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CHEMICAL CHARACTERIZATION AND SPECIATION OF PERCOLATED WATER FROM RED OXISOL WITH APPLICATION OF PHOSPHOGYPSUM AND GYPSITE

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ABSTRACT

The objective was to chemically characterize and determine the ionic species of nutrients in percolated water from Oxisol, with application of Phosphogypsum and Gypsite. The experiment was carried out in a greenhouse, applying Phosphogypsum and Gypsum at doses from 0.0 to 4.0 t ha^{-1}. Fifty-five days after application, a 60 mm rainfall simulation was simulated and the pH, conductivity, and content of calcium, magnesium, potassium, nitrogen, phosphorus, sulfur, sodium, and ionic speciation in the percolated water were analyzed. The definition of the source and adequate dose of gypsum for each soil favors less loss of nutrients in the percolated water. This implies that production costs related to plant nutrition do not increase.

Keywords: leaching; nutrientes; gypsum

CARACTERIZACIÓN QUÍMICA Y ESPECIACIÓN DEL AGUA PERCOLADA DE OXISOL ROJO CON APLICACIÓN DE FOSFOYESO Y YESO

RESUMEN

El objetivo fue caracterizar quimicamente y determinar las especies iónicas de nutrientes en agua percolada de Oxisol, con aplicación de Fosfoyeso y Yeso. El experimento se realizó en invernadero, aplicando Fosfoyeso y Yeso en dosis de 0 a 4 t ha^{-1}. A los 55 después de la aplicación se realizo una simulación de lluvia de 60 mm y se analizaron el pH, la conductividad y el contenido de calcio, magnesio, potasio, nitrógeno, fósforo, azufre, sodio y especiación iónica en el agua percolada. La definición de la fuente y dosis adecuada de yeso para cada suelo favorece menores pérdidas de nutrientes en el agua percolada. Esto implica que los costos de producción relacionados con la nutrición vegetal no aumentan.

Palabras clave: lixiviación; nutrientes; yeso

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INTRODUCTION

The practice of plastering in Brazilian agricultural production areas is justified by the ability of gypsum to soften the effects of soil acidity in depth due to its potential to reduce aluminum saturation. In addition, due to the solubility of this input, there is a supply of calcium in the subsurface and it is indicated as an interesting product to be used in summery periods, as it provides an increase in plant root development and this results in crop tolerance to water restriction.

The use of gypsum in areas where the soil has been corrected and has adequate potassium (K) and magnesium (Mg) contents, can be a management tool for the construction of soil profiles with high fertility. This is due to the possibility of formation of soluble ionic pairs between gypsum sulphate and the monovalent ionic forms of K (K^+) and divalent Mg (Mg^{2+}) present in the soluble and/or exchangeable forms in the soil that provides their percolation along of the profile (Ramos, 2012). The success of plastering and obtaining its benefits in productive areas are directly associated with knowledge of the sources available for use, the proper recommendation of the dose to be used in a given soil and the production system.

It is known that the use of gypsum provides changes in the chemical composition of the soil solution due to the increase of Ca and sulfur (S), in the sulfate form. The presence of these nutrients provides the formation of chemical species in the soil solution that can increase their mobility and that of other nutrients in the soil profile. At first sight, this would be interesting, considering the construction of soil fertility in depth. However, Brazilian Oxisols have low natural fertility due to many chemical characteristics, highlighting the very limited cation exchange capacity due to the source material (Costa-Coutinho et al., 2019). Thus, the formation of ionic pairs of greater solubility results in high chances of nutrients being lost and or little used by plants, in addition to not improving soil fertility at depth.

Based on the above, it is important to understand the effects of gypsum better, because, if added to the soil, in amounts above those recommended, it is possible for it to carry nutrients along the profile, leaching them into the underlying layers and/or aquifers. Thus, the objective was to chemically characterize and determine the ionic species of nutrients in percolated water of Dystrophic Red Oxisol, with application of phosphogypsum and gypsite.

MATERIAL AND METHODS

The experiment was carried out in a greenhouse, in a completely randomized design with a 3x5 factorial scheme and 4 replications. The first factor was gypsum sources (Phosphogypsum; Gypsite Maranhão and Gypsite Pernambuco) and the second was doses of 0; 0.5; 1.0; 2.0 and 4.0 t ha^{-1} applied to a Dystrophic Red Oxisol (Santos et al., 2018).

The soil was collected in layers of 0-0.2 m and 0.2-0.4 m in depth, in an area already cultivated and with a history of recent liming. Due to the result of pH close to 6.0, high contents of Ca and Mg and base saturation above 60% at a depth of 0.2 m, the practice of liming was not carried out, following the recommendations of Sousa and Lobato (2004). The chemical analysis of the soil showed that from 0-0.2 m the soil had pH $CaCl_2$=5.9; Ca = 3.1 $cmol_c$ dm^{-3}; Mg = 1.1 $cmol_c$ dm^{-3}; Al = 0.0 $cmol_c$ dm^{-3}; H+Al = 3.1 mg dm^{-3}; K= 0.4 mg dm^{-3}; P= 30.6 mg dm^{-3} (Mehlich 1); organic matter (OM) = 44.0 g dm^{-3}; bases saturation (V) = 68.6%; cátion exchange capacity (CEC)= 6.7 $cmol_c$ dm^{-3} and clay= 410 g dm^{-3}. For the 0.2 to 0.4 m layer the results were: pH $CaCl_2$=4.6; Ca = 1.2 $cmol_c$ dm^{-3}; Mg = 0.4 $cmol_c$ dm^{-3}; Al = 0.1 $cmol_c$ dm^{-3}; H+Al = 4.0 mg dm^{-3}; K= 0.2 mg dm^{-3}; P= 7.3 mg dm^{-3} (Mehlich 1); OM= 34.0 g dm^{-3}; V = 30.7%; CEC= 5.8 $cmol_c$ dm^{-3} and clay= 460 g dm^{-3}.
After collection, the soil was air-dried, sieved in a 2 mm mesh sieve and packed in PVC tubes with a diameter of 50 mm and 0.5 m in height. The gypsum was applied to the soil superficially, without incorporation and the doses were based on the gypsum requirement equation (NG): NG (t ha^{-1}) = (40 x % clay)/1000 (Souza et al., 2012). The dose calculated by the equation represents the amount of gypsum recommended for this soil, with the other doses being tested because they are usually applied in grain production areas in Brazil. Agricultural gypsum were chemically and physically characterized according to normative instructions no. 35 and 24 of the Ministry of Agriculture, Livestock and Supply published in 2006 and 2007. Phosphogypsum presented 22.62% CaO, 0.63% MgO and 15.01% S. Gypsite Pernambuco presented 23.68% CaO, 0.03% MgO and 14.70% S and Maranhão presented 24.67% CaO, 0.47% MgO and 15.70% S. All gypsums had more than 50% of the particles < 0.03 mm.

The PVC pipes were irrigated daily with deionized water, maintaining 60% of the soil's field capacity. Irrigation control was estimated by soil mass in field capacity using a precision scale. At 55 days after application of the treatments, a rain of 60 mm was simulated, and the water percolated from the ground was collected in 500 mL bottles that were attached to the end of each PVC pipe. The percolated water from the soil was characterized in the laboratory for conductivity, pH and the contents of calcium (Ca), magnesium (Mg), total nitrogen, sum of $N\text{-}NH_4^+$ and $N\text{-}NO_3^-$ (N), sodium (Na), sulfur (S), phosphorus (P) and potassium (K) following the methodology described by (Teixeira et al., 2017).

The data obtained were subjected to analysis of variance (ANOVA) in the Sisvar statistical program (Ferreira, 2019). When there was significance, the Tukey test ($p \leq 0.05$) was applied to compare means and regression to compare doses. Significant regression equations at $p \leq 0.05$ were presented. In addition, the chemical characteristics of the percolated water evaluated were analyzed for ionic speciation using the computer program Visual Minteq 3.1 (Gustafsson, 2016).

RESULTS AND DISCUSSION

The use of Phosphogypsum provided a significant increase ($p \leq 0.05$) in the K^+ content in the percolated water of the Dystrophic Red Oxisol (Figure 1 A). Comparing the averages, it is observed that Phosphogypsum increased the K content in the water by 1.83 times compared to the use of Gypsite Pernambuco and 2.44 times for Gypsite Maranhão. Leaching of exchangeable potassium due to the use of gypsum can occur depending on the planting system and the type of soil. However, intense leaching of e K^+ along the soil profile, caused by the application of high doses of gypsum is possible (Serafim et al., 2012).

The increase of 1.95 times in the S content in the water was provided when the Pernambuco Gypsum was used (Figure 1 B), even though this gypsum is characterized by the lower S content in its composition. When coming from agricultural gypsum, sulfate has great mobility in the soil, which is even greater in soils with a sandy texture. The application of gypsum to the surface often results in an increase in the sulphate content of the soil (Caires et al., 2011).

The doses of Phosphogypsum used provided a reduction in the total N content in the percolated water of the Oxisol (Figure 1C). It is observed that there was a reduction of 0.0133 mg L^{-1} of N at each applied dose of Phosphogypsum. The literature shows that gypsum application caused reduction in N contents in the 0.2 to 0.5 m layers (Zardo, 2011).

It is observed that the pH showed a significant quadratic response at 5% probability with the use of phosphogypsum and Gypsites Pernambuco and Maranhão, depending on the doses applied. The dose of 2.55 t ha^{-1} of Phosphogypsum and 2.87 t ha^{-1} of Gypsite Maranhão provided the highest water pH. The doses of Gypsite Pernambuco provided a maximum increase of 0.6 units in the pH of the leachate (Figure 1D). Gypsum indirectly influences the increase in soil pH, having the effect of increasing the plant rooting area, thus improving the use of water and nutrients by the plants. Under field conditions Caires et al. (2011) observed that gypsum did not influence soil acidity, but its long-term residual effect resulted in calcium, magnesium and sulfur availability after eight years of application.

The electrical conductivity (EC) of the leachate was significant with the application of doses of Phosphogypsum (Figure 1E). It is verified that the application of 2.50 t ha^{-1} resulted in a reduction of EC in the percolated water. The gradual dissolution of the gypsum applied to the soil maintains the electrical conductivity at adequate levels in order to assist in the physical properties of the soil (Ramos, 2012). There was a linear reduction in the Ca content in the percolated Oxisol with the use of Pernambuco Gypsite (Figure 1F). Each dose of gypsum used resulted in 0.31 mg L^{-1} of Ca less in the percolated water of the Oxisol. Recent study also show the tendency of calcium from gypsum to be displaced in the soil profile, thus increasing the exchangeable Ca^{2+} content (Araújo et al., 2016).

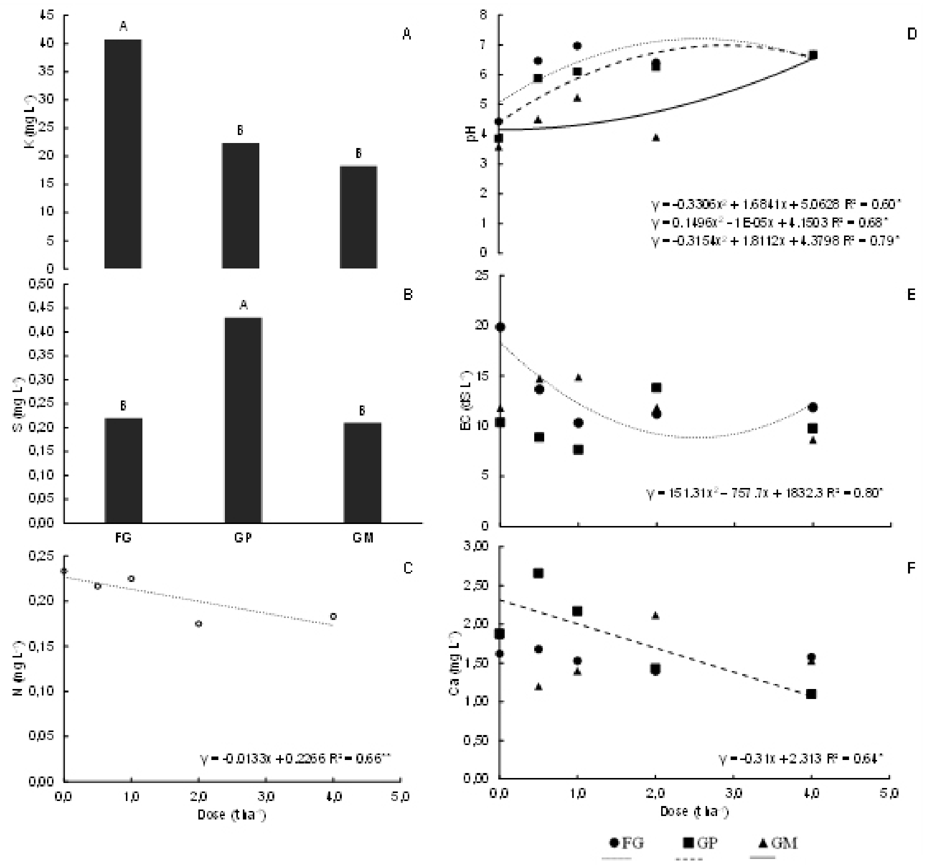

Figure 1. Content of potassium (A), sulfur (B) and nitrogen (C), hydrogen potential (D), electrical conductivity (E) and calcium content (F) in the percolated water of a Dystrophic Red Oxisoil. A, B and C significant in the isolated sources of variation and D, E and F in the interaction between gypsum and doses in the ANOVA. Means followed by the same letter do not differ by Tukey's test (p≤0.05), * Significant at p≤0.05, ** Significant at p≤0.01.

Figura 1. Contenido de potasio (A), azufre (B) y nitrógeno (C), potencial de hidrógeno (D), conductividad eléctrica (E) y contenido de calcio (F) en el agua percolada de un Oxisoil Rojo Distrófico. A, B y C significativos en las fuentes de variación aisladas y D, E y F en la interacción entre yeso y dosis en ANOVA. Medias seguidas de la misma letra no difieren por la prueba de Tukey (p≤0.05), * Significativo a p≤0.05, ** Significativo a p≤0.01.

It is observed that, regardless of the sources and doses of gypsum used, Ca was present in the percolated water of the Oxisol in at least 99.36% in the divalent ionic form, Ca^{2+} (Table 1). It is possible to verify that there was a portion of the element forming ionic pairs with sulfur ($CaSO_4$) and nitrogen ($CaNO_3^+$). As seen, the use of gypsum allows Ca to percolate in the soil profile, thus improving the environment so that root growth occurs and, consequently, greater absorption of water and nutrients by plants (Novais et al., 2007). Magnesium (Mg) also presented a percentage greater than 99.95 of its divalent form Mg^{2+} (Table 1). However, small percentages of Mg in the water were found to form an ion pair with sulfur ($MgSO_4$). There are works indicating that gypsum has the ability to form soluble ionic pairs with Mg (Ferreira et al., 2013). Due to this, the application of gypsum is not recommended in acidic soils with low Mg contents, as there is a possibility of Mg deficiency for plants.

Nitrogen formed ionic pairs in the Oxisol percolated water with Ca ($CaNO_3^+$), K (KNO_3) and with Na ($NaNO_3$). However, its most expressive presence in water occurred in the form of nitrate (NO_3^-), representing more than 99% of the chemical species found (Table 1). The N is a very reactive element in soil. Thus, the genesis of Brazilian soils does not favor the retention of this element, mainly in the form of nitrate. Therefore, it tends to be carried along the soil profile, until it reaches aquifers. Given the dynamics of N in the soil, there is a concern on the part of the agricultural sector that the management of nitrogen fertilization is carried out in a way that crops can efficiently use the nutrient and reduce losses by leaching (Gelain et al., 2011).

Table 1. Calcium, magnesium, nitrogen and sulfur chemical species and percentage of occurrence in leachate of Dystrophic Red Oxisol as a function of Phosphogypsum (FG), Pernambuco Gypsite (GP) and Maranhão Gypsite (GM) doses.

Tabla 1. Especies químicas de calcio, magnesio, nitrógeno y azufre y porcentaje de presencia en el lixiviado de Oxisol Rojo Distrófico en función de las dosis de Fosfoyeso (FG), Yeso de Pernambuco (GP) y Yeso de Maranhão (GM).

Gypsum	CS*	Doses (kg ha^{-1})				
		0.0	0.5	1.0	2.0	4.0
		Ca accurrence				
		%				
FG	Ca^{+2}	99.99	99.94	99.94	99.94	99.95
	$CaSO_4$	0.01	-	-	0.05	-
	$CaNO_3^+$	-	-	-	0.01	-
	$CaSO_4$ (aq)	-	0.06	0.06	-	0.05
GP	Ca^{+2}	99.99	99.93	99.83	99.36	99.99
	$CaSO_4$	0.01	-	-	0.44	-
	$CaNO_3^+$	-	-	-	0.20	-
	$CaSO_4$ (aq)	-	0.07	0.17	-	0.1
GM	Ca^{+2}	99.99	99.94	99.96	99.60	99.93
	$CaSO_4$	0.01	-	-	0.17	-
	$CaNO_3^+$	-	-	-	0.23	-
	$CaSO_4$ (aq)	-	0.06	0.04	-	0.07
		Mg accurrence				
FG	Mg^{+2}	99.98	99.95	99.95	99.96	99.96
	$MgSO_4$ (aq)	0.02	0.05	0.05	0.04	0.04
GP	Mg^{+2}	99.98	99.94	99.86	99.66	99.92
	$MgSO_4$ (aq)	0.02	0.06	0.14	0.34	0.08
GM	Mg^{+2}	99.98	99.95	99.96	99.86	99.95
	$MgSO_4$ (aq)	0.02	0.05	0.04	0.14	0.05
		N accurrence				
		%				
FG	NO_3^-	99.91	99.49	99.57	99.54	99.54
	$CaNO_3^+$	0.07	0.42	0.41	0.36	0.41
	KNO_3 (aq)	0.01	0.08	0.02	0.08	0.05
	$NaNO_3$ (aq)	-	-	-	0.03	-
GP	NO_3^-	99.91	99.33	99.39	0.49	99.68
	$CaNO_3^+$	0.07	0.64	0.55	88.95	0.31
	KNO_3 (aq)	0.01	0.02	0.05	9.61	0.01
	$NaNO_3$ (aq)	-	-	-	0.94	-
GM	NO_3^-	99.91	99.64	99.59	0.35	99.56
	$CaNO_3^+$	0.07	0.34	0.39	93.35	0.41
	KNO_3 (aq)	0.01	0.02	0.02	5.70	0.03
	$NaNO_3$ (aq)	-	-	-	0.61	-
		S accurrence				
FG	SO_4^{-2}	92.67	74.70	78.36	78.25	76.88
	$MgSO_4$ (aq)	3.00	8.62	4.09	6.21	5.98
	$CaSO_4$ (aq)	4.16	16.01	17.39	14.60	16.81
	$NaSO_4^-$	0.04	0.09	0.04	0.38	0.03
	KSO_4^-	0.13	0.57	0.12	0.56	0.29
GP	SO_4^{-2}	92.67	69.18	73.92	-	82.31
	$MgSO_4$ (aq)	3.00	9.30	5.10	15.58	3.25
	$CaSO_4$ (aq)	4.16	21.34	20.50	82.71	14.31
	$NaSO_4^-$	0.04	0.04	0.14	0.25	0.04
	KSO_4^-	0.13	0.13	0.34	1.45	0.09
GM	SO_4^{-2}	92.67	82.11	80.34	-	78.17
	$MgSO_4$ (aq)	3.00	2.21	2.42	18.54	4.38
	$CaSO_4$ (aq)	4.16	15.51	17.09	80.50	17.20
	$NaSO_4^-$	0.04	0.04	0.02	0.15	0.03
	KSO_4^-	0.13	0.13	0.13	0.80	0.22

* chemical species

The ionic speciation for sulfur of percolated water from the Dystrophic Red Oxisol indicate is noticed that this presented ionic pairs with Ca, Mg, K, Na in smaller percentages and predominated in the form of sulfate (SO_4^{2-}) (Table 1). The use of gypsum in agricultural areas is highlighted due to the input's ability to minimize the negative effects of soil acidity by reducing aluminum (Al) saturation. There are many studies that show the efficiency of SO_4^{2-} in reducing Al activity in the soil, making gypsum a technique widely adopted by rural producers.

Among the elements evaluated, phosphorus formed the most ionic pairs, in a total of 13 (Table 2). It appears that, regardless of the dose of gypsum applied, the chemical species $H_2PO_4^-$ predominated in the percolated water, with at least 61% of occurrence. However, the application of 2 t ha^{-1} of the Gypsites provided the formation of ionic pairs of P bound to Ca ($CaHPO_4$), this being 92.86% (Gypsite Pernambuco) and 48.23% (Gypsite Maranhão).

Phosphorus is an element that has low mobility in the soil, making the absorption process difficult, consequently limiting crop productivity. Brazilian agriculture has stood out in the world production scenario, largely due to care aimed at increasing the efficiency of phosphate fertilization. Thus, gypsum is a practice that can provide improvements in the construction of soil profiles with high fertility, since, depending on the gypsum solubility, it is possible to transport nutrients to deeper layers, so much so that although P tends to remain retino in the soil, it was observed that there was percolation of the same depending on the doses and sources of gypsum used.

The use of Gypsites provided low percentages of potassium (K) linked to sulfur and nitrogen (≤0.05%). The ionic forms found with the application of 2 t ha^{-1} were: KSO_4^- (Pernambuco) and KNO_3 (Pernambuco and Maranhão). However, it is noteworthy that in the other doses, there was the presence of 100% K^+ in the percolated water. The dose of 2 t ha^{-1} of Gypsites provided the formation of ionic pairs $NaSO_4^-$ and $NaNO_3$ in percentages ≤ 0.02%. The other doses of Gypsites and the use of Phosphogypsum provided 100% of the Na percolated water of the Oxisol in its monovalent form (Na^+).

Results published in the literature show the success of gypsum in agricultural production areas, making this practice widely used in Brazil. However, it is noteworthy that the presence of nutrients in the percolated Oxisol studied reinforces the technical recommendations described in the literature. Therefore, the use of gypsum, regardless of the source, must be carried out considering the particularities of each soil, climate, cultivation system and soil fertility management in each region.

Table 2. Phosphorus chemical species and percentage of occurrence in leachate of Dystrophic Red Oxisol as a function of Phosphogypsum (FG), Pernambuco Gypsite (GP) and Maranhão Gypsite (GM) doses.

Tabla 2. Especies químicas de fósforo y porcentaje de presencia en lixiviados de Oxisol Rojo Distrófico en función de las dosis de Fosfoyeso (FG), Yeso de Pernambuco (GP) y Yeso de Maranhão (GM).

Pernambuco (GP) y Yeso de Maranhão (GM).

Gypsum	CS*	Doses (kg ha^{-1})				
		0.0	0.5	1.0	2.0	4.0
		P occurrence				
		----------% ----------				
FG	NaH_2PO_4 (aq)	0.01	0.04	0.01	0.15	0.02
	HPO_4^{-2}	14.64	17.34	17.42	14.93	6.90
	$H_2PO_4^-$	81.89	65.85	69.52	72.91	85.52
	H_3PO_4	-	-	-	-	0.01
	$MgHPO_4$ (aq)	1.64	6.94	3.15	4.11	1.86
	$CaHPO_4$ (aq)	1.31	7.42	7.71	5.56	3.01
	$CaH_2PO_4^+$	0.42	1.99	2.03	1.86	2.53
	$KHPO_4^-$	-	0.14	0.03	0.11	0.03
	KH_2PO_4 (aq)	0.04	0.17	0.04	0.17	0.11
	$NaHPO_4^-$	0.01	0.05	0.02	0.16	-
	$CaPO_4^-$	-	0.07	0.07	0.04	-
	Na_2HPO_4 (aq)	-	-	-	-	-
	K_2HPO_4 (aq)	-	-	-	-	-
GP	NaH_2PO_4 (aq)	0.01	0.01	0.05	0.98	0.02
	HPO_4^{-2}	14.64	16.86	17.32	-	6.75
	$H_2PO_4^-$	81.89	61.87	65.98	0.07	87.97
	H_3PO_4	-	-	-	-	0.01
	$MgHPO_4$ (aq)	1.64	7.86	4.15	0.30	0.92
	$CaHPO_4$ (aq)	1.31	10.38	9.58	0.92	2.34
	$CaH_2PO_4^+$	0.42	2.82	2.57	92.86	1.93
	$KHPO_4^-$	-	0.03	0.09	-	-
	KH_2PO_4 (aq)	0.04	0.04	-	4.38	0.03
	$NaHPO_4^-$	0.01	0.02	0.07	-	-
	$CaPO_4^-$	-	0.10	0.09	-	-
	Na_2HPO_4 (aq)	-	-	-	0.02	-
	K_2HPO_4 (aq)	-	-	0.10	0.46	-
GM	NaH_2PO_4 (aq)	0.01	0.01	-	0.13	0.01
	HPO_4^{-2}	14.64	17.57	17.50	-	6.86
	$H_2PO_4^-$	81.89	72.29	71.14	0.01	86.15
	H_3PO_4	-	-	-	-	0.01
	$MgHPO_4$ (aq)	1.64	1.64	1.83	19.31	1.33
	$CaHPO_4$ (aq)	1.31	6.62	7.43	48.23	3.01
	$CaH_2PO_4^+$	0.42	1.73	1.95	19.36	2.51
	$KHPO_4^-$	-	0.03	0.03	0.26	0.02
	KH_2PO_4 (aq)	0.04	0.04	0.03	0.52	0.08
	$NaHPO_4^-$	0.01	0.02	0.01	0.10	-
	$CaPO_4^-$	-	0.06	0.06	0.27	-
	Na_2HPO_4 (aq)	-	-	-	0.45	-
	K_2HPO_4 (aq)	-	-	-	11.39	-

* chemical species

CONCLUSION

The use of gypsum, in addition to mitigating the effects of soil acidity, provides changes in the chemistry of the percolated water. These changes will be more or less intense depending on the source and dose of gypsum used. The ionic speciation of percolated water from Red Oxisol with the application of phosphogypsum, Gypsite Maranhão and Pernambuco revealed that there was a predominance of absorbable forms by nitrogen, calcium, magnesium, potassium, phosphorus and sulfur plants. This shows that the inadequate management of gypsum can cause nutrient losses, having as some of the consequences, the lack of these for the cultures and the increase in the production cost.

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