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Technical Note

PERFORMANCE OF MEHLICH-1 AND MEHLICH-3 EXTRACTORS IN QUANTIFYING PHOSPHORUS IN SOILS FERTILIZED WITH LIQUID SWINE MANURE

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ABSTRACT

Phosphorus is considered the most limiting essential macronutrient for plant production in soils of tropical climates. As the chemical and physical properties of the soil influence the availability of P to crops, the use of suitable extractants can increase the precision of the results obtained in soil analysis to enable the maximization of the efficiency of using phosphate fertilizers in agriculture. With this, the objective was to evaluate the capacity of the Mehlich-1 (M1) and Mehlich-3 (M3) extractors to determine the extractable P content in the soil in agricultural areas fertilized with liquid swine manure. The sampled sites were under the sandy loam (Sl) and sandy (S) textural class and were separated into four plots (repetitions) of 15 hectares each, where soil samples were collected in three depths 0.00-0.05; 0.05-0.10; and 0.10-0.20 m. For a depth of 0.20 m, the average levels of extractable P found in the evaluated textural classes were 34.83 (Sl) and 12.56 $mg \cdot dm^{-3}$ (S) for extractor M1; and 2.91 (Sl) and 0.81 $mg \cdot dm^{-3}$ (S) for extractor M3. It is clear that, although the Mehlich-1 solution has a greater capacity to extract the P content than the Mehlich-3 solution, both extractants can be used to extract the P content in sandy loam/sandy soils fertilized with liquid swine manure in the Cerrado region of Mato Grosso (Brazil).
Additional Keywords: Available phosphorus, extraction methods, pig slurry, sandy soils

RESUMEN

Uso de los extractores Mehlich-1 y Mehlich-3 en la cuantificación de fósforo en suelos fertilizados con estiércol líquido porcino

El fósforo se considera el macronutriente esencial más limitante para la producción vegetal en suelos de clima tropical. Dado que las propiedades químicas y físicas del suelo influyen en la disponibilidad de P para los cultivos, el uso de extractantes adecuados puede aumentar la precisión de los resultados obtenidos en el análisis del suelo, con el fin de permitir maximizar la eficiencia del uso de fertilizantes fosfatados en agricultura. Con esto, el objetivo fue evaluar la capacidad de los extractores Mehlich-1 (M1) y Mehlich-3 (M3) para determinar el contenido de P extraíble en el suelo en áreas agrícolas fertilizadas con estiércol líquido porcino. Los sitios muestreados estuvieron bajo la clase textural franco arenoso (Sl) y arenoso (S), y fueron separados en cuatro parcelas (repeticiones) de 15 hectáreas cada una, donde se recolectaron muestras de suelo en tres profundidades 0,00-0,05; 0,05-0,10; y 0,10-0,20 m. Para una profundidad de 0,20 m, los niveles promedio de P extraíble encontrados en las clases texturales evaluadas fueron 34,83 (Sl) y 12,56 $mg \cdot dm^{-3}$ (S) para el extractor M1; y 2,91 (Sl) y 0,81 $mg \cdot dm^{-3}$ (S) para el extractor M3. Es claro que, aunque la solución Mehlich-1 tiene mayor capacidad para extraer el contenido de P que la solución Mehlich-3, ambos extractores pueden usarse para extraer el contenido de P en suelos franco arenosos/arenosos fertilizados con estiércol líquido porcino en la región Cerrado de Mato Grosso (Brasil).
Palabras clave adicionales: Fósforo disponible, métodos de extracción, suelos arenosos

INTRODUCTION

Currently, it is estimated that the pig herd in Mato Grosso has approximately 3.16 million animals (ACRISMAT, 2024), generating around 9,919,240 $m^3 \cdot year^{-1}$ of waste (27,176 $m^3 \cdot day^{-1}$), considering that each pig produces, on average, 8.6 $kg \cdot day^{-1}$ (Oliveira, 1993). Due to this expansion of swine farming in the state, mainly in the confinement system, liquid swine manure

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(LSM) has been used as an organic fertilizer in agriculture to minimize the use of mineral fertilizer.

According to Perdomo (2001), a large amount of P, Cu and Zn ingested by animals via feed is excreted in the form of feces and urine, producing waste rich in nutrients. Thus, these residues can improve the chemical, physical and biological attributes of the soil with the supply of organic matter and nutrients to crops, and as long as they are applied appropriately, they do not pose risks of contamination and pollution of soil and water (Lourenzi et al., 2016). Therefore, analyzing the content of nutrients, such as phosphorus, in soils fertilized with LSM is essential to promote sustainable agricultural practices, ensure the efficient use of fertilizers, prevent environmental pollution and maintain soil health and quality.

The diversity of extractants used to extract P is an indication of the complexity of the element's dynamics in the soil (Larsen, 1967; Silva and Raij, 1999), as well as the difficulty of correlating the content extracted from the soil with what is effectively absorbed by the plant (Arruda et al., 2015). Therefore, using suitable extractants can increase the accuracy of the results obtained in soil analysis to minimize the adverse effects of inadequate recommendations for phosphate fertilizers and maximize the efficiency of using this resource in agriculture (Santos and Conceição, 2018).

The Mehlich-1 extractor (M1) has been widely used in routine laboratories in Brazil (Arruda et al., 2015; Santos and Conceição, 2018), being the predominant method, while the Mehlich-3 extractor (M3) has been proposed as a viable alternative to M1 (Mumbach et al., 2018; Reis et al., 2020). Both extractants act by acid dissolving the elements adsorbed to the colloids (Carneiro et al., 2023); in the case of M3, with subsequent complexation with a chelating agent (Andrade and Sobral, 2016). However, the efficiency of the extraction process of these acidic solutions is dependent on several characteristics and chemical and granulometric properties of the soil, such as pH, potential acidity, the presence of iron and aluminum oxides, clay and organic matter contents, fertilization management, the cultivation system adopted, and the soil-extractor relationship (Novais and Smyth, 1999).

The soils of the Brazilian Cerrado are considered acidic, deep, highly weathered (Instituto de Potassa e Fosfato, 1998) and have low levels of P available to plants (Garcia and Mendes, 2022). In addition, the physical and chemical properties of the soil can influence the availability of P and the efficiency of extractors used to determine its levels (Novais and Smyth, 1999). Therefore, the objective was to evaluate the capacity of the Mehlich-1 (M1) and Mehlich-3 (M3) extractors to determine the extractable P content in the soil in agricultural areas fertilized with LSM in the Cerrado region of Mato Grosso (Brazil).

MATERIALS AND METHODS

Soil sampling and characterization. The soils from the selected areas in the Mato Grosso municipalities of Lucas do Rio Verde (Sl) and Nova Mutum (S) (Brazil) were with classes sandy loam (Sl) and sandy (S) textures (Santos et al., 2005), respectively. Both areas were under the Cerrado biome, with surrounding vegetation of the Cerradão type (Ribeiro and Walter, 1998) and cultivated with forage grasses. The two municipalities were classified as having a tropical climate with a dry winter season – Aw, according to Köppen and Geiger (1928) (Aparecido et al., 2020).

To collect the soil samples, the agricultural areas fertigated with LSM stabilized in a biodigester were separated into four plots of 15 hectares each, with sampling carried out with a Dutch auger at five different and random points within each plot, at depths 0.00-0.05; 0.05-0.10; and 0.10-0.20 m, with four repetitions each, after 5 and 12 years of application respectively in S and Sl.

Subsequently, the samples were dried in a forced air circulation oven at 65 °C, crushed and sieved through a 2.0 mm mesh for chemical and granulometric characterization, according to the methodology described by Teixeira et al. (2017), whose results are presented in Table 1.

Table 1. Chemical characteristics of sandy loam (Sl) and sandy (S) texture soils in the Mato Grosso municipalities of Lucas do Rio Verde and Nova Mutum, respectively, in areas under application of liquid swine manure (LSM)

Soil attributes	------ Sandy loam (Sl) ------			--------- Sandy (S) ---------		
	Depth (m)					
	0.00-0.05	0.05-0.10	0.10-0.20	0.00-0.05	0.05-0.10	0.10-0.20
pH H_2O (1)	5.43	5.40	5.35	6.03	6.08	6.05
pH $CaCl_2$ (1)	4.43	4.38	4.23	4.98	4.88	4.85
Ca (2)	1.52	1.01	0.51	1.86	1.37	0.95
Mg (2)	0.33	0.20	0.13	0.63	0.42	0.28
Al (2)	0.11	0.19	0.34	0.02	0.03	0.03
H+Al (3)	5.03	4.66	4.26	2.89	2.72	2.56
Organic matter (4)	29.42	22.57	19.5	19.46	16.86	13.82
Clay (5)	156.38	147.9	156.37	64.77	64.72	64.76
Silt (5)	59.43	59.36	59.44	26.12	26.1	26.12
Sandy (5)	784.19	792.75	784.19	909.11	909.19	909.12

(1)by potentiometry; (2)in potassium chloride, by titration ($cmol_c \cdot dm^{-3}$); (3)in calcium acetate, by titration ($cmol_c \cdot dm^{-3}$); (4)dry way, on a weight basis, in muffle ($g \cdot kg^{-1}$); (5)granulometry by the pipette method ($g \cdot kg^{-1}$).

Determination of extractable P contents with Mehlich-1 (M1) and Mehlich-3 (M3). The extraction of P content with the Mehlich-1 extracting solution (H_2SO_4 0.0125 mol L^{-1} + HCl 0.05 mol L^{-1}) (Mehlich, 1978) was carried out according to the methodology described by Teixeira et al. (2017); and, in Mehlich-3 (CH_4COOH 0.2 mol L^{-1} + NH_4NO_3 0.25 mol L^{-1} + NH_4F 0.015 mol L^{-1} + HNO_3 0.013 mol L^{-1} + EDTA 0.001 mol L^{-1}) (Mehlich, 1984), according to the methodology described by Silva (2009).

The P content in the extracts was determined using a UV-Vis spectrophotometer (Micronal AJX-1000). The absorbance was measured at 660 nm (M1) and 880 nm (M3), with the P concentration in the extracts calculated using the equation proposed by Teixeira et al. (2017):

$$P = \frac{(L - b)}{a} \times d \times 10$$

Where: P is the concentration of extractable P (mg dm^{-3}); L is the sample reading in absorbance; a is the slope of the pattern line (intercept); b is the linear coefficient of the pattern line; d is the dilution factor of the Mehlich-1/Mehlich-3 extractor, if necessary dilute the extractor; value 10 is the factor that takes into account the soil: extractor dilution.

Data analysis

The results were submitted to the non-parametric Mann-Whitney test (p-value≤0.05), where the P contents extracted with Mehlich-1 (M1) and Mehlich-3 (M3) were compared for each textural class studied. Subsequently, the results were also subjected to Spearman correlation (p-value≤0.05), where the relationship between the rank coefficients of the P contents extracted with M1 and M3 was tested. The aforementioned statistical analyses were performed using the IBM SPSS Statistics software, version 22.0.

RESULTS

For the sandy loam (Sl) soil, extractor M1 extracted 12.8, 10.6 and 12.4 times more P than extractor M3 in layers of 0.00-0.05; 0.05-0.10; and 0.10-0.20 m, respectively. Higher P levels were also determined in the extracts obtained with M1 for sandy textured soils (S), these results being more expressive, as the M1 extracted 14.1, 16.1 and 18.9 times more P than the M3 extractor in layers of 0.00-0.05; 0.05-0.10; and 0.10-0.20 m, respectively (Table 2).

Table 2. Extractable phosphorus (P) levels in Mehlich-1 (M1) and Mehlich-3 (M3) stratified in depth in sandy loam (Sl) and sandy (S) texture soils in the Mato Grosso municipalities of Lucas do Rio Verde and Nova Mutum, respectively, in areas under liquid swine manure (LSM) application

Textural class	Depth (m)	Extractable P ($mg \cdot dm^{-3}$)		p-value$^{*(1)}$
		M1	M3	
Sandy loam (Sl)	0.00-0.05	52.00	4.06	0.029
	0.05-0.10	31.84	3.00	0.029
	0.10-0.20	20.66	1.66	0.029
	p-value$^{*(2)}$	0.211	0.174	-
Sandy (S)	0.00-0.05	17.71	1.26	0.029
	0.05-0.10	12.06	0.75	0.029
	0.10-0.20	7.92	0.42	0.029
	p-value$^{*(2)}$	0,246	0,292	-

*Mann-Whitney test, $^{(1)}$ For comparisons of extractors M1 and M3 in each layer; $^{(2)}$ For comparisons of depths 0.00-0.05, 0.05-0.10 and 0.10-0.20 m for each extractor studied (M1 and M3).

Therefore, as no differences were observed between depths 0.00-0.05, 0.05-0.10 and 0.10-0.20 m, only the average levels for the layer 0.0-0.20 m deep were considered, obtained through a simple arithmetic average of the stratified results found in the aforementioned layers (Table 2). As a result, the same behavior as previously identified was observed, where extractor M1 extracted more P than extractor M3 for both soils (Sl and S) (Figure 1).

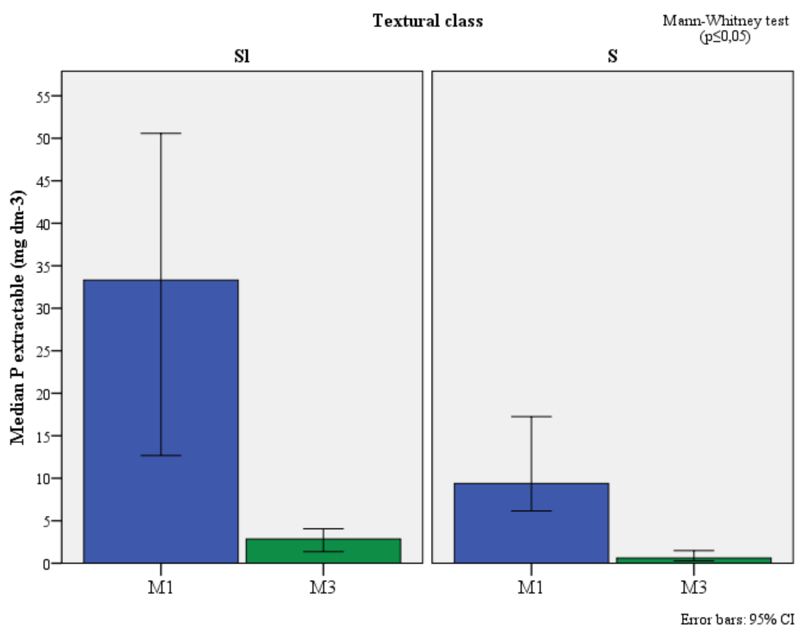

Figure 1. Extractable phosphorus (P) with Mehlich-1 (M1) and Mehlich-3 (M3) at 0.20 m depth in sandy loam (Sl) and sandy (S) texture soils in Mato Grosso municipalities from Lucas do Rio Verde and Nova Mutum, respectively, in areas under liquid swine manure (LSM) application. Error bars represent 95% confidence intervals

The average P contents found at 0.20 m depth for textures Sl and S were 34.83 and 12.56 $mg \cdot dm^{-3}$, respectively, for extractor M1, and 2.91 and 0.81 $mg \cdot dm^{-3}$, respectively, for the M3 extractor.

When the results were submitted to Spearman's correlation (p-value=0.029), a positive association of 0.909 was observed between the extractor solutions, indicating the existence of a high degree of association between extractors M1 and M3, even given the differences identified in their extractive capacities in both textural classes, Sl and S (Table 3).

Table 3. Spearman's correlation between the extractor solutions M1 and M3

Extractor							Coeff.	p-value
M1	52.0	31.8	20.6	17.7	12.0	7.92	0.909	0.029
M3	4.06	3.00	1.66	1.26	0.75	0.42		

DISCUSSION

The observed results did not reveal the existence of a concentration gradient of extractable P in depth in the areas evaluated (Table 2), supporting the results observed by Magalhães and Weber (2022) in conditions similar to ours. This result probably comes from the increase in microbial activity in the soil resulting from the addition of a highly labile residue that facilitates decomposition (Ceretta et al., 2003), combined with the tropical climate of the region and the sandy loam-sandy texture of the studied soil have catalyzed the LSM mineralization process, with the recycling of P contributed via waste, preventing the accumulation of the macronutrient in significant quantities in the first layers of soil (Primavesi, 2016) (Table 2).

As seen in Figure 1, the extractive capacity of extractor M1 was much greater than the extractive capacity of extractor M3 for the soils evaluated (Sl and S), as well as at all depths studied (Table 2). Typically, P is found in the soil under highly energetic bonds with organic and inorganic colloids (Pantano et al., 2016; Sasabuchi et al., 2023), especially bound to Fe and Al oxides (Mumbach et al., 2018). The availability of P is controlled, mainly, by the partition of its chemical fractions according to their lability in the soil (Larsen, 1967): labile in the form of P-H_2O and P-Al, and non-labile in the form of P-Fe and P-Ca (Santos and Conceição, 2018; Pereira et al., 2020). Furthermore, it is known that in soils with a more acidic pH, the P fixation process occurs, preferably, together with the Fe (P-Fe) and Al (P-Al) ions, and, in more alkaline soils, the precipitation, mainly with the Ca ion (P-Ca) (Medeiros et al., 2021). Therefore, soil pH is a determining attribute in the dynamics of the availability of this macronutrient for plants (Gatiboni et al., 2008; Rosa et al., 2018) since acidity influences the load balance of solid soil particles, consequently in the sorption process of this anion together with colloids (Vasconcelos et al., 2022).

In other words, extraction efficiency does not depend exclusively on the chemical nature of the extracting solution, but also on the chemical and granulometric properties of the soil (Nunes et al., 2021), as well as on the soil-extractor interaction, as both methods studied (M1 and M3) are sensitive to variations in soil clay and organic matter content (Mumbach et al., 2020). According to Pereira et al. (2020), in less weathered soils with a sandier texture, the extractive capacity of M1 is greater than that of M3 in the extraction of P when there is a predominance of forms of P linked to the Ca ion (P-Ca); the opposite occurs in highly weathered soils with a more clayey texture, with a greater extractive capacity of M3 compared to M1 (Mumbach et al., 2020). According to Mumbach et al. (2018), extractor M1 has its extractive capacity reduced in more clayey soils, in addition to the initial pH of the extractor solution (pH 1.2), which is quickly changed in the soil-extractor interaction (Nunes et al., 2021), in addition to the wear and tear that may eventually occur in the solution itself during the extraction process, as well as the occurrence of the P re-adsorption phenomenon with soil colloids during the resting time foreseen in the methodology itself (Mumbach et al., 2020). Unlike the M1 extractor, the M3 extractor, due to the presence of the fluoride ion and its initial pH (pH 2.5) (Nunes et al., 2021), is less affected by the variation in soil pH during the extraction process of the nutrient, as well as the assortative effects arising from the increase in the clay content of the soil (Mumbach et al., 2020). Therefore, it is believed that the results obtained in this study were influenced by the granulometry of the soils in the studied areas (Table 1), as well as the effects of the region's climate (Aw) on the rapid decomposition and mineralization of organic matter contributed via LSM.

However, even with the significant differences found between the extractable P contents with M1 and M3 for both soils studied (Figure 1), a high degree of association was observed between these extraction solutions M1 and M3, with a positive correlation of 90.9%, supporting the results of Kabala et al. (2018), Mumbach et al. (2018) and Pereira et al. (2020) who, when relating the extractive capacity of the same solutions (M1 and M3), observed the same behavior in different types of soil. In other words, extractants M1 and M3 can be used to extract the P content in the soil (Díaz et al., 2023), since both solutions have similar extraction principles (acid dissolution), with M1 being a weak acid and M3 is a strong acid with complexing agents (Simonete et al., 2015). Thus, additional studies are needed in soils equally fertilized with LSM and with different textural classes within the limits of the Cerrado of Mato Grosso to elucidate the extractive capacity of these methods (M1 and M3) in extracting P.

CONCLUSION

The extractive capacity of the Mehlich-1 solution is greater than the extractive capacity of the Mehlich-3 solution in extracting P content. However, both extractants can be used to extract P content in fertilized sandy loam/sandy soils with liquid swine manure in the Cerrado region of Mato Grosso (Brazil).

It is suggested that complementary studies be carried out on agricultural soils in a tropical climate under the application of liquid swine manure and with different textural classes for an effective understanding and calibration of the extractive capacity of Mehlich-1 and Mehlich-3 solutions in the extraction of extractable P content of the soil, as well as studies that correlate the available levels extracted by different extractors with the levels absorbed by plants.

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