

Division - Soil Use and Management | Commission - Lime and Fertilizer

Soil Solution Nutrient Availability, Nutritional Status and Yield of Corn Grown in a Typic Hapludox under Twelve Years of Pig Slurry Fertilizations

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ABSTRACT: Nutrient content in soil solution may vary in response to the applied amount of pig slurry (PS). Thus, the aim of this research was to evaluate the nutrient content in soil solution and its relationship to nutritional status and yield of corn under 12 years of annual fertilization with increasing doses of PS, soluble mineral fertilizer (MF) and PS combined with MF. The experiment was conducted under field conditions in a *Latossolo Vermelho Distroférrico* (Typic Hapludox). The treatments consisted of pig slurry at annual rates of 50 and 100 m³ ha⁻¹ (PS50 and PS100), soluble mineral fertilizer (MF), and pig slurry at a rate of 25 m³ ha⁻¹ complemented with mineral fertilizer (PS25 + MF), distributed in a randomized block design. Soil solution was sampled at two depths, 0.40 and 0.80 m, on six dates after the fertilizer applications. The nitrate (NO¬¬N) and K levels in the soil solution were measured 40, 108 and 135 days after the last application of fertilizer (DAAF), being subsequently correlated with corn yield. PS50 provide contents of NO¬¬N, P and K to the soil solution similar to those supplied by MF or PS25+MF. The critical limit of 10 mg L⁻¹ NO¬¬N in the soil solution was exceeded on 108, 135 and 230 DAAF, at rates of 100 and 50 m³ ha¹ of PS and on 230 DAAF when using MF.

Keywords: organic fertilizer, manure, leaching.

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INTRODUCTION

Brazilian pig farming has been growing and thereby increasing the amount of generated waste. This fact raises concerns about the proper disposal of this residue, which is usually applied as fertilizer on farmlands, following technical recommendations with the aim of improving the chemical, physical and biological soil properties and, as consequence, increasing crop yields (Grohskopf et al., 2015; Mafra et al., 2015). Meanwhile, the use of pig slurry (PS) as a fertilizer requires the development of application technologies that enable taking advantage of its favorable effects on soil and agricultural production. Thus, it should be considered that this residue has a quite variable elemental composition, mainly regarding the type of pig rearing management adopted, the amount of water used and the manner of storage (Diesel et al., 2002).

When applied to the soil, PS can promote changes in the contents and forms of mineral nutrients, especially macronutrients such as N, P and K, by virtue of the expressive added quantities (Cassol et al., 2012; Mafra et al., 2014). Most of the N content in PS is in the form of ammonium (NH_{+}^{+}) (Schirmann et al., 2013) and as applications are made on the soil surface, part is lost in the form of ammonia and another part is rapidly transformed into nitrate (NO_{3}^{-}). Nitrate is mobile in the soil (Payet et al., 2009; Tuli et al., 2009), which favors N leaching. However, despite the low mobility of P in the soil, successive applications of mineral and mostly organic fertilizers increase the concentrations of organic and inorganic P in the solution, which favors its movement along the soil profile (Heathwaite, 2000; Fernandes et al., 2015). In contrast, K is in mineral form in PS and after its application, a large share is adsorbed to negative charges in the soil, but is easily exchanged and made available within the solution (Ernani et al., 2007; Werle et al., 2008).

Another possible effect of the continuous application of PS on soil is the accumulation of Cu and Zn, which are found at high concentrations within this waste, since they are added in high amounts to feed formulations as a way to compensate the low utilization of such nutrients by the animals (Marcato, 1997). However, Cu and Zn are strongly held in the soil by specific adsorption to organic matter and oxides which compose the soil (Sipos et al., 2008).

The Conama's Resolution N. 420/2009 (Conama, 2009) sets guideline values for groundwater environmental management of 10.0, 2.0 and 1.05 mg L^{-1} for NO_3^- , Cu and Zn, respectively. However, there are no guidelines or reference levels for P and K in the Brazilian legislation. Studying the concentrations of these elements in the solution of soils fertilized with PS, primarily NO_3^- and P is necessary because of their potential to promote water eutrophication, which reduces water oxygen level and quality as well as aquatic species diversity (Correll, 1998; Smith et al., 2007; Usepa, 2009). For this to transpire, the soil solution must contain concentrated amounts of nutrients, especially NO_3^- and P, and there must be a movement of this solution along the soil profile, reaching groundwater, springs and surface water.

Therefore, knowing the dynamics of nutrients within the soil solution is essential to prevent surface and ground water contamination, reduce production costs and correctly manage fertilizations, especially with organic fertilizers. Nevertheless, the complexity and difficulty in extracting soil solution, as well as the low concentration and difficulty of quantifying nutrients, render the use of this method a technical criterion for fertilizer recommendation, being restricted to a few researches (Schlotter et al., 2012; Souza et al., 2013).

Fertilization, both with mineral and organic fertilizers, increases the concentration of nutrients within the soil solution, which increases the absorption of such elements by plants, with a consequent increase in foliar content. Thus, we have raised as a hypothesis for this study that the continuous use of PS could increase the concentration of nutrients within the soil solution in proportion to the dose applied, thereby increasing the yield of corn cultivated. However, this residue provides an excessive increase when applied in annual doses exceeding 50 m³ ha⁻¹, especially to a depth of 0.8 m. As a result, amounts



of NO_3^- , P, Cu and Zn may reach values higher than those considered safe for human consumption and environmental quality. In this way, quantifying the nutrient amounts within the soil solution of areas fertilized with PS and/or mineral fertilizer, for 12 continuous years, can yield a great understanding of their dynamics, aside from comprehending their relationships with mineral and organic fertilizations. This knowledge would contribute to define appropriate PS doses to prevent the loss of nutrients and water pollution.

The main objective of this study was to determine the soil solution contents of N, P, K, Cu and Zn within samples of a *Latossolo Vermelho Distroférrico* (Typic Hapludox) at depths of 0.4 and 0.8 m from an area grown with no-tillage corn-oat rotation and 12 years of annual fertilizations with pig slurry, soluble mineral fertilizer and combined fertilization (pig slurry + mineral fertilizer), as well as relating these data with plant nutritional status and corn grain yield under no-tillage system.

MATERIALS AND METHODS

The experiment was carried under field conditions in 2001, in Campos Novos, Santa Catarina, Brazil, in an area with an average altitude of 863 m. Local climate is classified as humid mesothermal with balmy summer (Cfb), according to the Köppen's classification system, with an average annual rainfall of 1,480 mm, well distributed throughout the year and an average annual temperature of 16 °C. The soil is classified as *Latossolo Vermelho Distroférrico* (Typic Hapludox) derived from basalt rocks.

Soil chemical properties in the 0.0-0.2 m layer (mean of four composite samples of 10 subsamples) were analyzed according to Tedesco et al. (1995), with the following results: pH(H_2O) 6.1; pH(SMP) 6.0; Al $^{3+}$, Ca $^{2+}$, and Mg $^{2+}$ 0.0, 8.2 and 4.6 cmol $_c$ kg $^{-1}$, respectively; P (Mehlich-1) 6.4 mg kg $^{-1}$; K (Mehlich-1) 97 mg kg $^{-1}$; total organic carbon 25 g kg $^{-1}$; and base saturation 87 %. Soil physical properties were analyzed by the methods suggested by Claessen (1997): clay 680 g kg $^{-1}$, and bulk density 1.3 Mg m $^{-3}$. Clay fraction mineralogy is mainly composed of kaolinite, iron oxides (goethite and hematite) and clay minerals of the 2:1 type with hydroxy-Al polymers within interlayers and the minimal occurrence of gibbsite and quartz (Almeida et al., 2003).

Before experiment implementation, the area was used for commercial cropping (corn, soybeans, wheat and oat) and managed under no-tillage system for 12 years. In the 2000/2001 harvest, one year before the beginning of experiment, the farmer started fertilizing soil with PS throughout the entire area, applying 25 m³ ha⁻¹.

The experiment comprises the annual application of the following treatments: pig slurry (PS) in annual rates of 50 (PS50) and 100 m³ ha⁻¹ (PS100), soluble mineral fertilizer (MF), and fertilization with 25 m³ ha⁻¹ PS complemented with mineral fertilizer (PS25 + MF). The MF treatment consisted of urea, triple superphosphate and potassium chloride at annual rates of N, P and K, respective to 130, 44 and 58 kg ha⁻¹, from 2001 to 2006, respective of 170, 57 and 66 kg ha⁻¹, from 2007 to 2012. These rates were defined based on recommendations aimed at yielding 8 Mg ha⁻¹ of corn grains, in the first period, and 11 Mg ha⁻¹ in the second (Tedesco et al., 2004). The treatment PS25 + MF, besides PS, included applications of the soluble fertilizers mentioned above, with doses of N, P and K adjusted so that the amounts of the three nutrients were similar to those provided in the MF treatment. Treatments were performed in plots of 12×6.3 m, in a randomized block experimental design with four replications.

Applications were held 15 to 20 days after crop-desiccation spraying with glyphosate, every October. Fertilizers were broadcast on top of the soil/ residue surface, distributing the PS with the aid of a manure spreader and spreading MF manually. In MF treatment, the amount of N was split, applying 20 % in the planting furrow and the rest was divided into two seasons, being applied by throwing as corn top dressings.

The slurry used in the experiment was derived from the manure of growing pigs, being stored in continuous flow in open dunghills for about four months prior to the application.



Liquid swine slurry used in the experiment was characterized (Table 1), by withdrawing a representative sample analyzed in duplicate. A dry matter determination was performed by drying in an oven with air circulation at 65 °C. The pH determination was made by reading with a pH meter directly in PS samples and nutrient analyses were carried using "in natura" aliquots of the residue (wet basis) and performed as described by Tedesco et al. (1995).

Corn (*Zea mays*) and black oat (*Avena strigosa*) were grown yearly in succession under a no-tillage system (NT), except in the summer of 2002/2003, when black beans (*Phaseolus vulgaris*) was grown replacing corn, and in the winters of 2005 and 2008, when radish (*Raphanus sativus*) was cropped to replace oat. A simple hybrid of corn was used in a planting density of seven plants m⁻², with a 0.60 m space between rows. For oat and radish, we used the 'Comum' and 'IPR-116' cultivars in sowing densities of 60 and 10 kg ha⁻¹ seeds, respectively, both with 0.20 m spacing between rows. The bean crop was carried using the EMPASC 201 cultivar, in a density of 20 plants m⁻².

Corn was sown seven to 12 days after treatment application, usually in the first week of November, while winter crops were sown in the first fortnight of June each year. Sowing of all crops was carried out with a non-tillage tractor-drawn seed drill, composed of a stubble-cutting disc and double disc openers with gauge wheels, a chisel plow and a compactor with two rubber wheels. Grain yield was obtained by manual collection of all fallen and mechanically threshed corncobs over the plot floor area (10.2 \times 4.9 m), and adjusted to 13 % moisture. For winter crops, we evaluated the biomass production within the plot floor area, collecting three 1-m² subsamples per plot. These samples of winter crops were dried at 65 °C until a constant weight was reached.

Thirty samples of leaves were taken opposite corn ear within the middle third of corn plants at tasseling stage to evaluate nutrient uptake (50 % tasseled plants), as described in the fertilization and liming handbook for the states of Rio Grande do Sul and Santa Catarina in Brazil (Tedesco et al., 2004). Following collection, leaf tissue was washed and placed to dry in an oven with forced air at 65 °C, and subsequently ground. The contents of N, P and K within the corn leaf tissue were determined according to methods described by Tedesco et al. (1995).

Soil solution sampling was held from October 2011 to November 2012, up to 320 days after treatment application, encompassing corn and oat cycles (Table 2), as significant rainfalls enabled sampling and the extraction of soil solution.

Table 1. Chemical properties of the pig slurry applied yearly in field experiment within the period from 2001-2012 on a Typic Hapludox

Year	рН	DM	ТОС	TN	Р	K
				— kg m ⁻³ —		
2001	6.7	66	19	3.4	1.4	1.2
2002	7.1	26	11	2.6	1.0	1.2
2003	6.9	32	13	2.6	1.1	1.3
2004	7.3	43	17	3.7	1.4	1.5
2005	7.8	56	17	3.2	1.5	1.1
2006	7.0	114	36	4.6	2.8	1.7
2007	7.3	55	20	2.7	1.8	1.1
2008	7.1	68	24	2.4	0.4	1.3
2009	7.2	69	26	6.6	1.1	3.5
2010	7.4	57	33	3.9	1.9	2.0
2011	7.2	61	28	3.5	1.7	1.8
2012	7.1	43	31	3.8	1.4	1.9
Mean	7.2	58	23	3.6	1.5	1.6

DM: dry matter; TN: total nitrogen; TOC: total organic carbon. Determinations carried according to Tedesco et al. (1995).



These samples were collected three days after high-intensity rains (Figure 1), using extractors as in Reichardt et al. (1977). Two extractors were assembled in the middle of each plot, one at a depth of 0.4 m and the other at 0.8 m. The system was maintained at 60 kPa for three days using a syringe inside extractors. During vacuum application, toluene was dropped into collection flasks, aiming at inhibiting microbial activity. The solution volume extracted varied according to soil moisture and hence to water retention energy in the soil, with around 80 mL solution being collected from each extractant at the depth of 0.4 m, and 40 mL at 0.8 m. After collecting, field samples were frozen and kept at -10 °C until the time of analysis.

The analysis evaluated the amounts of NH_4^+ , NO_3^- ($NO_2^- + NO_3^-$), P and K, as in Rice et al. (2012), as well as contents of Cu and Zn following the method of Tedesco et al. (1995). The contents of NH_4^+ , NO_2^- and NO_3^- were measured via means of absorption spectroscopy in continuous flow injection analysis (FIA), using the colorimetric method, P via ascorbic acid method and K by spectrophotometry (KMnO₄).

Table 2. Soil solution extraction and analysis dates, represented in days after the application of fertilizers (DAAF) and days after crop emergence (DAE) of each season, and conditions or crop development stage

DAAF	DAE	Crop	Conditions/ Development stage
40	27	Corn	Vegetative – 9 th leaf
90	77	Corn	Vegetative – 18 th leaf
108	95	Corn	Reproductive - blooming
135	128	Corn	Reproductive - chalky grains
230	-	Without crop	Corn residue on soil surface
320	62	Oat	3 rd node detectable

Corn sowing was performed on 3 DAAF and the harvest on 165 DAAF. Oat sowing was done on 250 DAAF and the desiccation on 340 DAAF.

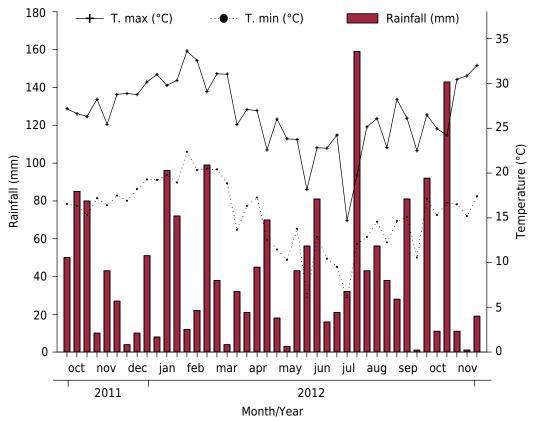


Figure 1. Means of maximum and minimum temperatures and sum of rainfalls at 10-day intervals during the experiment.



Data underwent variance homogeneity and normality analyses that indicated no need for data transformation. After all parametric statistical assumptions were met, the variance analysis was performed using the F-test, according to a randomized block design. When significant, the effect of treatments and sampling times were compared using the LSD test (p<0.05). Nutrient contents within the soil solution were correlated to respective plant tissue amounts and corn grain yields by Pearson correlation (p<0.05).

RESULTS AND DISCUSSION

Soil solution ammonium concentration (NH_4^+-N) on all sampling dates was below the lower detection limit set by this determination method. A possible explanation may be related to ammonium adsorption to surface charges on the soil colloids, both in clays and organic matter, thus turning from the liquid to the solid phase. In addition, there is also a fast transformation of NH_4^+-N into nitrate (NO_3^--N), regarding the nitrification process in an oxidizing environment (Payet et al., 2009; Zhang et al., 2011), which is conditioned by high drainage and aeration of *Latossolo Vermelho Distroférrico* (Typic Hapludox). According to Aita et al. (2007), the entire amount of NH_4^+-N in the pig slurry (PS) can be nitrified within ten days after application. It is considered that on average, 60 % of total N in manure is in the mineral form (NH_4^+-N) (Schirmann et al., 2013). Fast nitrification increases the concentration of NO_3^--N in soil solution, allowing N great mobility within soil profile.

Nitrate contents sampled from SS at depths of 0.4 and 0.8 m varied with sampling times as well as type of fertilizers (Table 3). These variations may be attributed to the dose and type of fertilizer used (Lafleur et al., 2012; Coscione et al., 2014), to the rainfall intensity which, in turn, influences the water infiltration rate into the soil (Kaiser et al., 2010; Cambier et al., 2014), to groundwater table fluctuation, to crop development and to the soil surface management adopted.

The PS100 had an NO_3^-N content that was superior to the others on 40, 108, 135 and 230 days after the application of fertilizer (DAAF) at the depth of 0.4 m (Table 3). It can be justified by the N supply of 350 kg ha⁻¹ provided in 2011 and of 380 kg ha⁻¹ in 2012 through PS applications, which were twice the dose applied in PS50 and MF. The largest amount of NO_3^-N provided by PS100 was also observed on 40, 90, 108 and 135 DAAF at a depth of 0.8 m. However, on 90 DAAF, PS25+MF had results similar to PS100. On 230 DAAF, MF showed the highest content compared to the others at a depth of 0.8 m (Table 3). Such result suggests a possible N displacement within soil profile and may be due to the MF soluble form as well as fertilization splitting in corn crop when compared to single application and pre-sowing using PS.

The lowest soil solution contents of NO_3^--N were observed on all evaluated dates and both depths for PS50. Drinkwater et al. (1998) demonstrated that N losses were reduced by using organic fertilizers. Similarly, Basso et al. (2005) reported the absence of groundwater contamination with NO_3^--N , after applying pig slurry at rates of up to 40 m³ ha¹ in an Argissolo Vermelho Distrófico arênico (Ultisol).

The differences between sampling times with respect to the soil solution NO_3^-N content can be explained by variations in the phenological stage of crops (Table 2). The largest NO_3^-N content might have favored N mineralization and coincided with periods of lower demand by the crops. In addition, treatments with PS might have been influenced by the slower release of nutrients of PS compared to MF.

The PS100 and MF treatments exceeded the critical limit of 10 mg L^{-1} for NO_3^--N in soil solution on 40, 108, 135 and 230 DAAF at a depth of 0.4 m; and on 108, 135 and 230 DAAF at a depth of 0.8 m (Table 3). Thus, both organic and mineral fertilizers promote NO_3^--N contents above the critical values. Furthermore, it should be considered that these soil



Table 3. Soil solution content of nitrate in a Typic Hapludox under 12 years of no-tillage corn-oat rotation and yearly fertilized with with pig slurry (PS), mineral fertilizer (MF) and pig slurry combined with mineral fertilizer (MF + PS), at two depths (0.4 and 0.8 m), over time in days after fertilizer application (DAAF)

Treatment	DAAF								
Treatment -	40	90	108	135	230	320			
	NO ₃ -N (mg L ⁻¹)								
			4 m						
MF	4.7 Bc	2.6c ^{ns}	10.3 Bb	9.8 Bb	23.6 Ba	1.8 c ^{ns}			
PS25+MF	2.8 Bc	2.3 c	9.0 Bb	8.5 Bb	15.4 Ca	1.7 c			
PS50	3.3 Bb	1.8 bc	9.7 Ba	9.2 Ba	4.0 Db	0.6 c			
PS100	16.4 Ac	2.2 d	20.6 Ab	20.2 Ab	25.6 Aa	1.0 d			
		0.8 m							
MF	0.6 Bc	1.1 Bc	3.2 Bb	2.6 Bb	10.9 Aa	0.4 c ^{ns}			
PS25+MF	0.7 Bb	2.0 ABb	4.2 Ba	3.8 Ba	4.0 Ba	1.0 b			
PS50	0.6 Bb	0.9 Bb	4.0 Ba	3.6 Ba	5.1 Ba	0.5 b			
PS100	3.1 Ac	3.3 Ac	17.5 Aa	17.0 Aa	5.4 Bb	1.8 c			
			P (m	ıg L ⁻¹)					
			0.4	4 m					
MF	2.2 ABbc	1.0 d	3.0 Ba	2.7 Bab	0.4 cd	0.7 d			
PS25+MF	1.8 Bb	0.5 c	3.1 Ba	2.7 Ba	0.5 c	0.4 c			
PS50	2.2 ABb	0.7 c	3.1 Ba	2.7 Bab	0.4 c	0.5 c			
PS100	2.5 Ab	0.6 c	3.8 Aa	3.7 Aa	0.6 c	0.4 c			
		0.8 m							
MF	4.3 Aa	0.7 b	2.5 Aba	2.2 Aba	0.5 b	0.5 b			
PS25+MF	3.8 BCa	0.2 c	2.3 ABb	2.0 Bb	0.5 c	0.2 c			
PS50	3.4 Ca	0.4 c	2.2 Bb	1.9 Bb	0.5 c	0.2 c			
PS100	3.9 ABa	0.2 c	2.8 Ab	2.5 Ab	0.7 c	0.2 c			
	K (mg L ⁻¹)								
	0.4 m								
MF	2.0 B ^{ns}	2.9 ^{ns}		1.6 B	3.0 B	2.0			
PS25+MF	1.4 Bb	3.8 ab	2.3 Bab	1.8 Bb	5.1 Ba	3.1 ab			
PS50	3.5 B ^{ns}	2.1	2.4 B	1.9 B	3.0 B	1.2			
PS100	16.3 Aa	2.7 d	13.4 Ab	13.0 Ab	10.7 Ac	1.8 d			
	0.8 m								
MF	2.7 Abc	4.1 Ba	3.0 ^{ns} bc	2.6 Bbc	2.2 Cc	3.3 Bb			
PS25+MF	3.4 Aa	3.3 Ca	2.3 b	1.8Bb	3.2 Ba	2.5 Cab			
PS50	1.5 Bb	3.0 Ca	2.4 ab	1.9 Bb	2.3 Cab	2.2 Cab			
PS100	3.2 Ad	5.7 Aa	4.2 bc	3.8 Acd	4.7 Ab	4.9 Ab			

 $^{^{(1)}}$ Pig slurry (PS) at annual doses of 50 (PS50) and 100 (PS100) m³ ha⁻¹; soluble mineral fertilization (MF); and PS at a dose of 25 m³ ha⁻¹ complemented with MF (PS25+MF). Means with different letters (lowercase horizontally and uppercase vertically) differ by the LSD test (p≤0.05).

solution extractants might underestimate the nutrient content in the solution, because the tension in these devices can only be implemented and maintained under conditions of high humidity in the soil (Souza et al., 2013). However, there is a need for further studies using this method of soil solution extraction, by porous capsule extractants, mainly in relation to its use under field conditions.

The lowest soil solution contents of NO_3^-N on 90 DAAF (Table 3) in all treatments were related to the corn phenological stage of development, in which an elevated absorption of nutrients occurred. Furthermore, after 90 DAAF, most of the applied NH_4^+N had already been transformed into NO_3^-N , raising the probability of N leaching.



At 108 and 135 DAAF, the NO_3^-N contents were higher at both depths (0.4 and 0.8 m), which might have been due to the low nutritional demand of corn. At 230 DAAF, at 0.4 m depth, the MF, PS25+MF and PS100 achieved the highest amounts of NO_3^-N (Table 3). During this period, there is no nutrient absorption since crops had been removed, in addition to nutrient cycling in corn residues left on the soil surface (Table 2) and roots in the soil, which are mineralized and subsequently nitrified.

After 320 DAAF, at both depths, NO₃-N contents decreased (Table 3). It might have occurred mainly due to the lower residual effect of N fertilizer, added to leaching losses over time, and to N absorption by oat plants introduced on 250 DAAF. At 320 DAAF, oat plants were at the third tiller stage (Table 3), at which there is higher N uptake and a decrease in soil solution. It is worth mentioning that PS was applied once a year, a week prior to corn sowing, i.e. with some residual effect from oat crop.

Regarding the soil solution P content, there were variations with sampling dates and fertilizer type at both depths (Table 3). There was slight variation in the contents among treatments at both depths; however, PS100 stood out by providing the highest amounts, being higher than PS25+MF at 40, 108 and 135 DAAF, as well as than MF and PS50 at 108 and 135 DAAF at 0.4 m; and treatment PS50 at 40,108 and 135 DAAF, and PS25+MF at 135 DAAF at 0.8 m. These findings can be explained by the P intake of 140 kg ha⁻¹ yr⁻¹ in PS100, compared to an intake of 57 kg ha⁻¹ in MF and PS25+MF and 70 kg ha⁻¹ in PS50.

There is a lack of studies comparing methods of soil solution extraction and nutrient determination (Schlotter et al., 2012). Phosphorus contents between 0.2 and 0.3 mg L^{-1} are taken as an ideal for soil solution and further plant growth (Basso et al., 2005). In this way, all treatments yielded adequate concentrations of P during corn vegetative and reproductive stages.

Furthermore, there are few studies in the literature regarding the use of solution extractants with porous capsules under field conditions to evaluate the soil solution regarding contents of N and mainly for P and K evaluations. The studies developed with these extractants are more common under controlled conditions (Souza et al., 2013; Fernandes et al., 2015).

The lowest P contents were observed after 90 DAAF. This behavior was also observed in NO_3^-N and might be due to the greater absorption of these nutrients by corn plants during this period (Table 3). At 108 and 135 DAAF, we observed an increase in the soil solution P contents at both depths. In this period, crop has a reduced demand for nutrients.

Corn residue and root decomposition contributes to organic C provision; however, for low molecular weight structures, it aids in orthophosphate complexation, which implies a reduction in the soil solution P content. This may account for the reduction of P in the solution between 135 and 230 DAAF, together with the concentration of this element in corn grains, reducing the contribution of crop residues in P cycling when compared to N and K.

The P concentration on 320 DAAF was below those on 40, 108 and 135 DAAF. During this period, the greatest nutrient absorption by oat plants justifies this outcome (Table 3), which was also observed for NO_3^-N within the same period. The rapid adsorption of P in a Typic Hapludox, even the most stable P forms (non-labile), and the absence of restitution of this element into the system are factors that have contributed to reduce the amount of P in the solution on 230 and 320 DAAF.

The largest amounts of K in soil solution were found for PS100, on 40, 108, 135 and 230 DAAF at both depths, except for 108 DAAF, when there was no difference at the depth of 0.8 m (Table 3). These results highlighted the potential for loss of K for the application volume of $100 \text{ m}^3 \text{ ha}^{-1} \text{ yr}^{-1}$ implemented in this treatment, which corresponds to 180 and 190 kg ha⁻¹ in 2011 and 2012 against the application of 66 kg ha⁻¹ K in MF and PS25+MF and 90 and 95 kg ha⁻¹ for PS50 in 2011 and 2012.



Except for PS100 at 0.4 m, the soil solution K contents were similar for all evaluated dates and treatments at both depths. It can be influenced by the high mobility of this nutrient throughout soil profile and the rapid desorption from colloids, which quickly balance the ionic stability of soil solution (Ernani et al., 2007).

The K, in solution, poses no risk of water contamination. The Brazilian legislation (Conama's Resolution N. 420/2009) does not provide guidelines for the environmental management of K in areas fertilized with organic composts to ensure the groundwater quality.

Even after 12 years of annual application of swine manure and soluble mineral fertilizer, Cu and Zn forms could not be detected within the soil solution at both depths. The amounts of Cu and Zn were below the lower limit of detection (0.005 mg L^{-1}) of the device used; therefore, such data were not displayed in the results. Both micronutrients are strongly retained by specific adsorption, forming covalent bonds with organic matter and with crystalline forms of Fe and Al oxides within the clay fraction (Sipos et al., 2008; Paradelo et al., 2011). It is relevant to mention that kaolinite clay fraction and iron oxide crystalline forms, such as goethite and hematite, are predominant in *Latossolos Vermelhos Distroférricos* (Almeida et al., 2003).

For both depths, the soil solution NO_3^-N content had no correlation with the N amounts found in corn plant tissue in all evaluation dates. On the other hand, this ion showed correlation with crop yield at both of the assessed layers, for the dates of 40, 108 and 135 DAAF, during the entire corn cycle (Table 4). We must highlight that leaf sampling for N, P and K determinations was performed during the same period of soil solution collection at 90 DAAF.

Table 4. Correlation of nitrogen, phosphorus and potassium contents in the soil solution with concentrations in corn leaf tissue and grain yields in a Typic Hapludox subjected to 12 years of fertilization with with pig slurry, mineral fertilizer and pig slurry combined with mineral fertilizer, at two depths (0.4 and 0.8 m) and on different days after the application of fertilizers (DAAF)

		DAAF								
Depth Par. (1)		40		9(90		108		135	
		Content ⁽²⁾	Yield ⁽³⁾							
m										
		Nitrate								
0.4	a b	ns	8050 95	ns	ns	ns	7321 112	ns	7391 110	
	r	0.32	0.66	0.33	-0.2	0.28	0.65	0.26	0.66	
0.8	a b	ns	8296 328	ns	ns	ns	8007 96	ns	8044 98	
	r	0.09	0.50	0.14	0.32	0.16	0.70	0.16	0.71	
		Phosphorus								
0.4	a b	ns	ns	ns	ns	ns	ns	ns	ns	
	r	0.05	0.33	-0.03	-0.43	-0.25	0.28	-0.01	0.43	
0.8	a b	ns	ns	ns	ns	ns	ns	ns	ns	
	r	0.32	-0.46	-0.01	-0.45	0.27	0.13	0.33	0.13	
		Potassium								
0.4	a b	ns	8136 98	ns	ns	ns	8197 100	ns	8245 100	
	r	0.28	0.73	0.46	-0.34	0.30	0.64	0.31	0.64	
0.8	a b	ns	ns	ns	ns	ns	7154 520	ns	7428 510	
	r	0.36	-0.02	0.43	0.45	0.29	0.58	0.27	0.56	

⁽¹⁾ Evaluated parameters, of which a and b are the linear model parameters and r is the correlation coefficient. (2) Content of the respective element in the diagnosed samples of corn leaf. (3) Corn yield (kg ha⁻¹). Pearson correlations with a significance level of p<0.05. ns: non-significant correlations.



The average content of total N in the tissue of corn plants was 25, 24, 24 and 28 g kg⁻¹ for MF, PS25+MF, PS50 and PS100, respectively. However, some of these levels are considered suboptimal for corn crops, which ranges between 28 and 32 g kg⁻¹ according to Malavolta et al. (1997). The low levels of this nutrient in plant tissue were likely influenced by droughts during the months of November, December and January (Figure 1), when plants have high demand for water, thus reducing the levels of N in plant tissue.

Corn grain yields for MF, PS25+MF, PS50 and PA100 were 7829, 8235, 9056 and 9695 kg ha⁻¹, respectively (Table 4). Also, in order to reach 90 % of these values and present the content of NO_3^--N in solution that results in this level of effective response, we found contents of 7, 12 and 13 mg L¹ of NO_3^--N in the soil solution at 0.4 m and 2, 10 and 9 mg L¹ at 0.8 m on 40, 108 and 135 DAAF, respectively.

There was no correlation between P content in the soil solution with its concentration in plant tissue or with crop yield (Table 4). The average content of P in the tissue of corn plants was 2.3, 2.1, 2.4 and 3.1 g kg $^{-1}$, respectively, for the treatments MF, PS25+MF, PS50 and PS100. These levels are within the range considered adequate for the corn crop, which is 1.9 to 3.5 g kg $^{-1}$ as stated by Malavolta et al. (1997).

The average levels of K in the tissue of the corn plants were 23, 22, 22 and 24 g kg⁻¹ for MF, MF, PS25+MF, PS50 and PS100, respectively, therefore being within the range considered adequate for corn crop, which is from 18 to 30 g kg⁻¹ according to Malavolta et al. (1997). The K content in the soil solution was not correlated with the content of this element in plant tissue for both collection times and depths; however, presenting a correlation with grain yields on 40 DAAF at 0.4 m, on 108 and 135 DAAF at both depths.

Based on linear adjustment and using 90 % of the maximum yield, it was observed that the effective levels of K in the soil solution were 6.8, 10.0 and 9.3 mg L^{-1} on 40, 108 and 135 DAAF at 0.4 m, as well as 3.4 and 3.2 mg L^{-1} on 108 and 135 DAAF at 0.8 m (Table 4). These data can assist in soil fertility decision-making, aiming at the efficient use of economic resources, yield increase and environmental monitoring improvement.

After 12 years of application, using MF, PS25+MF, PS50 and PS100, the cumulative extractable P concentrations in the soil were low, with the exception of PS100 that presented a P content that is considered high. In contrast, the K values are considered very high (Table 5), according to the fertilization and liming handbook for the states of Rio Grande do Sul and Santa Catarina (Tedesco et al., 2004).

The levels of organic matter, P and K are referred to contents in the soil after corn and oat cultivations, in which all fertilizers were applied as a single dose and before sowing (Table 5). Unlikely, in MF, nitrogen was parceled, being 80 % of the total dose applied in top dressing.

Table 5. Content of soil organic matter (SOM), available phosphorus (P) and potassium (K) in the layer of 0.00-0.20 m in a Typic Hapludox after 12 years of fertilization with pig slurry (PS), mineral fertilizer (MF) and pig slurry combined with mineral fertilizer (MF + PS)

Treatment ⁽¹⁾	SOM ⁽²⁾	P ⁽²⁾	K ⁽²⁾
	g kg ⁻¹	mg	kg ⁻¹ ———
MF	46	1.4	236
PS25+MF	46	1.9	318
PS50	47	2.2	345
PS100	51	8.5	538

⁽¹⁾ Pig slurry (PS) at annual doses of 50 (PS50) and 100 (PS100) m³ ha⁻¹; soluble mineral fertilization (MF); and PS at a dose of 25 m³ ha⁻¹ complemented with MF (PS25+MF). (2) Determinations carried according to Tedesco et al. (1995).



CONCLUSIONS

The soluble mineral fertilizer and pig slurry applied yearly for twelve years in a Typic Hapludox increases the nutrient content in the solution, especially nitrate and potassium at depths of 0.4 and 0.8 m.

The annual fertilization with the dose of 50 m³ ha⁻¹ pig slurry in no-tillage corn-oat crop rotation provides amounts of nitrate, phosphorus and potassium in the soil solution that are similar to those supplied by mineral fertilizer and mineral fertilizer supplemented with pig slurry.

The nitrate and potassium levels in the soil solution are positively correlated with corn yield.

After 12 years of pig slurry application at doses of up to 100 m³ ha⁻¹ yr⁻¹, the contents of Cu and Zn in the soil solution at depths of 0.4 and 0.8 m remained below 0.005 mg L⁻¹.

The application of 100 m³ ha⁻¹ pig slurry provide nitrate amounts higher than 10 mg L⁻¹ in the soil solution, which is above the maximum ceiling set by Conama's Resolution N. 420/2009.

REFERENCES

Aita C, Giacomini SJ, Hübner AP. Nitrificação do nitrogênio amoniacal de dejetos líquidos de suínos em solo sob sistema plantio direto. Pesq Agropec Bras. 2007;42:95-102. doi:10.1590/S0100-204X2007000100013

Almeida JA, Torrent J, Barrón V. Cor de solo, formas do fósforo e adsorção de fosfato em Latossolos desenvolvidos de Basalto do extremo-sul do Brasil. Rev Bras Cienc Solo. 2003;27:985-1002. doi:10.1590/S0100-06832003000600003

Basso CJ, Ceretta CA, Durigon R, Poletto N, Girotto E. Dejeto líquido de suínos: II – Perdas de nitrogênio e fósforo por percolação no solo sob plantio direto. Cienc Rural. 2005;35:1305-12. doi:10.1590/S0103-84782005000600012

Cambier P, Pot V, Mercier V, Michaud A, Benoit P, Revallier A, Houot S. Impact of long-term organic residue recycling in agriculture on soil solution composition and trace metal leaching in soils. Sci Total Environ. 2014;499:560-73. doi:10.1016/j.scitotenv.2014.06.105

Cassol PC, Costa AC, Ciprandi O, Pandolfo CM, Ernani PR. Disponibilidade de macronutrientes e rendimento de milho em Latossolo fertilizado com dejeto suíno. Rev Bras Cienc Solo. 2012;36:1911-23. doi:10.1590/S0100-06832012000600025

Claessen MEC, organizador. Manual de métodos de análise de solo. 2ª ed. Rio de Janeiro: Centro Nacional de Pesquisa de Solos; 1997.

Conselho Nacional do Meio Ambiente – Conama. Ministério do Meio Ambiente. Resolução N. 420, de 28 de dezembro de 2009. Dispõe sobre critérios e valores orientadores de qualidade do solo quanto à presença de substâncias químicas e estabelece diretrizes para o gerenciamento ambiental de áreas contaminadas por essas substâncias em decorrência de atividades antrópicas. DOU. Brasília, DF, n. 249, 7 dez. Seção 1, p.81-4, 2009.

Correll DL. The role of phosphorus in the eutrophication of receiving waters: a review. J Environ Qual. 1998;27:261-6. doi:10.2134/jeq1998.00472425002700020004x

Coscione AR, Silva LFM, De Maria IC, Andrade CA, Ferracini VL. Solução do solo e análise de componentes principais para monitoramento da aplicação de lodo de esgoto. Rev Bras Cienc Solo. 2014;38:1654-62. doi:10.1590/S0100-06832014000500030

Diesel R, Miranda CR, Perdomo, CC. Coletânea de tecnologias sobre dejetos suínos. Concórdia: Empresa Brasileira de Pesquisa Agropecuária; 2002. (Boletim informativo BIPERS, 14).

Drinkwater L, Wagoner P, Sarranttonio M. Legume-based cropping systems have reduced carbon and nitrogen losses. Nature. 1998;396:262-5. doi:10.1038/24376

Ernani PR, Bayer C, Almeida JA. Mobilidade vertical de cátions influenciada pelo método de aplicação de cloreto de potássio em solos com carga variável. Rev Bras Cienc Solo. 2007;31:393-402. doi:10.1590/S0100-06832007000200022



Fernandes DM, Grohskopf MA, Gomes ER, Ferreira NR, Büll LT. Fósforo na solução do solo em resposta a aplicação de fertilizantes fluidos mineral e organomineral. Irriga. 2015;1:14-27. doi:10.15809/irriga.2015v1n1p14

Grohskopf MA, Cassol PC, Correa JC, Mafra MSH, Panisson J. Organic nitrogen in a Typic Hapludox fertilized with pig slurry. Rev Bras Cienc Solo. 2015;39:127-39. doi:10.1590/01000683rbcs20150080

Heathwaite L. A conceptual approach for integrating phosphorus and nitrogen management at watershed scales. J Environ Quality. 2000;29:158-66. doi:10.2134/jeq2000.00472425002900010020x

Kaiser DR, Reinert DJ, Reichert JM, Streck CA, Pellegrini A. Nitrate and ammonium in soil solution in tobacco management systems. Rev Bras Cienc Solo. 2010;34:379-87. doi:10.1590/S0100-06832010000200011

Lafleur B, Thiffault E, Paré D, Camiré C, Bernier-Cardou M, Masse S. Effects of hog manure application on the nutrition and growth of hybrid poplar (*Populus* spp.) and on soil solution chemistry in short-rotation woody crops. Agric Ecosyst Environ. 2012;155:98-104. doi:10.1016/j.agee.2012.04.002

Mafra MSH, Cassol PC, Albuquerque JA, Correa JC, Grohskopf MA, Panisson J. Acúmulo de carbono em Latossolo adubado com dejeto líquido de suínos e cultivado em plantio direto. Pesq Agropec Bras. 2014;49:630-8. doi:10.1590/S0100-204X2014000800007

Mafra MSH, Cassol PC, Albuquerque JA, Grohskopf MA, Andrade AP, Rauber LP, Friederich A. Organic carbon contents and stocks in particle size fractions of a Typic Hapludox fertilized with pig slurry and soluble fertilizer. Rev Bras Cienc Solo. 2015;39:1161-71. doi:10.1590/01000683rbcs20140177

Malavolta E, Vitti GC, Oliveira SA. Avaliação do estado nutricional das plantas: princípios e aplicações. 2ª. ed. Piracicaba: Potafos; 1997.

Marcato SM. Efeito da restrição alimentar sobre a excreção de minerais nos dejetos produzidos pelos suínos [dissertação]. Pelotas: Universidade Federal de Pelotas; 1997.

Paradelo R, Villada A, Barral MT. Reduction of the short-term availability of copper, lead and zinc in a contaminated soil amended with municipal solid waste compost. J Hazard Mater. 2011;188:98-104. doi:10.1016/j.jhazmat.2011.01.074

Payet N, Findeling A, Chopart JL, Feder F, Nicolini E, Macary HS, Vauclin M. Modeling the fate of nitrogen following pig slurry application on a tropical cropped acid soil on the Island of Réunion (France). Agric Ecosyst Environ. 2009;134:218-33. doi:10.1016/j.agee.2009.07.004

Reichardt K, Libardi PL, Meirelles NMF, Ferreyra FFH, Zagatto EAG, Matsui E. Extração e análise de nitratos em solução de solo. Rev Bras Cienc Solo. 1977;1:130-2.

Rice EW, Baird RB, Eaton AD, Clesceri LS. Standard methods for the examination of water and wastewater. 22nd.ed. Washington, DC: American Public Health Association; 2012.

Schirmann J, Aita C, Giacomini SJ, Pujol SB, Giacomini DA, Gonzatto R, Olivo J. Inibidor de nitrificação e aplicação parcelada de dejetos de suínos nas culturas do milho e trigo. Rev Bras Cienc Solo. 2013;37:271-80. doi:10.1590/S0100-06832013000100028

Schlotter D, Schack-Kirchner H, Hildebrand EE, Wilpert Kvon. Equivalence or complementarity of soil-solution extraction methods. J Plant Nutr Soil Sci. 2012;175:236-44. doi:10.1002/jpln.201000399

Sipos P, Nemeth T, Kis VK, Mohai I. Sorption of copper, zinc and lead on soil mineral phases. Chemosphere. 2008;73:461-9. doi:10.1016/j.chemosphere.2008.06.046

Smith DR, Owens PR, Leytem AB, Warne-Muende EA. Applications as impacted by time to first runoff event. Environ Pollut. 2007;147:131-7. doi:10.1016/j.envpol.2006.08.021

Souza ER, Melo HF, Almeida BG, Melo DVM. Comparação de métodos de extração da solução do solo. Rev Bras Eng Agric Amb. 2013;17:510-7. doi:10.1590/S1415-43662013000500007

Tedesco MJ, Gianello C, Anghinoni I, Bissani CA, Camargo FAO, Wiethölter S, editores. Manual de adubação e de calagem para os estados do Rio Grande do Sul e Santa Catarina. Porto Alegre: Sociedade Brasileira de Ciência do Solo; 2004.



Tedesco MJ, Volkweiss SJ, Bohnen H, Gianello C, Bissani CAA. Análise de solos, plantas e outros materiais. 2ª. ed. Porto Alegre: Universidade Federal do Rio Grande do Sul; 1995. (Boletim técnico de solo, 5).

Tuli A, Wei J-B, Shaw BD, Hopmans JW. *In Situ* monitoring of soil solution Nitrate: Proof of concept. Soil Sci Soc Am J. 2009;73:501-9. doi:10.2136/sssaj2008.0160

United States of Environmental Protection Agency – Usepa. National water quality inventory: report to congress, 2004 Reporting Cycle EPA 841-R-08-00; 2009.

Werle R, Garcia RA, Rosolem CA. Lixiviação de potássio em função da textura e da disponibilidade do nutriente no solo. Rev Bras Cienc Solo. 2008;32:2297-305. doi:10.1590/S0100-06832008000600009

Zhang JB, Zhu TB, Cai ZC, Qin SW, Müller CM. Effects of long-term repeated mineral and organic fertilizer applications on soil nitrogen transformations. Eur J Soil Sci. 2011;63:75-85. doi:10.1111/j.1365-2389.2011.01410.x