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Phosphorus Mobility and Behavior in Soils Treated with Calcium, Ammonium, and Magnesium Phosphates

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The fertilizer industry has attempted to increase P mobility and lability after fertilizer application by using nonconventional phosphates or by including additives in the fertilizer formulation. We incubated granular monoammonium phosphate (MAP), sulfur-coated MAP, humic acid-coated MAP, triple superphosphate (TSP), ammonium potassium polyphosphate (AKPP), and ammonium magnesium phosphate (struvite) with soils from the United States and Brazil in Petri dishes for 56 d. We estimated P mobility by measuring P movement away from fertilizer granules and assessed P lability through sequential chemical fractionation of soil collected from the dishes. In addition, we monitored the change in soil pH with distance from fertilizer placed in the Petri dish. Soil pH changed in response to fertilizer additions as a function of initial soil pH. In fertilized soils, the soil pH response followed a quadratic function as the distance from the fertilizer placement site increased. Soil characteristics influenced P mobility, with mobility decreasing from the Hubbard (12% clay; pH 5.3), to Brazil (20% clay; pH 6.5), to Normania (22% clay; pH 5.5), and then Barnes (31% clay; pH 8.0) soil. The use of MAP-based fertilizers resulted in the greatest mobility, while struvite provided the lowest mobility. In contrast, struvite granules dissolved the least resulting in the highest labile P concentrations, due to direct extraction of fertilizer P from undissolved granules (average of 73% of applied P). Comparatively, TSP provided the lowest amount of labile P (average of 52% applied P). Sulfur and humic acid-coated MAP had no effect on P lability or mobility.

Abbreviations: AKPP, ammonium potassium polyphosphate; MAP, monoammonium phosphate; TSP, triple superphosphate.

Core Ideas

- **Humic acid coatings on monoammonium phosphate had no effect on P lability or mobility.**
- **Struvite provided the lesser P mobility among the fertilizers tested.**
- **There was greater P mobility in soils with high sand content and low initial pH.**

A significant fraction, sometimes more than half, of soluble P from fertilizers becomes tied up in the soil in forms that are not available for plant uptake in as few as 3 d after fertilizer application (Khasawneh et al., 1979; Pagliari et al., 2010; Rajput et al., 2014). The P mobility in soils is governed by soil and fertilizer characteristics, such as fertilizer granule formulations [e.g., calcium-based phosphates (Ca-P) and ammonium-based phosphates], soil organic matter content, clay content and type, soil cation exchange capacity, soil pH, initial P status, soil sorption strength, soil exchangeable Ca, Fe, and Al, and soil moisture content (Parfitt, 1979; Sample et al., 1979; Tunesi et al., 1999; Alvarez et al., 2004; Hettiarachchi et al., 2006; Schmitt et al., 2017). Therefore, selecting a particular P-containing fertilizer to improve P availability to plants presents a challenge to nutrient managers (Hedley and McLaughlin, 2005; Owen et al., 2015).

Fertilizer granule characteristics, environmental conditions, or a combination of both contribute to fertilizer use efficiency, described by the partial nutrient balance or the ratio of nutrient uptake to nutrient applied. In acidic to neutral soils, Al and Fe hydrous oxide concentrations are a key factor controlling the dissolution of

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secondary P-containing minerals that form after initial fertilizer dissolution. Conversely, calcareous tropical soils contain large amounts of Ca-bonded P (Buehler et al., 2002). In these soils, hydrated calcium phosphate ($CaHPO_4{\cdot}2H_2O$) forms around P-containing fertilizer as the granule dissolves (Lombi et al., 2006). Over time, the mineral structure shifts to either hydroxyapatite ($Ca_{10}(PO_4)_6(OH)_2$) under high soil P concentrations or octacalcium phosphate ($Ca_8H_2(PO_4)_6{\cdot}5H_2O$) under low soil P concentrations (Yang et al., 2012). In alkaline calcareous soils, exchangeable Ca is the main soil property controlling P sorption reactions (Tunesi et al., 1999).

Several approaches have been developed in an attempt to improve the partial nutrient balance by manipulating the composition of the fertilizer granule. Some of those approaches include adding elemental S (S^0) during fertilizer formation (Friesen, 1996), the addition of humic substances (Giovannini et al., 2013), and the use of lower solubility granules (Chandra et al., 2009; Rahman et al., 2014). Although a change in the chemical formulation and physical state of a fertilizer might improve its lability and mobility under one condition, it might not change under others and may actually decrease in some cases (McLaughlin et al., 2011). For example, Holloway et al. (2001) reported increased partial factor productivity when using fluid fertilizer in calcareous soils. In contrast, Montalvo et al. (2014) reported that although P diffusion rate increased as a result of fluid fertilizer addition, there were no changes in P lability.

Elemental S has also been used during the production of N and P containing fertilizer in an attempt to increase the partial nutrient balance (McLaughlin et al., 2011). The use of S^0 in phosphate fertilizer manufacturing is thought to cause a pH reduction around the fertilizer granule due to S^0 oxidation, which could improve the solubilization of Ca precipitated P and phosphate rock (Aria et al., 2010; Friesen, 1996). Humic substances also have been added to phosphate fertilizer granules as an attempt to increase the partial nutrient balance (Giovannini et al., 2013). Humic substances are thought to act as a chelating agent, which would complex divalent and trivalent cations such as Ca, Mg, Al, and Fe, reducing the ability of those cations to react with phosphate ions as the fertilizer granules dissolve (Giovannini et al., 2013). Struvite is an ammonium magnesium phosphate that forms from the precipitation of NH_4^+, Mg^{2+}, and PO_4^{3-} under alkaline conditions (Escudero et al., 2015). Struvite has been reported to have lower solubility than Ca-phosphate fertilizers and could increase the use efficiency of phosphate fertilizers if P is released when the plants need it (Speece, 1996; Chandra et al., 2009; Rahman et al., 2014). In contrast, the use of struvite as a source of P could also limit plant uptake if the release is slower than the plant requirement for P.

Table 1. Selected chemical and physical properties of the soils used in this study prior to the 56-d incubation period. Three soils were from Minnesota, USA: a Calcic Hapludoll (Barnes), Entic Hapludoll (Hubbard), and Aquic Hapludoll (Normania). The fourth was from Sao Paulo, Brazil: a Typic Kanhaplastult (Brazil).

Property†	Barnes	Brazil	Hubbard	Normania
Soil order	Mollisol	Ultisol	Mollisol	Mollisol
pH (water)	8.0	6.6	5.3	5.5
Clay g kg^{-1}	309	200	118	224
WHC, g kg^{-1}	279	200	69	381
OM, g kg^{-1}	44	28	13	55
$CaCO_3$, g kg^{-1}	200	ND‡	ND	ND
Ca, mg kg^{-1}	3815	451	580	2414
Mg, mg kg^{-1}	526	137	95	608
Fe, mg kg^{-1}	48	43	95	231
Al, mg kg^{-1}	32	702	716	703
b, mg kg^{-1}	384	350	200	448
k	0.254	0.273	0.139	0.098

† WHC, water holding capacity; OM, soil organic matter determined by loss on ignition (Combs and Nathan, 1998); $CaCO_3$, calcium carbonate content (Bouyoucos, 1962); Ca, Mg, Fe, Al are Mehlich-3 extractable Ca, Mg, Fe and Al (Warncke and Brown, 1998) determined by ICP–OES; b is the P-sorption maximum; k is the P-sorption strength.

‡ ND, not detectable.

Fertilizer formulations designed to increase P availability to plants have become increasingly available in the United States and Brazil with little information available on their performance. The intent of this paper was to provide information regarding P movement and lability from different fertilizer formulations when applied to soils with differing properties collected in the United States (three soil sources) and Brazil (one soil source).

MATERIALS AND METHODS

Soil Samples Collection and Chemical Characterization

We collected samples from four soils with contrasting chemical, physical, and biological properties. Three were from Minnesota, USA: a Calcic Hapludoll (Barnes), Entic Hapludoll (Hubbard), and Aquic Hapludoll (Normania). The fourth was from Sao Paulo, Brazil: a Typic Kanhaplastult (Brazil). Soil samples were collected from the 0- to 15-cm depth, ground to pass through a 2-mm sieve, air-dried, and stored at room temperature (22°C) until analysis. Selected soil properties are listed in Table 1.

Phosphorus Adsorption Curve

Phosphorus adsorption curves were constructed to determine the sorption coefficients, maximum sorption (b) and sorption strength (k), according to Nair et al. (1984). Briefly, 1.0 g of dry soil was equilibrated with 25 mL of KH_2PO_4 in a 0.01 mol L^{-1} $CaCl_2$ solution with increasing P concentrations ranging from 0 to 100 mg P kg^{-1}. The soil suspension was shaken for 16 h, centrifuged for 5 min at 3562 g and the supernatant was transferred to a clean centrifuge tube. Phosphorus concentration in the supernatant was determined by inductively coupled plasma–optical emission spectrometer (ICP–OES; PerkinElmer, Optima 8x00, Norwalk, CT). The Langmuir adsorption model was used to fit the data and model the sorption parameters b and k, according to Eq. [1]:

$$Q=(bkC)/(1+kC) \qquad [1]$$

where *b* is the maximum sorption, *k* is the sorption strength, and *C* is the equilibrium P concentration. The *b* and *k* values obtained for each soil are reported in Table 1. Schmitt et al. (2017) reported a much more detailed relationship between *b* and *k* and soil properties for the soils used in this study in addition to several other soils from the United States and Brazil.

Fertilizer Characterization

All fertilizers used in this study were solid granular formulations: (1) MAP, with 110 g N and 220 g P per kilogram; (2) sulfur-coated MAP, with 90 g N, 190 g P, and 160 g S per kilogram; (3) humic acid-coated MAP, with 100 g N, 220 g P, and 3 g humic acid per kilogram; (4) triple super phosphate (TSP), with 200 g P per kilogram; (5) ammonium potassium polyphosphate (AKPP), with 210 g N, 30 g P, and 120 g K per kilogram; and (6) struvite, with 60 g N, 120 g P, and 100 g Mg per kilogram. We determined the P concentration in each product according to the methods of Yang et al. (2002). The concentrations determined in our laboratory matched those reported by the fertilizer manufacturer in all cases. We determined struvite solubility by measuring the pH where struvite granules dissolved completely after reacting with buffered sodium acetate solutions (1:50 solid/solution ratio) with an initial pH of 2, 3, 4, 5, 6, 7, and 8.

Phosphorus Mobility Study

Phosphorus mobility from fertilizer granules was evaluated in plastic Petri dishes (8.6 cm diameter and 1.1 cm tall) containing 85 g of dry soil replicated four times for each soil and fertilizer combination. This methodology has been previously used to assess nutrient mobility and reactions in the area surrounding the fertilizer granule (Hettiarachchi et al., 2006; Montalvo et al., 2014). The soil was moistened with deionized water to bring soil water content up to 60% of the soil water holding capacity (Priha and Smolander, 1997); the Petri dishes were then closed and sealed with plastic film and incubated for 24 h. The Petri dishes were opened 24 h after the deionized water addition, and a small opening was created in the soil at the center of the Petri dish for fertilizer placement. A fertilizer granule suppling 8.8 mg P dish^{-1}, was placed in the opening at 3 mm below the soil surface, and the fertilizer was covered with soil. Several hundred granules had to be individually weighed until granules of the exact same weight were found to use in each replicate.

Each Petri dish received a single fertilizer granule except for the struvite treatment. The struvite granules were too small, making it impossible to find the required number of single granules with the mass to provide the required amount of P (8.8 mg P). Therefore, three struvite granules were placed very close to each other. After adding the fertilizer granules, the Petri dishes were sealed with plastic film, wrapped in aluminum foil (to reduce the effect of light on biochemical reactions), and incubated at 23 ± 2°C for 56 d. One no-P fertilizer control (replicated four times) was also incubated for each soil. At the end of the incubation period we collected the soil from each Petri dish as four separate concentric rings using plastic cylinders with four different radii. The radii of the four concentric sections (labeled 1 to 4) measured 7.75, 13.5, 25.5, and 43 mm, respectively. The undissolved fertilizer granule that was present at the end of the incubation period was collected with section one. Each section was ground, sieved (<2 mm), and air-dried individually by Petri dish.

The amount of total P in the soil from each concentric section was determined using a $H_2SO_4 + H_2O_2$ acid digestion method (Brookes and Powlson, 1981). In addition, we determined P fractions for each concentric section by sequentially extracting a 0.5-g subsample with an anion exchange resin, then 0.5 mol L^{-1} $NaHCO_3$, then 0.1 mol L^{-1} NaOH, and then 1.0 mol L^{-1} HCl (Condron et al., 1985). The inorganic P extracted in the resin, 0.5 mol L^{-1} $NaHCO_3$, and 1.0 mol L^{-1} HCl was determined colorimetrically by the method of Murphy and Riley (1962). The inorganic P extracted in 0.1 mol L^{-1} NaOH was determined using the colorimetric method described by He and Honeycutt (2005). The total P in each fraction, excluding the resin fraction, was determined directly by ICP–OES according to Do Nascimento et al. (2015). For clarity, inorganic P is the P determined by colorimetric methods, and total P is the P determined by ICP–OES. Organic P in each fraction was calculated as the difference between the total and inorganic P of each sample, that is, $P_o = P_t - P_i$. It was observed that in this study the amounts of P_o extracted in the fertilizer treated soils were very close to those observed for the control soils, suggesting no changes in the organic P pool. Finally, soil pH was determined in a slurry of 1:5 soil/deionized water for each soil sample by section (Kabala et al., 2016).

The percentage of fertilizer-P extracted in each fraction for each concentric section ($\%P_fS_i$) was calculated as:

$$\%P_fS_i = \frac{(P_{1f}S_i - P_{0f}S_i)W_i}{\sum_{i=1}^{4}(P_fS_i)W_i} \qquad [2]$$

where *f* denotes each sequential fraction (that is, extraction by resin, 0.5 mol L^{-1} $NaHCO_3$, 0.1 mol L^{-1} NaOH, and 1.0 mol L^{-1} HCl); *i* denotes each concentric soil section (numbered 1 to 4); $P_{1f}S_i$ (mg kg^{-1}) is the concentration of fertilizer-P for each sequential fraction in each concentric section from the Petri dish where P fertilizer was added; $P_{0f}S_i$ (mg kg^{-1}) is the concentration of fertilizer-P for each sequential fraction in each section from the Petri dish that had no P fertilizer added (control); and W_i (kg), is the soil mass of concentric section *i* (Lombi et al., 2005).

Statistical Analysis

We evaluated the main effects and all possible interactions among fertilizer source, soil tested, and distance from the fertilizer granule on the amount of inorganic P, organic P, and total P recovered through multivariate analysis of variance combined with repeated measures analyses using generalized linear mixed models with the GLIMMIX Procedure in SAS 9.3 (SAS Institute, 2010; Littell et al., 2006). The model held replication as a random effect, while considering all other factors fixed effects. We treated distance from the fertilizer granule as a repeated variable. We selected the covariance model with the lowest

Akaike information criteria (AIC) that is capable of handling unequal spacing in sampling (uneven sampling distances). Mean comparison of significant interactions or main effects ($P \leq 0.05$) was performed using Fisher's least significance difference test (LSD). When the soil factor was significant, alone or in interactions, the results were separated by soil to facilitate the visualization of the results for each soil. Linear and nonlinear multiple regression analyses were used to evaluate the effects of fertilizer, soil, soil section, or their interactions on soil pH using R (R Development Core Team, 2007). All statistical analyses were performed on replicate data (Table 2).

RESULTS AND DISCUSSION

Phosphorus Mobility

The MAP-based fertilizers (sulfur-coated MAP, humic acid-coated MAP, and MAP) had the highest total proportion of fertilizer-P recovered from outside the first concentric section (0–7.75 cm), indicating greater P mobility with these fertilizers than with AKPP, struvite, or TSP (Fig. 1). The amount of P found in the first section did not differ statistically among the three MAP-based fertilizers in the Barnes or Brazil soils, and the amount retained in the first section averaged 73% for the Barnes and 47% for the Brazil across all three MAP fertilizers. In contrast, the application of humic acid-coated MAP resulted in more P remaining in the first concentric section than MAP in the Hubbard and Normania soils. In the Hubbard soil, 41% of the P from humic acid-coated MAP remained in the first section compared with 34% of the MAP applied P. Similarly, in the Normania soil 67% of the applied P from humic acid-coated MAP applied was retained in the first section compared with 57% with MAP. The application of sulfur-coated MAP did not result in significantly different P mobility than the humic acid-coated MAP or MAP sources (Fig. 1). The lower P mobility observed for humic acid-coated MAP compared with MAP may be a result of chemical reactions among phosphate ions, Fe, and humic acids (Gerke and Hermann, 1992) since the amount of Fe found in the Hubbard and Normania soils was higher than in the Barnes and Brazil soils (Table 1).

Among the MAP-based fertilizers, sulfur-coated MAP had the highest mobility as greater amounts of P were found in the fourth section compared with humic acid-coated MAP and MAP in the Brazil and Hubbard soils (Fig. 1). Although sulfur-coated MAP has high concentrations of S^0 (160 g S kg^{-1}), the effects of S^0 on soil pH were not clear during this incubation study (Fig. 2; Table 3). It is possible that the lack of a measureable change in pH with sulfur-coated MAP was due to the short incubation time and minimal S^0 oxidation and release of H^+ into solution. Germida and Janzen (1993) reported that S^0 oxidation in soils depends in numerous factors, including time, soil biology, and surface area of S^0 as the most important factors. Also, it is possible that the anaerobic conditions experienced during the study hindered S^0 oxidation, which is typically an oxygen consuming and biologically mediated process (Nevell and Wainwright, 1987; Suzuki et al., 1999).

In the Hubbard soil, all the fertilizer formulations increased the soil pH above the control except for TSP. The observed pH increases may have occurred because when $H_2PO_4^-$ is added to soils with pH below 7.2 there is a tendency for the phosphate ion to remove H^+ from solution leading to an increase in soil pH (Schachtman et al., 1998; Cerozi and Fitzsimmons, 2016). The TSP was likely able to buffer the pH in the soil surrounding the fertilizer granule, probably because of its chemical properties. Furthermore, the low buffer capacity of Hubbard soil highlighted the differences between TSP and other fertilizers. In the Barnes soil, it was observed that the soil pH was depressed by fertilizer addition close to the fertilizer granule. In the Brazil soil, TSP and AKPP depressed pH near the granule, but, with TSP, it continued to decline as we moved further away from the granule. Struvite raised pH near the granule then went back to control levels as we moved away from the granule. In contrast to the other soils, the Normania soil seemed to be well buffered against pH changes except with struvite application. Generally, struvite raised the pH near the granule in all the acidic soils (Fig. 2).

Table 2. Summary of statistical analysis (probability levels) for total P, as well as inorganic and organic P recovered into the different pools of sequentially extracted samples as function of soil series (Soil), fertilizer source (Source), and soil section (Section, only for total P) following a 56-d incubation. Phosphorus was applied as a single fertilizer granule (three granules were used for struvite) to provide 8.8 mg P kg^{-1} soil.

		Inorganic P			Organic P		
Effect	Total P†	Labile‡	NaOH§	HCl¶	Labile	NaOH	HCl
	%	mg P dish^{-1}					
Soil	0.95	0.01	0.01	0.01	0.02	0.01	0.01
Source	0.98	0.01	0.01	0.01	0.01	0.01	0.01
Section	0.01	NA††	NA	NA	NA	NA	NA
Soil × Source	0.99	0.01	0.01	0.01	0.01	0.01	0.01
Soil × Section	0.01	NA	NA	NA	NA	NA	NA
Source × Section	0.01	NA	NA	NA	NA	NA	NA
Soil × Source × Section	0.01	NA	NA	NA	NA	NA	NA

† Total P, the sum of the total P extracted in each of the four fractions during the sequential fractionation.

‡ Labile-P, the sum of P extracted by anion exchange resin and 0.5 mol L^{-1} $NaHCO_3$.

§ NaOH, P extracted by 0.1 mol L^{-1} NaOH.

¶ HCl, P extracted by 1 mol L^{-1} HCl.

†† NA, not applicable as these fractions were summed over all four sections.

For all fertilizers applied to the Barnes soil, more than 70% of the fertilizer-P was found in the first section (Fig. 1). The results for the P mobility in the Barnes soil seems to be related to the changes observed in soil pH (Fig. 2; Table 3). In addition, it is possible that the high levels of clay content and extractable Ca may have limited P movement in Barnes soil (Table 1). The Barnes soil had the highest clay content among all soils and thus potentially more sorption sites where P could bind prior to moving further away from the fertilizer granule, as well as the second highest sorption maximum and sorption strength among the four soils (Table 1).

The TSP was the second most mobile P source, followed by AKPP, while P from struvite had the lowest mobility. However, in the Hubbard soil AKPP and TSP showed statistically similar amounts of fertilizer-P in the first section with 58 and 53%, respectively. We also found greater amounts of fertilizer-P in the third and fourth sections for TSP compared with AKPP (Fig. 1). The AKPP source also showed similar amounts of P in the second section compared with the TSP source for all soils except the Barnes soil (Fig. 1). Khasawneh et al. (1979) compared P movement in a sandy clay soil and reported that P mobility 1 wk after fertilizer placement was greater with diammonium

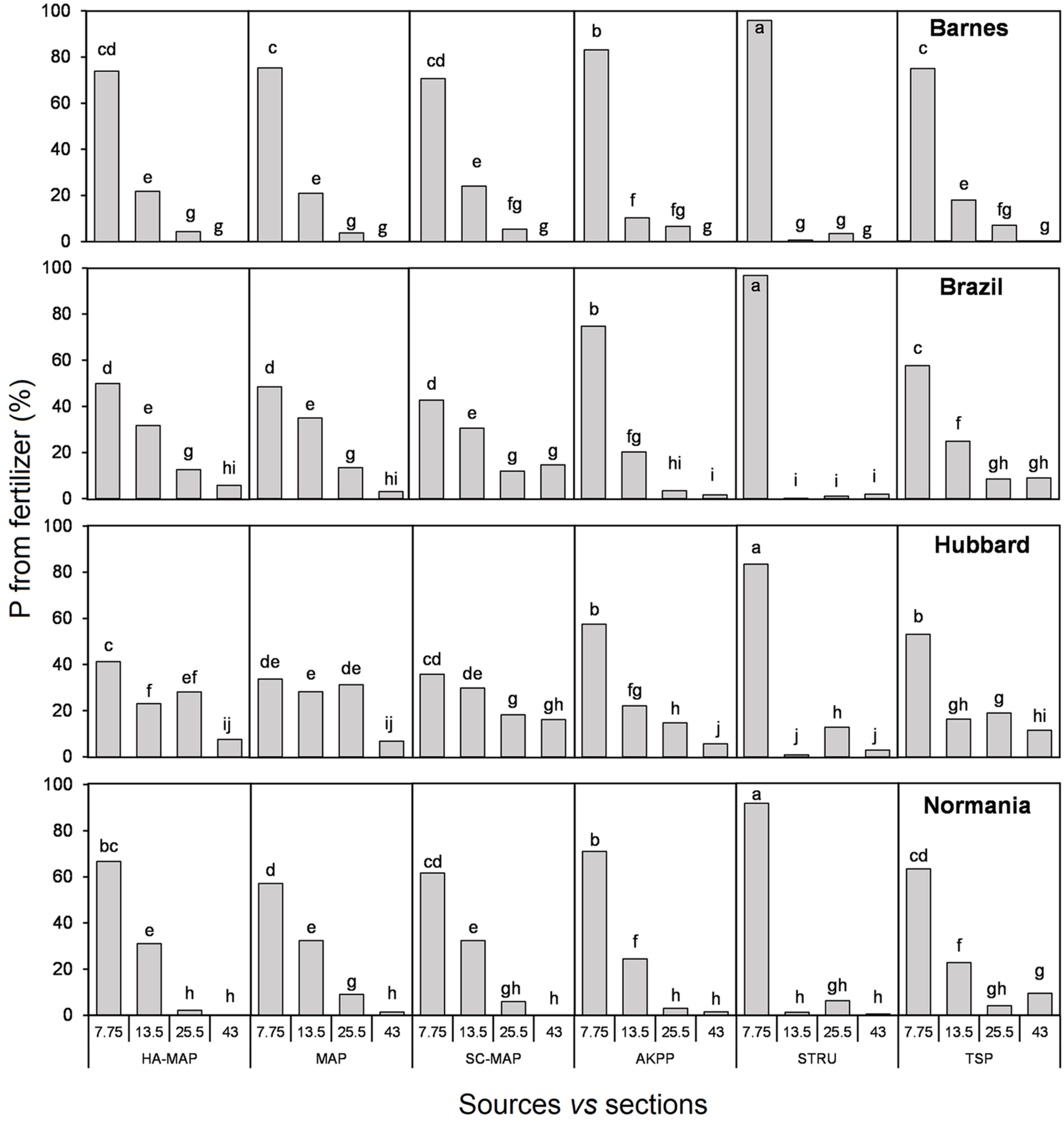

Fig. 1. Percentage of fertilizer-sourced P (mg P applied) recovered from samples collected in four concentric soil sections (radii of 0–7.75, 7.75–13.5, 13.5–25.5, and 25.5–43 mm) following a 56-d incubation. Phosphorus was applied as a single fertilizer granule (three granules were used for struvite) to provide 8.8 mg P per Petri dish. Bars followed by different letter for each soil are significantly different (*P* < 0.05).

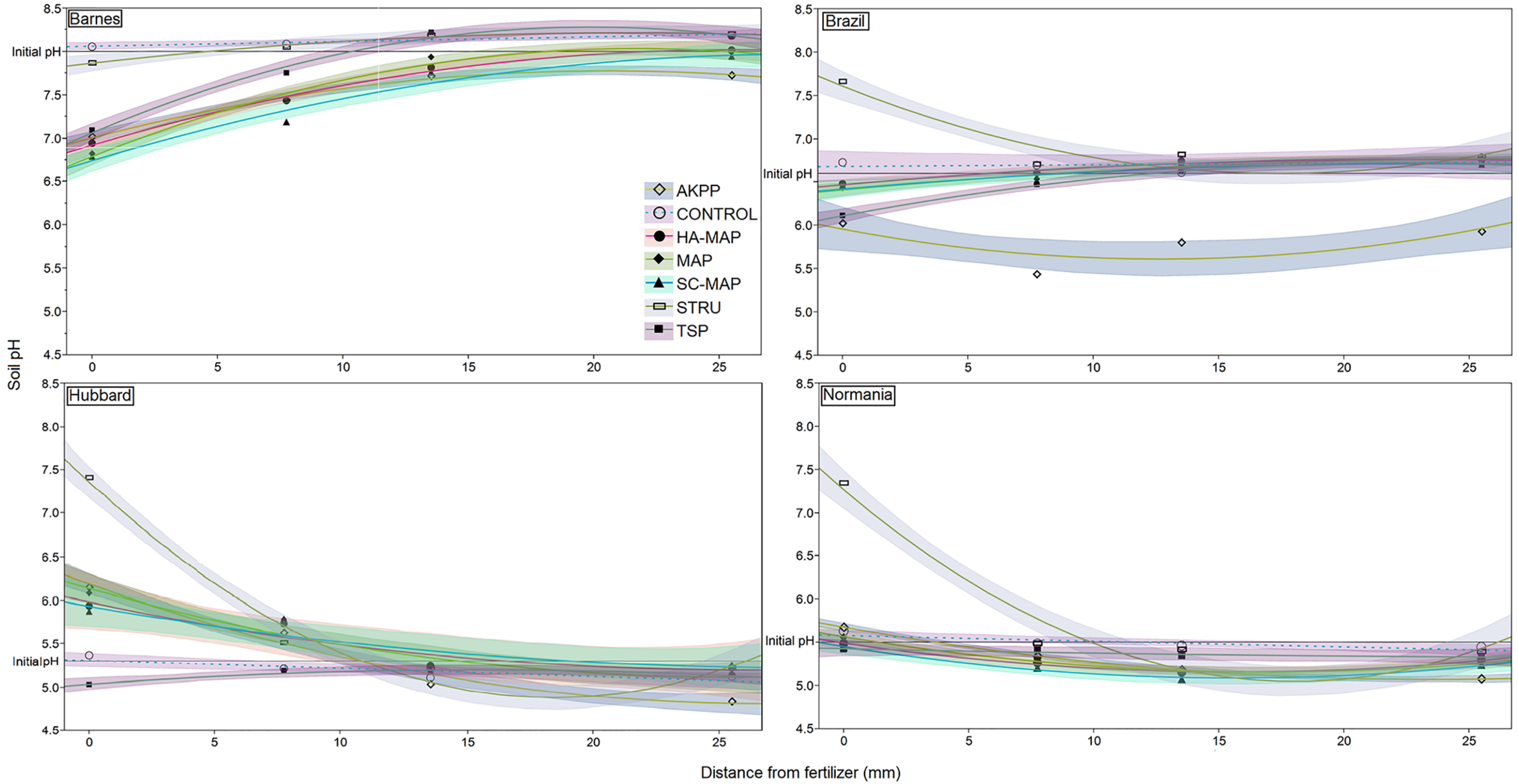

Fig. 2. Soil pH as function of soil series, fertilizer source, and soil section following a 56-d incubation. The shaded region around the line is the confidence interval of the fit (linear for control and quadratic for the others).

phosphate than with ammonium polyphosphate. Montalvo et al. (2014) investigated P behavior from liquid ammonium polyphosphate and MAP in six calcareous and non-calcareous soils and reported differences in P dynamics that varied with soil and fertilizer properties. Once in the soil, polyphosphate tends to precipitate in mineral forms that are less soluble than NH_4^+ polyphosphate, which hinders P movement from the fertilizer placement area (Khasawneh et al., 1979). When solid fertilizer is applied, P mobility in the soil surrounding the granule is restricted because cations such as K, Ca, Mg, and Al move toward the granule by mass flow. The large amounts of cations moving toward the fertilizer granule tend to increase P precipitation as soon as the granules start to dissolve (Hettiarachchi et al., 2006). In the present study, AKPP mobility was reduced when the initial soil pH was above 5.6 (Fig. 1 and 2). It is possible that the high concentration of acidity in low pH soils provides optimum conditions for AKPP dissolution leading to better P mobility compared with high pH soils. McBeath et al. (2007) reported that when the pH of the soils used in the study shifted from 6.4 to 5.8, the hydrolysis of polyphosphate to orthophosphate increased by about 10% at 25°C.

Phosphorus mobility was more restricted with struvite than with any other fertilizer source. On average, over 90% of fertilizer-P remained in the first section independent of soil when struvite was used. Struvite solubility was so limited that even after 56 d of incubation the struvite granules remained intact, and as a result were ground with the soil collected in the first section. It is possible that struvite impacted soil pH surrounding the granules; however, the results we observed were more likely a reflection of the struvite pH than the effect of struvite on the soil pH. Struvite has six water molecules in its chemical structure ($MgNH_4PO_4{\cdot}6H_2O$); as a result, it is possible that this molecule has a very low hygroscopicity and water flow carrying cations toward the fertilizer granule may not be significant. The struvite is sparingly soluble under neutral and alkaline conditions but highly soluble in acidic environments. Struvite is 100% soluble in solutions with pH below 4.0 and relatively insoluble in solutions with pH above 6.0 (data not shown). Le Corre et al. (2009) have reported that the solubility of struvite granules was 0.18 g L^{-1} in water, 0.33 g L^{-1} in 0.001 mol L^{-1} HCl, and 1.78 g L^{-1} in 0.01 mol L^{-1} HCl. The low solubility for struvite is the primary reason for the limited P mobility observed in this

Table 3. Summary of regression coefficients for soil pH (dependent variable) as function of soil series, fertilizer source, and soil section (independent variable) following a 56-d incubation. Phosphorus was applied as a single fertilizer granule (three granules were used for struvite) to provide 8.8 mg P kg^{-1} soil. Three soils were from Minnesota, USA: a Calcic Hapludoll (Barnes), Entic Hapludoll (Hubbard), and Aquic Hapludoll (Normania). The fourth was from Sao Paulo, Brazil: a Typic Kanhaplastult (Brazil).

	Barnes		Brazil		Hubbard		Normania	
Source†	Coefficient	*Pr* > | *t* |‡	Coefficient	*Pr* > | *t* |	Coefficient	*Pr* > | *t* |	Coefficient	*Pr* > | *t* |
				Intercept				
Control	8.0 a§	0.01	6.7 b	0.01	5.4 d	0.01	5.6 b	0.01
HA-MAP	6.9 cd	0.01	6.5 c	0.01	6.0 c	0.01	5.5 c	0.01
MAP	6.8 d	0.01	6.4 c	0.01	6.1 b	0.01	5.6 bc	0.01
SC-MAP	6.8 e	0.01	6.4 c	0.01	5.9 c	0.01	5.5 c	0.01
AKPP	7.0 c	0.01	6.0 e	0.01	6.2 b	0.01	5.7 b	0.01
Struvite	7.9 b	0.01	7.6 a	0.01	7.4 a	0.01	7.3 a	0.01
TSP	7.1 c	0.01	6.1 d	0.01	5.2 d	0.01	5.5 c	0.01
				Linear term				
Control	0.012 d	0.30	–0.017 c	0.16	–0.028 b	0.07	–0.020 ab	0.10
HA-MAP	0.085 b	0.01	0.026 b	0.03	–0.061 c	0.01	–0.042 bc	0.01
MAP	0.114 a	0.01	0.026 b	0.03	–0.080 d	0.01	–0.048 c	0.01
SC-MAP	0.087 b	0.01	0.025 b	0.04	–0.050 c	0.01	–0.046 bc	0.01
AKPP	0.075 b	0.01	–0.054 d	0.01	–0.103 e	0.01	–0.051 c	0.01
Struvite	0.033 c	0.01	–0.115 d	0.01	–0.268 f	0.01	–0.246 d	0.01
TSP	0.123 a	0.01	0.055 a	0.01	0.021 a	0.08	–0.006 a	0.61
				Quadratic term				
Control	–0.0003 d	0.55	0.0007 c	0.10	0.0007 c	0.10	0.0005 c	0.25
HA-MAP	–0.0017 bc	0.01	–0.0006 c	0.19	0.0012 bc	0.01	0.0014 b	0.01
MAP	–0.0026 ab	0.01	–0.0005 c	0.28	0.0016 bc	0.01	0.0014 b	0.01
SC-MAP	–0.0015 c	0.01	–0.0005 c	0.22	0.0009 c	0.05	0.0015 b	0.01
AKPP	–0.0018 bc	0.01	0.0021 b	0.01	0.0019 b	0.01	0.0011 b	0.01
Struvite	–0.0008 cd	0.08	0.0033 a	0.01	0.0073 a	0.01	0.0068 a	0.01
TSP	–0.0031 a	0.01	–0.0012 d	0.01	–0.0007 d	0.12	0.0001 c	0.84

† HA-MAP, humic acid-coated monoammonium phosphate; MAP, monoammonium phosphate; SC-MAP, sulfur-coated monoammonium phosphate; AKPP, ammonium and potassium polyphosphate; Struvite, magnesium and ammonium phosphate; TSP, triple superphosphate.

‡ Probability value for each parameter per fertilizer source; significant parameters were considered as $Pr < 0.05$.

§ Means followed by different letters for each parameter in each soil are significantly different ($P < 0.05$); The LSD = 0.13 for the intercept; The LSD = 0.024 for the linear term; The LSD = 0.001 for the quadratic term.

study as the initial soil pH values ranged from 5.5 (Normania) to 8.0 (Barnes) (Table 1).

Struvite, TSP, and AKPP altered the soil pH the most in the first section likely due to the lower P mobility observed in those fertilizers (Fig. 1 and 2). After the incubation study was completed, the soil and any fertilizer remaining in the first section were ground together. Struvite is an alkaline fertilizer and since it did not completely dissolve during the incubation study, it likely caused the soil pH in the first section to be close to the pH of the original granules. Changes in soil pH in the AKPP treatment may have been due to the high N content (210 g N kg^{-1}) of this fertilizer. The amount of N in AKPP was 210 g N kg^{-1}, which is higher than that in MAP-based fertilizers (110 g N kg^{-1}). Sample et al. (1980) attributed the pH changes of the soil surrounding the fertilizer granules to the inherent acidic (TSP and MAP) and alkaline (diammonium phosphate) characteristics of each fertilizer used in their study. In addition, H$^+$ displacement from the soil cation exchange capacity by cations introduced with the fertilizer can also cause changes in the soil pH (Montalvo et al., 2014).

Table 4. Amount of fertilizer-sourced P (mg P applied) recovered into the different pools of sequentially extracted samples collected following a 56-d incubation period. Phosphorus was applied as a single fertilizer granule (three granules were used for struvite) to provide 8.8 mg P kg^{-1} soil. Three soils were from Minnesota, USA: a Calcic Hapludoll (Barnes), Entic Hapludoll (Hubbard), and Aquic Hapludoll (Normania). The fourth was from Sao Paulo, Brazil: a Typic Kanhaplastult (Brazil).

Source†	Labile‡	NaOH§	HCl¶
	Barnes		
HA-MAP	5.0 cd††	1.5 de	0.5 d
MAP	5.0 cd	1.5 de	0. 6 d
SC-MAP	6.0 bc	1.7 cd	0.7d
AKPP	4.9 d	2.8 ab	0.9 bc
Struvite	6.0 bc	2.8 ab	0.3 de
TSP	4.9 d	1.9 cd	0.9 c
	Brazil		
HA-MAP	4.1 ef	2.1 c	0.1 d
MAP	4.7 de	1.7 cd	0.1 d
SC-MAP	4.7 de	2.1 bc	0.2 d
AKPP	4.4 ef	2.3 b	1.2 ab
Struvite	6.4 b	1.8 cd	0.0 e
TSP	3.8 f	2.1 c	0.4 d
	Hubbard		
HA-MAP	5.3 c	2.4 b	0.4 d
MAP	4.7 e	2.0 cd	0.6 d
SC-MAP	5.9 bc	2.5 ab	0.4 d
AKPP	4.6 e	1.9 cd	1.5 a
Struvite	5.8 bc	1.0 e	0.1 e
TSP	4.9 d	2.7 ab	0.9 bc
	Normania		
HA-MAP	4.1 ef	2.9 a	0.8 cd
MAP	4.6 e	2.9 a	0.5 d
SC-MAP	4.8 d	3.1 a	0.9 bc
AKPP	5.7 c	2.7 ab	1.0 b
Struvite	7.7 a	1.3 e	0.4 d
TSP	4.6 e	3.1 a	0.7 cd

† HA-MAP, humic acid-coated monoammonium phosphate; MAP, monoammonium phosphate; SC-MAP, sulfur-coated monoammonium phosphate; AKPP, ammonium and potassium polyphosphate; Struvite, magnesium and ammonium phosphate; TSP, triple superphosphate.

‡ Labile-P, the sum of P extracted by anion exchange resin and 0.5 mol L^{-1} $NaHCO_3$.

§ NaOH, P extracted by 0.1 mol L^{-1} NaOH.

¶ HCl, P extracted by 1 mol L^{-1} HCl.

†† Means followed by different letter within the column for each fraction are significantly different ($P < 0.05$).

Phosphorus Source on P Lability

The different sources of fertilizer had a significant effect on the distribution of P between the labile and non-labile pools (Table 2). In this study, labile P is defined as the sum of resin and $NaHCO_3$ extractable P, whereas the two non-labile P pools where defined as NaOH and HCl extractable P. For each of the three pools, we summed the P extracted in that pool over all four sections. The actual amounts of P_o detectable in the soils that received fertilizer were very close to the levels observed in the control soil (in the range of ± 0.05 mg kg^{-1} when detected). In most cases, we attributed those levels to errors associated with the determination procedure as found by several researchers (Do Nascimento et al. (2015); Daroub et al., 2000; Buehler et al., 2002; Negassa and Leinweber, 2009).

The amount of P recovered in the labile pool varied by soil and P source (Table 4). In the Barnes soil, struvite provided the highest concentration of labile P, while TSP, humic acid-coated MAP, and MAP provided the lowest (Table 4). Similar to the Barnes soil, struvite most increased labile P in the Brazil soil, but TSP provided the least labile P; the other P sources fell between these. In the Hubbard soil, struvite did not separate from the other P sources; instead struvite, humic acid-coated MAP, and sulfur-coated MAP increased the labile P concentration more than MAP and AKPP, which were statistically similar, and TSP, which produced statistically lower labile P concentrations than the other sources. For the Normania soil, struvite and sulfur-coated MAP had the highest levels of labile P while AKPP and TSP had lowest labile P (Table 4). Although struvite consistently produced the highest concentrations of labile P across the soils used in the study, these results can be misleading. The insolubility of struvite in these soils led to the intact granule being dissolved during the extraction processes. In contrast, AKPP and TSP tended to have the lowest P concentrations in the labile pools; while also having limited mobility compared with the humic acid-coated MAP and sulfur-coated MAP (Fig. 1). It is possible that precipitation reactions limited both mobility and lability of the AKPP and TSP sources. Phosphorus precipitation is expected when AKPP is applied to soils, especially in calcareous soils with high amounts of Ca and Mg like the Barnes soil (Sample et al., 1979). In calcareous soils, the dominant reaction around the fertilizer granule is precipitation as dicalcium phosphate dihydrate ($CaHPO_4{\cdot}2H_2O$) by surface rearrangement of amorphous phosphate into phosphate heteronuclei (Tunesi et al., 1999; Yang et al., 2012). While in non-calcareous soils P adsorption on the surface of Fe and Al hydroxides is likely to prevail (McLaughlin et al., 2011).

Sodium hydroxide extracts P that is known to be bound to Fe and Al oxide and hydroxide and has been labeled as moderately labile P (Negassa and Leinweber, 2009; McLaughlin et al., 2011). Therefore, soils with high pH, such as the Barnes soil, would be expected to have very little inorganic P extractable in the NaOH fraction. Not surprisingly, when the MAP-based fertilizers were applied the concentration of NaOH-extractable P in the Barnes soil was the lowest among all soils and P sources (Table 4). It is possible that most of the differences in the amount of P recovered in the NaOH fraction were related to the fertilizer effects on soil pH.

The relationship between the initial and the final soil pH surrounding the fertilizer granules is important to determine phosphate species present in soil solution and how they can affect the solution pH. Phosphate ions have three protonation constant (pKa) values at pH 2.1, 7.2, and 12.6. For example, at pH 7.2 the concentration of $H_2PO_4^-$ is in equilibrium with the concentration of HPO_4^{2-} (Schachtman et al., 1998; Cerozi and Fitzsimmons, 2016). When $H_2PO_4^-$ is added to soils with pH below 7.2, there is a tendency for the phosphate ion to remove H^+ from solution leading to an increase in soil pH. In contrast, when $H_2PO_4^-$ is added to soils with pH above the 7.2, there is a tendency for the phosphate ion to donate H^+ into solution leading to a decrease in the soil pH. Another factor that could lead to a significant change in soil pH is nitrification of the NH_4^+ present in MAP fertilizers. Under alkaline conditions, production of NO_3^- is markedly enhanced with the optimum pH between 7.0 and 8.0 (Ibrahim et al., 1995). Sample et al. (1980) attributed the pH changes of the soil surrounding the fertilizer granules to the inherent acidic (TSP and MAP) and alkaline (DAP) characteristics of each fertilizer. In addition, changes in soil pH resulting from fertilizer addition have also been attributed to H^+ displacement from the soil cation exchange capacity by cations introduced with the fertilizer (Montalvo et al., 2014). Some soil minerals have pH dependent surface charges, which could be switching between positive and negative charges as the pH changes, creating new binding sites for P or eliminating places where P could adsorb. Therefore, when the soil pH is lowered, protonation of the mineral surface may increase the anion exchange capacity leading to increased adsorption of P on Al and Fe hydroxide minerals (Wisawapipat et al., 2009).

Phosphorus extracted by HCl is typically thought to represent stable calcium phosphate compounds (Negassa and Leinweber, 2009; Yang et al., 2012). The amount of HCl-extractable P was significantly higher when AKPP was applied compared with the other P sources for all soils (Table 4). In addition, HCl extractable P in the AKPP treatments were higher in the soils with low Ca content than in soils with high Ca content (Tables 1 and 4). It is possible that the HCl extractant removed both polyphosphate groups as well as calcium phosphate groups since polyphosphate solubility at neutral and basic pH is limited (McBeath et al., 2007). The fact that the extracting solutions used prior to HCl had pH between 7 (water plus resin) and 13 (NaOH) likely kept the polyphosphate in an insoluble form during the fractionation until the HCl was used.

CONCLUSIONS

Our results showed that both soil and fertilizer characteristics influence P mobility and lability. Struvite was the least soluble and the least mobile fertilizer tested, exhibiting the least movement of P away from the granule. As a result, struvite-amended soils exhibited the greatest concentration of P in the labile pools, due to direct extraction of the undissolved granule. The MAP-based fertilizers were the most soluble and mobile fertilizers studied. Most likely, a combination of changes in soil properties associated with the accompanying cation in the MAP-based fertilizers helped increase the P mobility. With MAP-based fertilizer application soil pH changed significantly compared with the control in a direction that favored increased P solubility and mobility. The phosphate protonation and deprotonation reactions likely affected soil pH, especially in the soil around the fertilizer granule as it dissolved. Phosphorus exhibited greater mobility in soils with high sand content and low initial pH compared with soils with high clay content and initial soil pH. In this study, S and humic acid coatings on monoammonium phosphate had no effect on P lability or mobility compared with the uncoated MAP fertilizer.

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