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Vertical distribution of phosphorus in a sandy soil fertilized with recovered manure phosphates

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Abstract
Purpose The aim of the study was to evaluate the environmental fate of recovered manure phosphates for their effective use as crop fertilizers.
Materials and methods A greenhouse study was conducted to evaluate the leaching potential and vertical distribution of total and plant-available P in a deep sandy Coastal Plain soil of the southern US fertilized with recovered P from liquid pig manure (SRP) and solid broiler litter (LRP). The recovered manure P sources were compared to commercial triple superphosphate (TSP) and raw broiler litter (BL) applied at a single rate of 170 kg P_2O_5 ha^{-1} to 71-cm-tall soil columns with and without cotton plants. All materials were uniformly mixed into the surface 15 cm of soil in each column. All treated and control (unfertilised) soil columns were leached weekly with distilled water for 8 weeks. The water obtained after leaching the soil columns was analysed for total P. Cotton plant material was analysed for P content, and the soil columns were sectioned to determine the vertical distributions of total and plant-available P (Mehlich 3 test).
Results and discussion The vertical soil P distribution showed that most of the total and plant-available P applied with SRP, LRP, and BL materials remained within the top 15-cm of the soil columns, where materials were initially applied. The significant increase of both total and soil available P within 15–22.5 cm of soil columns treated with TSP indicated translocation and potential leaching of P applied as TSP. In the short term of this study (8 weeks), the soil leaching potential of both LRP and SRP was lower than the more water-soluble commercial TSP fertilizer.
Conclusions The use of recovered P could minimize manure P losses into the environment from confined animal production and provide a recycled P source for use as crop fertilizer.

Keywords Available phosphorus · Manure · Poultry litter · Phosphorus recovery · Soil leaching

1 Introduction

Phosphorus (P) is an essential nutrient and a major limiting factor for plant growth in soils with low P content. Thus, soils are usually supplemented with P using either mineral fertilizer or animal manure. In regions with a high density of confined animal production, recurrent land application of manure P at rates that exceed crop removal rates elevates the soil P to levels in excess of soil assimilative capacity (Novak et al. 2000; Ribaudo et al. 2003). Thus, excess P accumulated in soils is an environmental concern because it can pollute water resources by way of field runoff and soil leaching (Kaiser et al. 2009; Schroeder et al. 2004). On the other hand, mineral P is a non-renewable resource whose supply relies on dwindling rock phosphate reserves. Global demand for mineral P is mounting due to the increased demand of agricultural products (food, biofuels, and animal feed) in response to rising world population (Cordell et al. 2009; Keyzer 2010). Together, environmental concerns and global P scarcity forecasts have stimulated the interest on manure P recovery as an alternative to simple land disposal

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of animal manure (Cordell et al. 2011; Szogi and Vanotti 2009). Recovery and reuse of manure P could improve the sustainability of animal production by reducing P losses to the environment and help to make phosphate reserves last longer by providing an effective P fertilizer for crop production.

Transporting manure to croplands low in P is an option available through manure transfer programs. These transfer programs are designed to alleviate pollution concerns in regions with P overloaded soils (NRCS 2003). However, transportation of manure becomes less economical with increasing distance from the animal production farm (Kepplinger and Hauck 2006). Therefore, manure treatment processes that recover P in a concentrated solid form are desirable to facilitate transportation of manure P. This P recovery is possible by precipitating manure soluble P in a diversity of chemical forms such as magnesium ammonium phosphates known as struvites (Webb and Ho 1992) and calcium phosphates (Vanotti et al. 2003).

In the particular case of calcium phosphates, they have been recovered from liquid pig manure (Vanotti et al. 2007) or solid poultry litter (Szogi et al. 2008). Both of these P sources appear to be effective P fertilizers (Bauer et al. 2007; Szogi et al. 2010). Given that the risk of P leaching losses is augmented by over application of P to soils with low P assimilative capacity (Sims et al. 1998), significant vertical movement and leaching of P into shallow groundwater can occur for sandy Coastal Plains soils with low P sorption capacities (Nelson et al. 2005; Novak et al. 2000). Therefore, the environmental fate of non-conventional P fertilizers such as recovered manure phosphates should be evaluated to recommend their use for crop production in sandy soils. The objective was to evaluate the vertical distribution of total and plant-available P in a deep sandy Coastal Plain soil fertilized with recovered P from liquid pig manure and solid broiler litter as compared to raw broiler litter and commercial phosphate fertilizer.

2 Materials and methods

2.1 Recovered P manure materials

The recovered P material from liquid swine manure (SRP) was obtained from a full-scale wastewater treatment facility located on a 4,360-head finisher pig production unit in Duplin County, NC. The system consisted of three treatment stages: solid–liquid separation, biological nitrogen removal, and phosphorus removal. In the final stage, P was recovered as calcium phosphate with the addition of small quantities of liquid lime. Further details of the process extraction and recovery of P from wastewater can be found in Vanotti et al. (2007). The recovered P material from broiler litter (LRP) was obtained at the USDA-ARS Coastal Plains Soil, Water and Plant Research Center, Florence, SC, using a treatment process developed for selective extraction and recovery of P from animal waste solids called "quick wash" that produced a concentrated solid P material (Szogi et al. 2008). At the first step of this process, phosphorus from solid animal wastes is extracted by using mineral or organic acids. In the second step, the phosphorus is recovered by addition of liquid lime and an organic poly-electrolyte to the liquid extract to form a calcium-containing phosphorus precipitate. Untreated raw broiler litter (BL) and triple superphosphate (TSP) were evaluated in the experiment as P sources for comparison with the recovered manure P materials.

2.2 Soil material

A sandy-textured Uchee soil (loamy, kaolinitic, thermic Arenic Kanhapludults) was used in all experiments. The Uchee soils are very deep, well-drained soils characteristic of the Coastal Plains in the states of Alabama, Florida, South Carolina, North Carolina, and Virginia. They are formed in sandy and loamy marine sediment on ridge tops and dissected side slopes (National Cooperative Soil Survey 1997). For our study, the soil was collected from a wooded site located at the Pee Dee Research and Education Center, Clemson University, Florence, SC (latitude 34° 18′ N, longitude 79° 44′ W). Chemical analysis performed at Clemson University's Agricultural Service Laboratory indicated a strong acid reaction, a low exchange capacity, and a very low content of plant-available P (1.7 mg kg^{-1}) (Table 1).

2.3 Experimental setup

The study consisted of two soil column leaching experiments conducted in duplicate—two trials without plants and two trials with cotton (*Gossipium hirsutum* L.). The cotton plants served the purpose to determine if there was an effect of plant P uptake and root growth on vertical soil

Table 1 Chemical properties before liming the Uchee soil used in the greenhouse study

Soil properties	Value[a]
Carbon (g kg^{-1})	2.50±0.01
Cation exchange capacity (cmol kg^{-1})	2.0±0.6
Exchangeable acidity (cmol kg^{-1})	1.8±0.4
pH in water	4.9±0.1
Available phosphorus (mg kg^{-1})	1.7±0.1
Total phosphorus (mg kg^{-1})	55±2

[a] Data are mean of two samples±standard error (*n*=2).

P distribution. The leaching experiments consisted of soil columns that were prepared in 15-cm-wide×76-cm-long PVC cylinders. The bottoms of the columns were closed with a layer of cheesecloth. Prior to filling the columns, the soil was limed to raise the pH to about 6.5 according to Szogi et al. (2010). Each soil column was filled up to a height of 71 cm and packed to a bulk density of 1600 kg m^{-3}.

The experiments were conducted in a greenhouse with average daily air temperatures in the range of 25.5 to 32.0°C. Each experiment consisted of four P fertilizer materials as treatments and a control with no P addition. Each treatment had four replicates per experiment. The four P fertilizer materials (BL, SRP, LRP, and TSP) were added at a fixed rate of 0.134 g P per soil column (equivalent to 170 kg P_2O_5 ha^{-1}). The bottom 56 cm of the columns were filled with unfertilized soil. The four P sources were then uniformly mixed within the remaining surface 15 cm of soil and placed on top of the unfertilized soil in the columns. After allowing P materials to react with the soil for about 1 month, cotton was planted into designated soil columns with the four fertilizer treatments. In addition, each soil column with cotton received 2.73 g of 15–0–15 (N–P_2O_5–K_2O) fertilizer and 0.16 g of technical grade $(NH_4)_2SO_4$.

A volume of 2.0 L of distilled water was used to leach every soil column once a week for 8 weeks. This volume of water displaced at least one soil pore space volume of each soil column. This wetting regime simulated about 75% of the average annual precipitation (118 cm) at the soil location. Soil columns were lightly watered between leaching events to keep the soil at the surface of the columns moist. At the end of the 8 weeks of the study, plants were harvested and all soil columns, with and without plants, were sampled at the following six soil depths: 0–7.5 cm, 7.5–15.0 cm, 15.0–22.5 cm, 22.5–37.5 cm, 37.5–52.5 cm, and 52.5–71.0 cm.

2.4 Chemical analysis

Duplicate samples of SRP, LRP, and TSP were analyzed for total P (TP), available P (neutral ammonium citrate-soluble), and citrate-insoluble P according to AOAC Methods 958.01, 960.02, 963.01B(a) at Thornton Laboratories Inc. (Tampa, FL). The total P content of BL was determined in duplicate samples by inductively coupled plasma analysis (ICP, Vista Pro, Varian Instruments, Walnut Creek, CA) after automated digestion (AutoBlock, Environmental Express, Mt. Pleasant, SC) with nitric acid and hydrogen peroxide (Peters et al. 2003). In addition, we determined the water-soluble P fraction of the four materials using a procedure modified from Sharpley and Moyer (2000); 1.00 g of air-dried material was combined with 250 mL of deionized water and shaken end-over-end for 1 h. Following 10-min centrifugation and 0.45-µm filtration, the sample extracts were acidified and analysed for P using ICP.

Cotton plants were digested with concentrated sulfuric acid (Gallaher et al. 1976) and P concentration determined in the digest by automated colorimetric analysis adapted to digested extracts (Szogi and Vanotti 2008). Soil samples collected at the end of the experiment were extracted using the Mehlich 3 (M3) test procedure to determine plant-available P (Sims 1992). The plant-available P content in soil extracts was determined using automated colorimetric analysis (AAII, Technicon Instruments, Tarrytown, NJ). The TP content in soil leachate was determined by ICP after automated digestion.

2.5 Statistical data analysis

The experimental design was a randomised complete block with four replications. Both TP and plant-available soil P were analysed across the two duplicate experiments using a mixed model analysis with the GLIMMIX procedure of SAS (SAS Institute, Cary, NC). For the analysis of variance (ANOVA), experiments, trials, and replicates were considered random, and treatments (fertilizer materials) and depth were considered fixed. Plant P was analysed across both experiments using the same SAS mixed model analysis with trials and replicates as random effects and treatments as fixed effects. The effect of fertilizer source on P mass leached out weekly from soil columns was analysed using GLIMMIX repeated measures. Difference between least square means of any two treatments was considered significant when it was larger than the least square difference (LSD) with a probability of $t \leq 0.05$.

3 Results and discussion

3.1 Phosphorus content of fertilizer materials

The P content for the four fertilizer materials used in this study is shown in Table 2. The TP content of recovered phosphates, LRP (56 g kg^{-1}) and SRP (112 g kg^{-1}), was lower than the TP of the commercial TSP (201 gP kg^{-1}). Both LRP and SRP were much more concentrated in P than BL; their total P content was almost fivefold and ninefold the TP content of BL (12 g P kg^{-1}), respectively. In general, solubility of P fertilizers is assessed according to five categories: water-soluble, citrate-soluble, citrate-insoluble, total, and available P (AOAC International 2000). The fertilizer material sample is first extracted with water to remove water-soluble P; followed by filtration, the residue is extracted with neutral ammonium citrate to remove citrate-soluble P. Subsequently, the remaining residue after citrate extraction is digested to determine citrate-insoluble P. The difference between citrate-insoluble P and TP was

Table 2 Phosphorus characterization of source materials

Source	Total P (g kg$^{-1)}$	Available P (g kg^{-1})	Available P fraction (% total P)	Water-extractable P (g kg^{-1})	Water-soluble (% total P)
BL	12	nd	nd	2.2	18
SRP	112	111	99	1	1
LRP	56	52	92	0.5	1
TSP	201	201	100	173	86

BL raw broiler litter, *SRP* recovered P from pig manure, *LRP* recovered P from broiler litter, *TSP* triple superphosphate, *nd* not determined

reported as available P (AOAC Method 960.02). Our results shown in Table 2 indicate that SRP, LRP, and TSP had high available P. However, the available P did not clearly differentiate SRP and LRP from TSP because it represented 92 to 100% fraction of TP.

Since the water-soluble P fraction of conventional fertilizers such as TSP represents a significant portion of available P (Chien et al. 2011), we further determined the water-extractable P in all four materials to estimate the water-soluble percent with respect to TP. Both SRP and LRP had the lowest water-extractable P content (0.5 and 1.0 g P kg^{-1}) with respect to BL (2.2 g P kg^{-1}) and TSP (173 g P kg^{-1}). In the case of BL, a large fraction of P (> 50%) is in organic form and not readily bio-available for plant uptake (Turner and Leytem 2004); the P solubility in BL is controlled by inorganic phosphates such as di-calcium phosphate ($CaHPO_4$), amorphous calcium phosphate, and water-soluble P (Sato et al. 2005). The water-soluble fraction of BL that represented only 18% of TP (see Table 2) was within the 14–19% range of the water-soluble P fraction previously reported for raw broiler litter (Sistani et al. 2003). As expected, TSP had the highest water-soluble fraction (86%) with respect to its TP content. Since SRP was recovered from poultry litter, the lower water-soluble P fraction is probably related to the low solubility of inorganic P compounds. The mineral identification of SRP suggested the presence of amorphous calcium phosphate formed during the process of P recovery from liquid swine manure (Szogi et al. 2006). The LRP instead contained both water insoluble organic and inorganic P fractions as reported by Szogi et al. (2010). Thus, the low water-soluble P fractions of both SRP and LRP (1%) may qualify them as slow release P fertilizers.

3.2 Plant and total soil phosphorus

Accumulation of TP contents in the range of 400–800 mg P kg^{-1} has been reported for sandy Coastal Plain soils that were grossly overloaded by long-term manure applications (Nelson et al. 2005; Novak et al. 2000). In our study, the fixed application rate of 134 g of P per soil column for all treatments corresponded to about 170 kg P_2O_5 ha^{-1} when applied on a ground area basis. This P rate was selected from previous pot trials (Bauer et al. 2007; Szogi et al. 2010) to emulate a high soil P application, and it is according to annual application rates of manure P used in field trials (Sharpley et al. 2004). This P application rate significantly elevated the initial background soil P content from 55 mg TP kg^{-1} (control) to P levels within the range of 70 to 85 mg TP kg^{-1} in the top 15 cm of the soil columns (Fig. 1). Because the effect of plants was not significant, Fig. 1 shows the pooled least square means of TP concentration for all experiments.

Differences in both cotton biomass and plant uptake were not significant among treatments. Plant uptake with means in the range of 1.2 to 2.3 mg P per column was a very small percent (0.9 to 1.7%) of the high P rate (134 mg of P) applied per column (Table 3). Earlier, we carried out two plant response studies using annual ryegrass (Bauer et al. 2007; Szogi et al. 2010). Both studies showed that there were no significant differences in biomass production

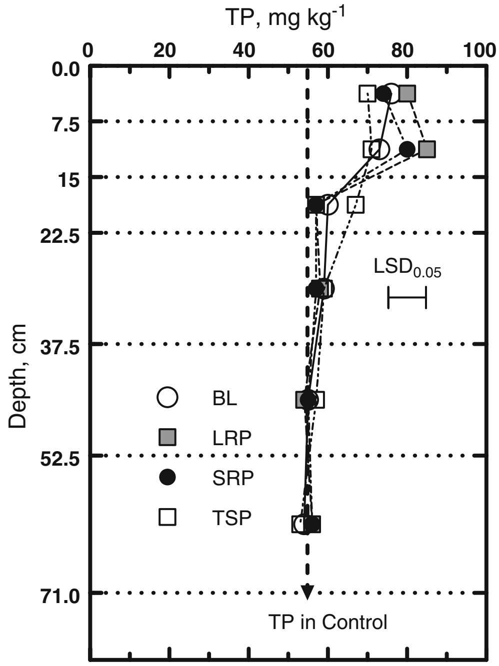

Fig. 1 Vertical distribution of total phosphorus (*TP*) concentration in soil columns; data points are the pooled least square mean of all experiments (with and without plants) for four fertilizer treatments. The *dotted line* indicates TP content (55±2 mg kg^{-1}) in the control soil columns with no fertilizer treatment application. The LSD *bar* applies for comparisons between any two means and the control at the 0.05 probability level.*BL* raw broiler litter, *SRP* recovered P from swine manure, *LRP* recovered P from broiler litter, *TSP* triple superphosphate

Table 3 Phosphorus removed by plant uptake (cotton shoots and leaves)

Treatment	Plant Uptake[a] (mg P per soil column)
BL	1.3±0.3
SRP	1.2±0.7
LRP	2.3±1.4
TSP	2.3±1.1

[a] Mean and standard error of the mean. Differences among means are not significant at $P=0.05$

BL raw broiler litter, *SRP* recovered P from pig manure, *LRP* recovered P from broiler litter, *TSP* triple superphosphate

among SRP, LRP, and TSP at any application rate within the range of 22 to 176 mg P kg soil^{-1}), but ryegrass plants had higher significant P uptake with TSP than with SRP or LRP. These results hinted that the less water-soluble P materials (SRP and LRP) could be used as slow release P fertilizer sources. However, the lack of significant differences in P uptake with cotton plants is probably because of the limited response of cotton to P application in Coastal Plain soils (Mitchell 2010). Since in this experiment we did not grow the plants to maturity, so we could determine plant yield, additional field experiments will be needed to determine the advantage of using SRP and LRP versus more water-soluble commercial P sources for cotton production.

Leaching accounted for a total mass of less than 0.50 mg of TP mass per soil column; differences in accumulated TP mass leached among treated and control columns were not significant. Nevertheless, the soil depth and the interaction between fertilizer material and depth (treatment × depth) had a significant effect on the vertical distribution of TP concentration in the soil columns. Differences in TP content among materials were significant above 22.5-cm depth (Fig. 1). Nearly all of the TP remained mostly concentrated on the top 15 cm for the BL, SRP, and LRP. Below 15-cm depth, the TP concentration at 15–23 cm supplied by TSP treatment was significantly higher than the TP in untreated control columns. These results indicate translocation of TP from the top 15 cm into this lower 15–23-cm depth within the TSP treated soil columns. This P translocation agrees with the higher water-soluble P fraction of TSP (86%) shown in Table 2. Below 23-cm depth, differences in TP concentrations for all treatments were not significant with respect to the TP content of the control soil columns (55 mg/kg). This explains the lack of significant differences of TP in leachate samples among the control and treated soil columns.

3.3 Plant-available soil phosphorus

The M3 extractant is widely used in the Mid-Atlantic USA and other states as an agronomic soil test that measures plant-available soil P. In addition, M3 test results can be used as an effective approach to guide environmentally based P recommendations for commercial fertilizers, manures, biosolids, and other sources (Sims et al. 2002). Our ANOVA results indicate that fertilizer materials and soil depth and their interaction (material × depth) had a significant effect on vertical distribution of plant-available P in soil columns. Therefore, Fig. 2 shows, as in the case of TP, the pooled least square means of plant-available P (M3) for all experiments.

Although cotton plants developed long roots that reached the bottom of the soil columns, the interaction of plant with either fertilizer material or soil depth had no significant effect on plant-available P distribution. Except for the TSP source, the plant-available P remained mostly concentrated on the top 15 cm where it was initially applied (see Fig. 2); the highest P concentration was 18.9 mg kg^{-1} (SRP), and the lowest was 14.4 mg kg^{-1} (TSP) within the top 15 cm. Below 15-cm depth, the soil P concentration at 15–23 cm supplied by TSP was significantly higher than the lower P concentrations in soil columns with BL, SRP, and LRP treatments. This large difference between TSP and the other three P sources is likely due to the effect of chemical properties of the soil and P fertilizer materials (e.g., pH) on both P solubility and reaction with soil minerals. Phosphorus solubility and reaction with soil minerals are both controlled by pH. In our study, the soil pH was corrected to between 6.0 to 6.5

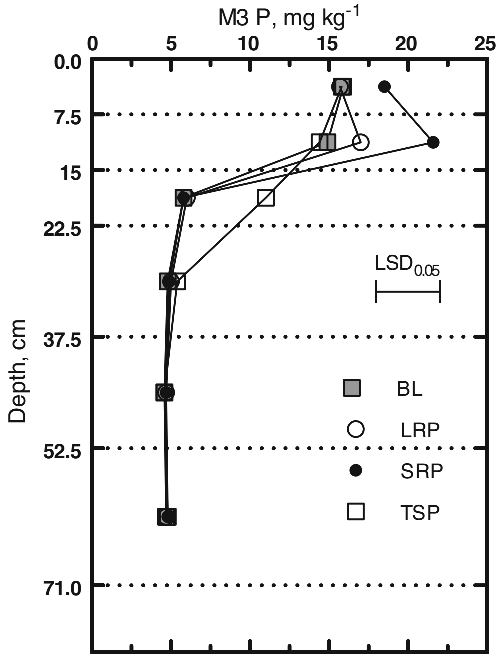

Fig. 2 Vertical distribution of plant-available phosphorus (*M3*) concentration in soil columns; data points are the pooled least square mean of all experiments (with and without plants) for four fertilizer treatments. The LSD *bar* applies for comparisons between any two means at the 0.05 probability level. *BL* raw broiler litter, *SRP* recovered P from swine manure, *LRP* recovered P from broiler litter, *TSP* triple superphosphate

units to favour P dissolution and availability to plants (Lindsay 1979). Since TSP is highly soluble in water, it first dissolves rapidly in moist soil and subsequently reacts with soil and becomes available to plants with a favourable pH (Havlin et al. 1999). Because BL had much lower water solubility than TSP, and both SRP and LRP were almost insoluble in water, these three P sources were less susceptible to translocate than TSP under the soil conditions of our experiments. For the SRP and LRP manures, available P comprises a high percentage of TP which is associated with calcium used to precipitate the P from the original raw manure. This calcium phosphate compound is less soluble than the P contained in TSP, and therefore when deionised water was applied to the soil surface, TSP migrated to a greater extent than SRP and LRP. Below 23-cm depth, soil P concentrations were low (< 5.4 mg kg^{-1}) for all treatments with no significant differences among P sources. The pooled average of all experiments (both trials without and with plants) of the M3–TP ratio supports the soil distribution profiles shown in Fig. 3. In summary, the M3–TP ratio > 20 indicates accumulation of P within the top 15 cm, while the significantly higher ratio of 17 within the 15–22.5-cm soil depth is indicative of plant-available P translocation for the TSP treatment. The lack of significant differences among treatments and much lower M3–TP ratios (8 to 9) below 22.5-cm depth indicate very little translocation of P to the lower parts of the soil column.

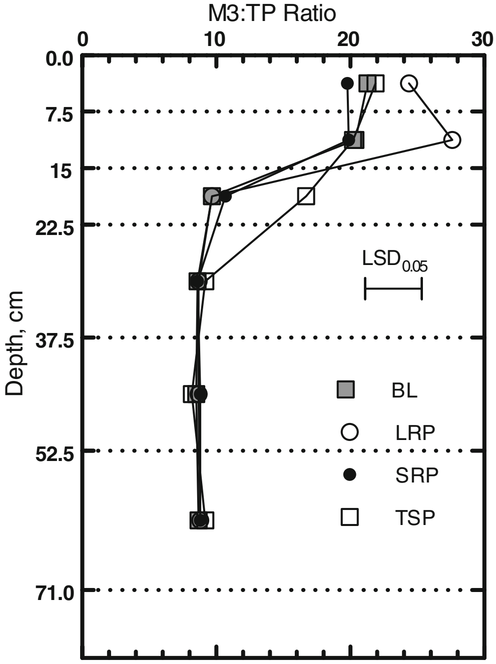

Fig. 3 Vertical distribution of plant-available soil phosphorus to total phosphorus ratio (*M3:TP*) in soil columns; M3:TP=(mg M3 P kg^{-1}/mg total P kg^{-1})×100. Data points are the pooled least square mean of all experiments (with and without plants) for four fertilizer treatments. The LSD *bar* applies for comparisons between any two means at the 0.05 probability level. *BL* raw broiler litter, *SRP* recovered P from swine manure, *LRP* recovered P from broiler litter, *TSP* triple superphosphate

We conclude from this short-term study that the soil leaching potential of both SRP and LRP is much lower than the commercial TSP fertilizer under a soil pH range (6.0–6.5) that favours P dissolution and availability to plants. This is a desirable pH range to induce P uptake by cotton since cotton plants are not very efficient in utilizing low soluble phosphorus sources such as mineral aluminum and calcium phosphates (Wang et al. 2011). In the long-term, substantial P solubilization and leaching could occur under field conditions with favourable soil pH and large amounts of precipitation. However, P may become less plant available and accumulate in the soil profile since abundant precipitation may lower both ionic strength and soil pH such that P may react with exchangeable aluminium to form insoluble aluminium phosphates. On the other hand, we speculate that long-term application of SRP and LRP at the high P rate of our study may turn soil pH to neutral or alkaline reaction leading to the accumulation of rather insoluble calcium phosphates. Additional field research with lower P application rates, more soil types, and other crops will provide additional information on the fertilizer effectiveness and long-term risk of P loss from soils amended with recovered manure P materials.

4 Conclusions

To assess the environmental fate of P, the vertical soil P distribution was evaluated after leaching a sandy soil fertilized with two non-conventional phosphates recovered from animal manure, commercial TSP, and raw broiler waste. Both recovered manure P sources (SRP and LRP) provided sufficient plant-available P for cotton plant growth. Differences in total mass P leached out of the fertilizer-treated soil columns were not significant with respect to the unfertilized control. In the short-term (8 weeks) of our study, the soil P vertical distribution of both LRP and SRP was similar to BL with most of the plant-available P supplied by these three materials remaining within the top 15-cm soil, where materials were initially applied. The statistically significant increase of both total and soil available P within 15–22.5 cm of soil columns treated with TSP indicated translocation and down movement of P applied as TSP. Under the conditions of our study, the soil leaching potential of both SRP and LRP was much lower than the more water-soluble commercial TSP fertilizer. Although further research is needed with more soil types, and long-term field applications, the recovered P materials from liquid manure and broiler litter have the potential for use as plant fertilizers. These recovered phosphates appear to have use as slow release fertilizer without further chemical processing into other P materials, such as the acid treatment typically used to process rock phosphate for fertilizer production. Because these recovered phosphates have much higher P concentration than raw animal manure, they could be transported longer distances than raw manure to substitute commercial inorganic P fertilizer. The use of recovered phosphates could minimize manure P losses into the environment from intensive confined animal production and provide a recycled P source for use as an effective crop fertilizer.

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