield in the present experiment was 10.3 g plant<sup>-1</sup> at 50 days after sowing. As a comparative, shoots of *U. decumbens* cv. Basilisk - BRA 001058 grown in 5,5 dm<sup>3</sup> pots with and Oxisol, in a greenhouse, for 56 days using 150 mg kg<sup>-1</sup> soluble P (as triple superphosphate), and soil pH 5.2, produced 29.31 g of dry mass per plant (Santos et al., 2002). Powdered rocks promoted better growth than the control (untreated soil) and kyanite in this adverse situation, but the yield was still low as compared to those obtained by Santos et al. (2002).

Copper in shoots was not significantly different among treatments which received soluble P. However, the Cu content in shoots presented a mean of 184.7 mg plant<sup>-1</sup>; s=101.9; 35.7 mg kg<sup>-1</sup> of dry weight; s=7.1), much higher than the adequate levels for Cu in grass shoots (from 4 – 12 mg kg<sup>-1</sup> of dry weight; Werner et al., 1997). Copper DTPA extractable concentrations of 0.8 mg dm<sup>-3</sup> in soils are considered high (van Raij et al., 1997). The highest content of Mehlich-1 extractable Cu in a basalt-treated soil sample was 3.5 mg kg<sup>-1</sup>. The soil treated with biotite schist was 1.0 mg kg<sup>-1</sup> Cu; the other treatments were all below 0.8 mg dm<sup>-3</sup>. The lack of Mn, therefore, caused a decreased growth of the studied grass plants, and increased concentration of, mainly, Cu and K.

Although the soil pH was high, low concentrations in shoots of Mn, and high of K and Cu, affected plant growth. However, it would be expected to affect all treatments evenly. Some powdered rocks present variable concentrations of Mn in their minerals (Table 3). Probably minerals present in biotite schist, syenite, basalt, vermiculite and bentonite were dissolved during grass growth, and released Mn which was partially taken up by plants. The released Mn was not enough, however, for normal plant growth as compared to other publication (Santos et al., 2002). Probably, part of the released Mn may have oxidized (MnOx) and become insoluble and unavailable to plant uptake. As the quantity of powdered rocks was added in a large amount (10%), it would be expected that differences in soil texture could change the outcome of Mn availability, as many rocks presented appreciable amounts of particles below 300 µm (Table 1). However, kyanite presented 49.3% of particles below 300 µm, and there was no significant influence on Mn uptake by plants or plant yield. Therefore, probably, changes in physical attributes of the soil were not a decisive factor that impacted the studied grass yield.

There is need of further research to investigate the role of amorphous Fe formed after agrominerals are bio-weathered in soils on the soluble P availability to plants. Studies must comprise the desorption of P in soils where powder rocks were added and weathered, as well as on the Fe forms in soil. Also, the effect the amorphous Fe from

powder rocks have on P availability to plants in conditions where all nutrients and pH are balanced.

## CONCLUSIONS

Perhaps by the fact that plants experienced Mn deficiency during the experiment, there was no evidence that agrominerals provide better conditions to plants uptake P than the control.

The studied soil pH must be lower than 6.5 to to grow *U. decumbens*, cv. Basilisk.

Powder rocks provide better conditions to *U. decumbens*, cv. Basilisk growth in conditions where high pH decreases Mn availability.

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