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RESEARCH ARTICLE

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PERFORMANCE TEST OF GRANULATED POTASSIUM CHLORIDE WITH HUMIC ACIDS ADDITION

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ABSTRACT

The objective of this work was to evaluate the efficiency of granulated fertilizers based on potassium chloride with humic acids in the production of dry matter and the accumulation of macronutrients in corn plants. The experiment was carried out in a greenhouse, using soil samples from the 0-20 cm depth layer of *a Planossoloháplico*. A completely randomized block design was adopted, in a 2 x 5 + 1 factorial scheme with two sources of humic acid extraction (vermicompost and peat), five concentrations of humic acids in the granulations (0; 2.5; 5.0; 7.5 and 10.0%) and one additional treatment that consisted of the application of commercial potassium chloride, with three replications. The plants were collected at sixty days after sowing. There was a slight difference in the production of the dry root mass. The P accumulation presented the most pronounced differences, whereas the granulated potassium chloride with humic acid extracted from vermicompost showed the best performance.

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INTRODUCTION

The largest known potassium reservoirs in the world are located in Belarus, Canada, and Russia. These three countries together hold more than sixty percent of potash production due to their large and high-quality deposits. According to a report by the Food and Agriculture Organization of the United Nations (FAO), the global demand for potash fertilizers is expected to be 37.04 Mt of K_2O in 2020, with an increase of 10.51% when compared to 2016. There is a projection that demand in Latin America and the Caribbean will be 7.8 Mt of K_2O fertilizer, with an estimated availability of 1.24 Mt, which will result in a shortage of 6.60 Mt (FAO, 2017). With the present potassium fertilizer production rates, the world's known potash reservoirs would last for more than 250 years. However, with the increase in annual production, the cost per ton of K_2O produced will increases due to the different accessibility and quality of the deposits. Also, potash deposits are unevenly distributed throughout the world (Lanzerstorfer, 2019). In the world context, Brazil is a significant importer of potassium fertilizer. In 2014, the country imported a total of 5.43x106 Mg of K_2O equivalent, with an import cost of US\$ $2.9x10^9$.00. This amount of fertilizer corresponded to 95% of the potassium consumed in this same year (DNPM, 2015). Potassium is a primary macronutrient essential for plant development and is associated with water and nutrients transport in plant tissues. Besides, it is a vital nutrient for photosynthesis and protein synthesis. It plays a fundamental role in stomatal regulation, opening, and closing, which directly influences the exchange of water vapor, oxygen, and carbon dioxide. If the potassium available to plants falls short of adequate amounts, their growth will reduce, and yield will decrease (Lanzerstorfer, 2019). Potassium loss by leaching commonly occurs when using highly soluble mineral fertilizers, especially in regions with intense rainfall. Losses by leaching increase nutrient demand and the cost of agricultural production,in addition to promoting eutrophication of watercourses.

Many techniques have been developed to improve potassium fertilizer efficiency, such as polymer-coated fertilizers (Said *et al*., 2018). Potassium chloride (KCl) is the most used potassium fertilizer worldwide due to its relatively low cost (Pereira *et al*., 2019). Therefore, studies on increasing the efficiency of potassium fertilization are critical. However, research that aims to increase efficiency in the use of this fertilizer is mainly focused on the application methods, not evaluating alternative sources or technologies that modify its solubility (Leal *et al*., 2015). Humic acids (HA) can supply nutrients to plants through mineralization, thus being able to stimulate their development. HA has a positive influence on the absorption of ions, as they increase the rate of enzymatic reactions in the Krebs cycle, which results in higher production of ATP that is related to the content of chlorophyll and synthesis of nucleic acids (Olaetxea *et al*., 2018).

Results from the literature indicate that HA stimulate both the activity and the synthesis of the H+- ATPase plasma membrane (Morozesk *et al*., 2017). The most accepted hypothesis to explain this stimulus is that the structural subunits of HA can access receptors on the surface or inside the plasma membrane of the root cells. As a result, a greater differential in electrochemical potential is formed through plasmalemma, which facilitates passive potassium absorption, for example (Olaetxea *et al*., 2018). The chemical extraction of organic matter is based on the solubility of humic substances (SH) and is described in detail by the International Humic Substances Society (IHSS, 2014). When reducing the pH of the alkaline extract with HCl, the fraction of the larger molecular mass precipitates, this fraction is nominatedhumic acids. The benefits provided by humic substances to plant growth and nutrition can be related mainly to their ability to form stable organometallic complexes and to the activation of enzymes involved in the acquisition of nutrients. This action is more pronounced in some nutrients, such as Fe, N, and P (Olaetxea *et al*., 2018). The use of inputs based on humic substances in commercial crops has been gaining importance due to the responses obtained, mainly in crops of high economic interest (Meirelles *et al*., 2017). Humic acids influence plant development, with the humic fraction having the highest bioactivity (Gholami *et al*., 2018). When added to the fertilizer, it can positively influence the acquisition of nutrients due to its influence in the root architecture and the increase in the specific surface of the root system. Given the above, the present study aimed to evaluate the efficiency of granulated fertilizers based on potassium chloride with the addition of humic acids in the accumulation of dry matter and NPK incorn crop.

MATERIAL AND METHODS

The humic acids were isolated and purified from vermicompost and peat, following the recommendations of the International Humic Substances Society (IHSS, 2014). After purification, total nitrogen, phosphorus, and potassium were determined, following the Tedesco *et al*. (1995) methodology. The results are shown in Table 1.

Table1. Nitrogen, phosphorus, and potassium content of the humic acids used in the fertilizers production

Extraction source	N	P	K
	-------------------	%-------------	
Peat	3,78	0,22	0,67
Vermicompost	3,09	0,25	0,05

The commercial KClwas macerated in a mortar and then passed through a sieve of 0.42 mm mesh opening (ABNT / ASTM 40) to facilitate homogenization and improve the granulation process. The other components of the fertilizers consisted of humic acid, polyvinylpyrrolidone (PVP), and washed sand. PVP was added to the mixture to promote greater hardness to fertilizers. Like KCl, all other components of the mixture passed through a sieve with a 0.42 mm mesh. After homogenization, the mixtures were granulated using a pelletizer disk. After granulation, the fertilizer was classified according to the diameter of the granules. The granules that passed through a sieve with a 4 mm mesh opening and were retained in a sieve with a 2 mm mesh opening were used in the experiment. Then, the granules were transferred to a forced-air ventilation oven at a temperature of 45 ºC for 16 hours. At the end of this period, the fertilizers passed through 4 and 2 mm sieves, being separated according to the diameter. Then, the K_2O contents of each granulometric fraction was determined to verify the possible segregation of the components of the mixture. The result is shown in Table 2.

Table 2. K_2O percentage in each fertilizer fraction sieved.

HA (%)	Ø < 2 mm	2 mm < Ø < 4 mm	Ø > 4 mm
	----------------------------	K_2O %----------------------------	
0.0	56.42	55.57	55.44
2.5	56.41	56.70	53.29
5.0	58.00	57.55	55.47
7.5	54.87	58.47	56.95
1.0	55.77	56.84	56.51

For the corn cultivation, the soil used was collected in the 0-20 cm depth layer of a soil classified as *Planossolo Háplico,* with a sandy texture (780 g kg^{-1} of sand). The soil was incubated with dolomitic limestone, in order to raise the pH to 6.0, following the methodology proposed by Stafanato (2009). After pH stabilization, chemical soil analysis was performed (Table 3) according to Teixeira *et al*. (2017). Then, the soil was air-dried, sieved with a 4 mm mesh sieve and homogenized. The experimental units consisted of PVC tubes with 20 cm in diameter and 40 cm in height.

Tabela 3. Chemical characterization of the soil after incubation

C Tot.	pH	P	K	Ca	Mg	H+Al	Al	S	T	V
g/dm^3		--mg/dm^3--		-------------------		$cmol_c dm^{-3}$	-------------------			%
4	6.0	33	89	3.4	1.1	1.4	0	4.73	6.13	77

Micronutrients

B	Cu	Fe	Mn	Zn
-------------------	-------------------	mg dm^{-3}	-------------------	-------------------
0.42	1.2	47	8.8	3.1

S – sum of basis; T – cation exchange capacity at pH 7.0; V – base saturation.

The experiment was conducted in a greenhouse in a randomized block design, in a 2 x 5 + 1 factorial scheme with three replications, totaling 11 treatments and 33 experimental units. The treatments consisted of two sources of humic acid extraction (peat and vermicompost), five concentrations of humic acids (0, 2.5, 5.0, 7.5, and 10.0%) and an additional treatment consisting of potassium chloride fertilizer. The plant used as the indicator was the hybrid corn AG-1051. Before planting, each experimental unit received fertilization with 1.60 g of urea and 0.36 g of triple superphosphate, equivalent to 200 kg ha^{-1} of N and 60 kg ha^{-1} of P_2O_5, quantities extracted by this crop to produce a yield of 17.13 t ha^{-1} of silage dry matter (Coelho and França, 1995). Twenty seeds of the corn weresown per experimental unit. Immediately after sowing, fertilizers were applied on the surface and in the total area, according to each treatment. Then, the pots received water to raise the moisture to 70% of the field capacity. During the experiment, the soil moisture was maintained in the range of 50 to 70% of the field capacity, performing daily water replenishment based on weighting the pots. The temperature and humidity were monitored during the experiment using a digital thermo-hygrometer installed inside the greenhouse (Figure 1). Forty days after sowing (DAS), there was a slight shortage in the greenhouse when the ventilation system became inoperative. On this day, the maximum temperature recorded inside the greenhouse exceeded 45 ºC.

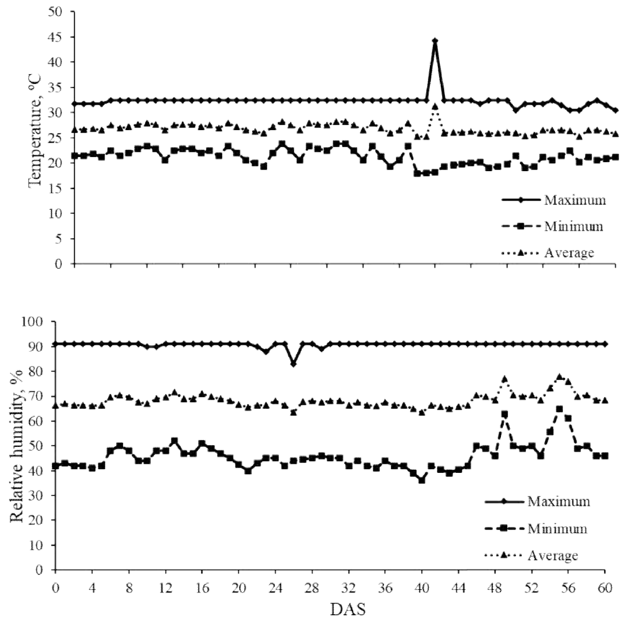

Figure 1. Average temperatureandrelative humidity, maximumand minimum, inside the greenhouse during the experiment.

At 7 DAS, thinning was carried out, leaving two plants per pot. At 60 DAS, corn plants were harvested, with leaves, stems, panicles, and roots being collected separately. The samples were placed in paper bags, identified, and dried in an oven with forced air circulation at 65º C until they reached a constant weight. The determination of the dry matter and NPK accumulation was then carried out, following the methodology proposed by Tedesco *et al*., 1995. With these results in hand, data were tested with the Kolmogorov-Smirnov test to verify the normality of the residues. The variables that met normality were subjected to analysis of variance. When significant at 5% probability by the F test, the Tukey test at 5% compared the averages of the qualitative factor (sources of HA extraction) and the data of the quantitative factor (HA concentration in the fertilizers) were submitted to regression analysis. The significance of the determination coefficients was performed using the 5% F test.

RESULTS AND DISCUSSION

Table 4 summarizes the analysis of variance of the accumulation of the dry mass of leaves, stems, panicles, roots, and total dry mass of corn plants according to the application of fertilizers. There was a significant difference in the dry mass accumulation of the roots in response to the HA extraction source. There was no difference between the additional treatment and the factorial for any of the dependent variables.

The KCl granulated with peat HA provided higher accumulation of dry mass in corn roots when compared to the granulated KCl plus vermicompost HA. However, the commercial KCl treatment was statistically the same as the granulated fertilizers (Figure 2). The recalcitrance and lability of humic fractions are chemical properties that define their ability to stimulate root parameters. The growth of more extensive roots is related to more complex structures and less chemical functionalization, while the emission of smaller roots is related to less complex and more functionalized structures, presenting higher structural lability (Garcia *et al*., 2016). According to Morozesk et al. (2017), HA can directly influence the development and productivity of plants due to the presence of organic acids that can interfere in their metabolism and improve soil fertility by favoring the availability of nutrients. However, in the present study, there was already an initial high availability of nutrients in the soil (Table 3), which may have minimized the beneficial effects of HA when applied together with mineral fertilization.

The application of HA via fertilizer can bring a series of benefits to crops, such as an improvement in seed germination, seedling growth, and the use of nutrients by plants. The benefits to plant growth happen by improving the physical and chemical properties of the soil, and by increasing root growth and permeability of the cell membrane (Guo *et al*., 2019). When evaluating the effect of humic acid on endogenous hormone levels and the activity of antioxidant enzymes during the *in vitro* rooting of azalea, Elmongy *et al*. (2018) found a beneficial effect exerted by HA on rooting. This effect was related to physiological and metabolic changes during the formation of adventitious roots. It was observed that HA (1 and 2 mg L^{-1}) increased the endogenous hormone levels of indole-3-acetic acid and gibberellic acid in rooted shoots, especially at the beginning of root development. Table 5 summarizes the analysis of variance of nitrogen, phosphorus, and potassium accumulation in leaves, stems, panicles, roots, and total accumulation in corn plants in response to the application of the fertilizers. An influence of HA concentration on the accumulation of total N in corn plants was verified. There was also a difference between the sources of extraction in the accumulation of P in the roots. Also, the additional treatment and the factorial in the total accumulation of P in corn plants presented differences. The extraction source of HA influenced the accumulation of potassium in the leaves, total aerial part, and total accumulation.

With the increase in the HA concentration in fertilizers, there was a linear increase in nitrogen accumulation in the aerial part and total in corn plants (Figure 3). Granulated fertilizers without HA (concentration of 0%) had a negative effect on the accumulation of N compared to commercial KCl. The addition of humic substances (HS - fulvic acids, humic acids and humines) in the rhizosphere or on leaves in different plant species and different soil conditions provides a significant improvement in N absorption by the roots with subsequent increase in the assimilation of this nutrient (Olaetxea *et al*., 2018). Studies carried out with HS extracted from vermicompost indicated a positive regulation of nitrate transporters in the leaves of corn, but not in the roots. The beneficial effects of HS on the acquisition and assimilation of N by vegetables are also related to the increase in the activity of enzymes related to N metabolism, such as nitrate reductase, as well as by other enzymes involved in the ammonium assimilation (Olaetxea *et al*., 2018).

Table 4. Mean squares of the analysis of variance of the dry mass of leaves, stalks, panicles, roots and total dry mass accumulated by corn plants depending on the application of treatments (VF= Variation Factor; SE = source of extraction; CAH = concentration of humic acids; Ad. = additional treatment; Fact. = factorial, CV= coefficient of variation).

VF	GL	Mean square				
		Leaves	Stalks	Panicles	Roots	Total
(SE)	1	75.64^{ns}	78.55^{ns}	0.19^{ns}	114.43*	41.28^{ns}
(CAH)	4	26.42^{ns}	103.62^{ns}	2.99^{ns}	19.67^{ns}	296.76^{ns}
FE x CAH	4	43.07^{ns}	130.39^{ns}	0.60^{ns}	55.38^{ns}	410.86^{ns}
Ad.x Fact.	1	19.88^{ns}	34.31^{ns}	0.46^{ns}	6.84^{ns}	185.31^{ns}
Resídue	20	34.68	157.05	1.37	18.65	223.57
CV (%)		7.15	20.85	13.42	10.05	7.70

*significant at 5% de probability by the F test;^{ns}: non-significant.

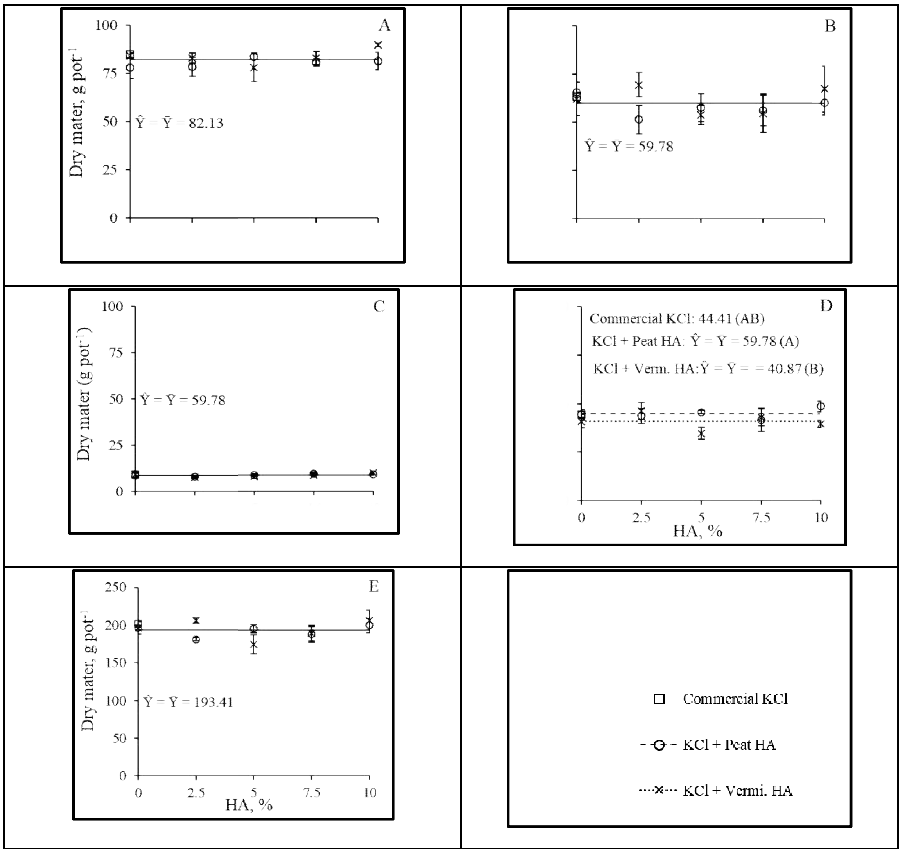

Figure 2. Dry matter of leaves (A), stalks (B), panicles (C), roots (D),and total(E) produced by corn plants ammended with commercial potassium chloride (KCl), potassium chloride granulated with increasing concentrations of humic acids extracted from peat (KCl + Peat HA) and from vermicompost (KCl + Verm. HA).Columns with the same letter, within each plant part, are not significant at 5% de probability by the F test.

Table 5. Mean squares of the analysis of variance of nitrogen, phosphorus, and potassium accumulation in leaves, stalks, panicles, roots, and total in corn plants depending on the application of the fertilizers. (VF= Variation Factor; SE = source of extraction; CAH = concentration of humic acids; Ad. = additional treatment; Fact. = factorial, CV= coefficient of variation)

VF	GL	Square Mean				
		Leaves	Stalks	Panicles	Rooots	Total
Nitrogen						
SE	1	42401.52^{ns}	31.27^{ns}	0.00^{ns}	342.33^{ns}	37271.23^{ns}
CAH	4	48765.60^{ns}	1931.32^{ns}	4193.37^{ns}	2169.16^{ns}	116624.67*
FE x CAH	4	3105.26^{ns}	3126.62^{ns}	1161.67^{ns}	5256.57^{ns}	19709.36^{ns}
Ad. x Fact.	1	12073.38^{ns}	1587.14^{ns}	6777.15^{ns}	452.53^{ns}	44424.29^{ns}
Residue	20	21246.31	2852.87	1865.46	1920.29	28577.30
CV (%)		19.35	40.68	20.21	22.39	13.06
Phosphorus						
SE	1	305.22^{ns}	627.00^{ns}	4.06^{ns}	270.78*	787.87^{ns}
CAH	4	127.95^{ns}	1289.89^{ns}	28.09^{ns}	26.53^{ns}	950.47^{ns}
FE x CAH	4	106.15^{ns}	554.43^{ns}	5.01^{ns}	172.01^{ns}	1121.90^{ns}
Ad. x Fact.	1	209.59^{ns}	720.85^{ns}	54.29^{ns}	88.91^{ns}	1882.89*
Residue	20	72	520.62	18.69	61.50	405.09
CV (%)		7.34	24.70	14.20	17.00	7.07
Potassium						
SE	1	158503.45*	16109.64^{ns}	32.53^{ns}	340.50^{ns}	250889.42*
CAH	4	29822.07^{ns}	11015.52^{ns}	2853.37^{ns}	375.58^{ns}	52770.02^{ns}
FE x CAH	4	31275.81^{ns}	5718.98^{ns}	392.91^{ns}	1972.05^{ns}	56629.44^{ns}
Ad. x Fact.	1	2781.80^{ns}	343.60^{ns}	2130.15^{ns}	2130.15^{ns}	100.20^{ns}
Residue	20	29860.28	20802.26	940.95	940.95	48318.48
CV (%)		7.30	21.12	11.48	11.48	6.08

*significant at 5% de probability by the F test;^{ns}: non-significant.

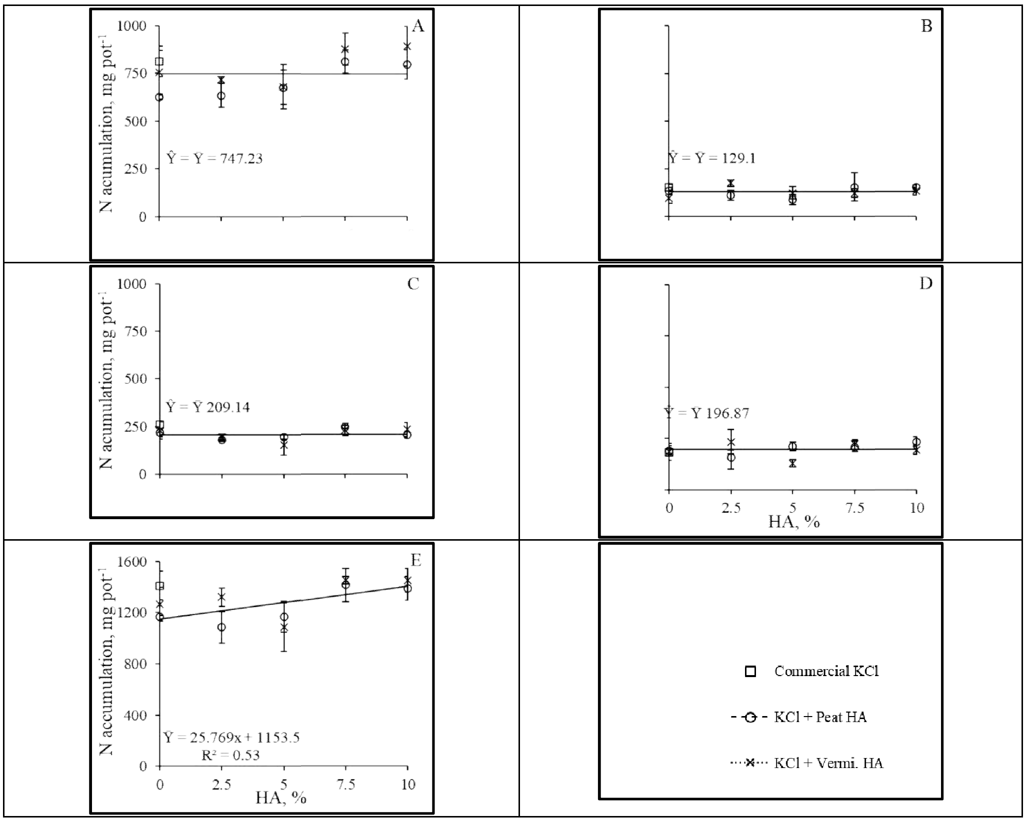

Figure 3. Nitrogen accumulation (A), stalks (B), panicles (C), roots (D) and total (E) produced by corn plants ammended with comercial potassium chloride (KCl), potassium chloride granulated with increasing concentrations of humic acids extracted from peat (KCl + Peat HA) and from vermicompost (KCl + Verm. HA).Vertical bars indicate the standard deviation of the means.

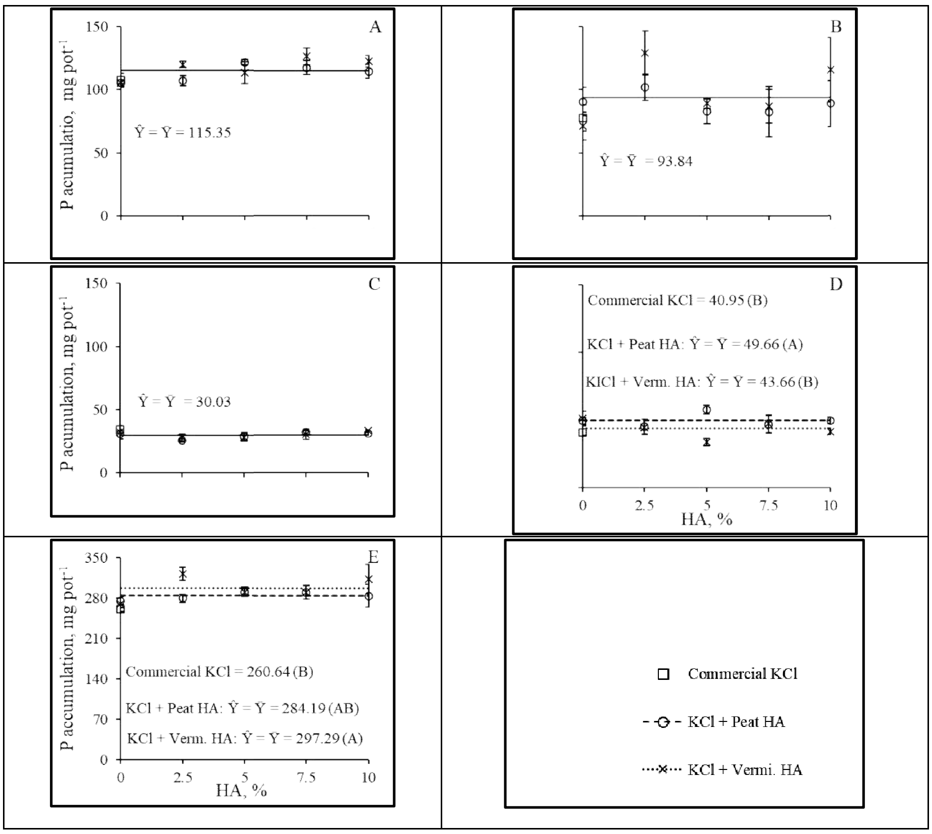

Figura 4. Phosphorus accumulation (A), stalks (B), panicles (C), roots (D) and total (E) produced by corn plants ammended with comercial potassium chloride (KCl), potassium chloride granulated with increasing concentrations of humic acids extracted from peat (KCl + Peat HA) and from vermicompost (KCl + Verm. HA). Columns with the same letter, within each plant part, are not significant at 5% de probability by the F test. Vertical bars indicate the standard deviation of the means

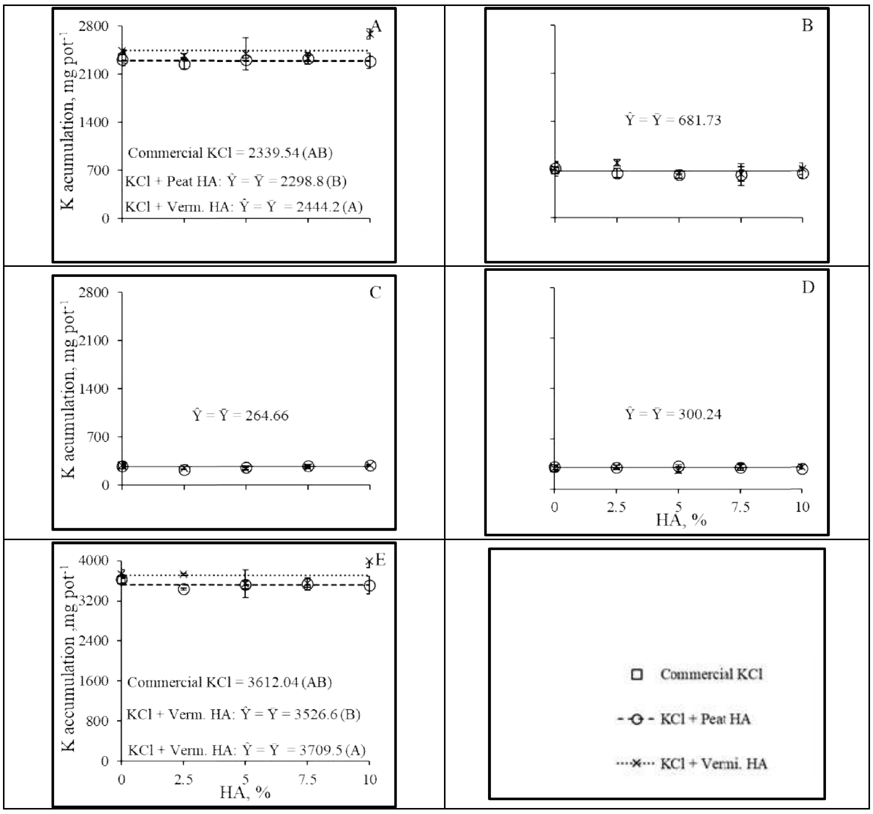

Figure 5. Phosphorus accumulation (A), stalks (B), panicles (C), roots (D) and total (E) produced by corn plants ammended with comercial potassium chloride (KCl), potassium chloride granulated with increasing concentrations of humic acids extracted from peat (KCl + Peat HA) and from vermicompost (KCl + Verm. HA). Columns with the same letter, within each plant part, are not significant at 5% de probability by the F test. Vertical bars indicate the standard deviation of the means.

In order to evaluate the efficiency of urea with the addition of HA, Zhang *et al*. (2019) found an increase of up to 21.33% in the dry matter production of aerial parts of plants fertilized with urea plus HA (0.5% of HA) when compared to plants fertilized with urea only. N absorption was also higher when HA was added to the fertilizer, promoting an increase of 29.46% when compared to urea only. N losses were also minimized by up to 30.05% when HA was added to the fertilizer. When estimating the volatilization of N-NH_3 after the application of fertilizers formulated with humic acids and urea, Gurgel *et al.* (2016) found a decrease in losses of up to 38% with the addition of 5 and 10% humic acids extracted from peat. It was also observed that the effect of humic acids in reducing ammonia volatilization was greater when fertilizers were applied to sandy texture soils (Gurgel *et al.,* 2016). Granulated KCl with HA extracted from peat promoted higher accumulation of P in the roots when compared to commercial KCl and granulated KCl with HA extracted from vermicompost (Figure 4). However, the later provided a higher total P accumulation in corn plants compared to commercial KCl. The soil used in the present study had a sandy texture. Thus, the phenomenon of specific adsorption had little influence on the uptake of P by the plant. There was a small difference in the P content between treatments with humic acids. The HA extracted from peat and vermicompost showed 0.22 and 0.25% of P, respectively. These concentrations are very similar, not being the main reason for the difference in P accumulation. The most accepted hypothesis for the differences found in the P accumulation by corn plants relies on the effect provided by HA in the absorption kinetics of nutrients that, depending on their structure, can act more or less intensely. When applying a dose equivalent to 20 m^3 ha^{-1} of an HA-rich compound (53 g kg^{-1}), Solaiman *et al.* (2019) observed a significant increase in the production of root and total dry mass of lettuce plants grown in pots when compared to the application of the same dose of a compound with low HA content (3 g kg^{-1}). In this work, the application of the humic acid-rich compound promoted higher concentrations of N and P in the plant tissue compared to the compound poor in HA. The addition of granulated KCl with HA extracted from vermicompost provided higher accumulations of K in the leaves, total aerial part, and in the total K accumulation when compared to the granulated KCl with HA extracted from peat (Figure 5). Commercial KCl did not differ from any of the granulated HA fertilizers in what concerns the potassium accumulation.

However, granulated KClwith HA extracted from peat provided less accumulation of K in the leaves and total aerial part. When evaluating the kinetic parameters of K absorption in bean cropin response to the application of increasing doses of humic substances extracted from mineral coal, Rosa *et al.* (2009) observed a decrease in Imáx, Cmín, and Km, with a consequent reduction in the rate of absorption of K, with the decrease in Imáx being the determining parameter for the decline. One of the main effects of HS in plant metabolism is the hydrolysis of ATP due to the stimulus on the activity of proton pumps of the plasmalemma. Morozesk *et al.* (2017) found an increase in the activity of H^+ ATPases in corn plants treated with HA extracted from vermicompost and sewage sludge. These proteins act in the active transport of H^+ from the cytosol to the outer side of the cell. Due to the increase in the difference of electrochemical potential generated through plasmalemma, potassium can be passively absorbed in a higher speed, depending on the external concentration (Geifus, 2017).

Conclusion

The application of the different fertilizers tested did not have a pronounced influence on the dry mass production of corn, showing a higher efficiency when KCl was granulated with humic acid extracted from vermicompost in comparison to humic acid extracted from peat. The most pronounced differences in nutritional parameters were obtained in P accumulation, with the granulated potassium chloride plus humic acid extracted from vermicompost showing the best performance. For N and K, the differences found were small, with granulated fertilizers having performance similar to commercial potassium chloride.

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