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Soil Phosphorus Sources and Their Relative Water Solubility and Extractability

Daniel Herrera^a, Rao S. Mylavarapu^b, Willie G. Harris^b, and James Colee^c

^aAffiliate Professor, EARTH University, Costa Rica; ^bSoil and Water Sciences Department, University of Florida, Gainesville, Florida, USA; ^cStatistical Consulting Unit, IFAS, University of Florida, Gainesville, Florida, USA

ABSTRACT

Soil testing is a tool used to predict crop response to residual soil phosphorus (P). It is commonly perceived that high soil test P indicates high potential for off-site movement and impact on surface water quality. However, P source potentially confounds the relation between soil test P and soil P solubility. This study evaluated how soil test P (Mehlich-1 and Mehlich-3) and total P (TP) related to water-soluble P (WSP) for manure- ($n = 120$) and fertilizer-amended ($n = 120$) soils, and soils forming in phosphate-rich parent material (phosphatic soils; $n = 60$). Results document marked differences in slopes of WSP as a function of soil test P and TP for the three P sources. Manure-amended soils showed the highest regression coefficients ($R^2 = 0.89, 0.89$ and 0.92) and slopes for WSP but phosphatic soils, in contrast, showed no tendency for increased WSP with increasing soil test P or TP despite having the highest range of TP content. Fertilizer-amended soils had lowest values for all P measures. Manure-applied P accumulated to much higher levels in these sandy soils than P applied as inorganic P suggesting the latter is either quickly recovered by crops or lost. Phosphate minerals in naturally phosphatic soils do not likely constitute the same extractable P pool as for agriculturally amended soils. Hence, soil test extractions may not be well-calibrated for phosphatic soils. Standard soil test P values may not be a reliable indicator of high P leaching loss potential for naturally phosphatic soils, to the extent that WSP is related to leaching risk.

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Introduction

Phosphorus (P) is a fundamental constituent in the metabolic cycle and biochemistry of all living organisms. While nitrogen (N) is commonly referred to as the most limiting nutrient for terrestrial plant growth, P is considered particularly to be the principal yield limiting factor for annual crop production in acid and alkaline soils of temperate as well as tropical regions (Fageria, Baligar, and Jones 1997). A calibrated soil test is a tool that serves as the primary critical step in determining P and other nutrient needs for optimum crop production. Traditional soil- and plant-testing programs provide recommendations for applications of P to ensure long-term economic agricultural production based on estimations of the P supplying capacity of the soil (Mylavarapu 2010). For successful crop production, mineral and/or organic P inputs are often necessary to optimize productivity. However, over-fertilization is common in many regions, particularly where the only feasible option is to apply organic wastes (due to limited land area available and/or high cost of transportation) or where high cash value crops are grown and fertilizer cost is not a major concern (Sims et al. 2000). Elevated soil P concentrations under certain conditions can be a potential threat to the environment leading to eutrophication problems in sensitive surface water bodies (Brye et al. 2002). As a consequence,

agriculture is consistently identified as one of the largest contributors of diffused pollution of P to surface waters (Duriancik et al. 2008; Kronvang, Rubæk, and Heckrath 2009; Torrent, Barberis, and Gil-Sotres 2007).

According to the United States Environmental Protection Agency (U.S. Environmental Protection Agency 2017), agriculture is the leading source of water quality impairments in rivers and lakes in the USA.

In developed countries, where significant reductions have been made in point-source P pollution, agricultural non-point sources are often the greatest source of P to eutrophic water bodies (Dubrovsky et al. 2010; U.S. Environmental Protection Agency 2010).

The initial magnitude of P concentrations in runoff from applied P wash-off is generally a function of the rate of manure or commercial fertilizer application and the solubility of applied P (Kleinman et al. 2007). Notably, the P in mineral fertilizers tends to be so soluble that a disproportionate fraction of applied fertilizer P is translocated into soil by infiltrating rainfall where it is rapidly sorbed.

Identification of factors contributing to potential P losses from agriculture is critical. Advantages of soil tests include their immediate availability, relatively low cost, and extensive existing data. Standard soil test P measures, including Mehlich-1 (M-1 P) and Mehlich-3 (M-3 P) extraction procedures, among others, have been proposed for use as environmental risk indicators because they have been found in some cases to correlate well with risk for offsite P movement through run-off (Sharpley 1995; Torbert et al. 2002).

Water-soluble P (WSP) has been considered as the soil P fraction that is most susceptible to P losses. Therefore, WSP in soils likely controls dissolved reactive P concentrations in runoff (McDowell and Sharpley 2001; Pote et al. 1996; Sims et al. 2000) and WSP also positively correlates with P concentrations from leaching studies (Maguire and Sims 2002).

While integration of soil testing and environmental risk assessment may be an option, the source of P is another variable that affects the relation between WSP and soil test P concentrations. Common sources include inorganic fertilizers and manures. Also, soils forming in phosphatic parent materials (“phosphatic soils”) contain phosphate minerals that are a source of P. Phosphatic soils occupy significant land areas in certain regions of the USA (e.g., Florida and Kentucky) as well as other areas of the world, where phosphate-rich geologic deposits are exposed at the land surface. They are commonly distinguishable from soils anthropogenically enriched in P by having an increase in total-P (TP) with depth. Despite the high P-content of these soils, mobility is thought to be limited, particularly in soils that have been intensely weathered because more stable P-forms (e.g., wavellite) are prevalent and therefore significant P-leaching is unlikely to occur (Wang, Harris, and Yuan 1989). However, evidence documenting this inference is lacking.

In Florida, inorganic P fertilizers are widely used in vegetables, strawberry (*Fragaria ananassa*), sugarcane (*Saccharum officinarum*), citrus (*Citrus species*), and many other crops grown with intensive management of nutrients and water for commercial viability. Inorganic fertilizers tend to have high solubility, which can elevate risk of P loss from soil through leaching and/or runoff. However, this risk can be partially offset by precipitation reactions between fertilizer solution components and soil components such as Mn, Ca, Fe, and Al (Lindsay, Frazier, and Stephenson 1962), the latter two being common in acid-mineral soils of Florida. The chemical reaction following P fertilizer application to soil determines the corresponding end products, and consequently the solubility and the fate of P.

In regions across the USA, including Florida (Allen 1987; Chakraborty et al. 2011; Josan et al. 2005; Nair, Graetz, and Portier 1995), manures from animal agriculture (e.g., dairies, confined feeding operations) are routinely applied to farmland to meet crop N requirements. This approach typically exceeds crop P requirements, resulting in buildup of soil-bound P that can ultimately lead to surface water quality degradation (e.g., Allen 1987; Sharpley 1996). In dairy manure, P fractions depend on the P concentrations in the diet. Dou et al. (2002) found that increase in dietary P concentrations from 3.4 to 6.7 g P kg⁻¹ DM, correspondingly increased water-soluble P from 24.2 to 94.4 g cow⁻¹ day⁻¹ in the feces, while the other P fractions remained small and with little or no variation. In the same study, they also characterized P in the feces, where water-soluble amount was between 56% and 64% and inorganic was between 46% and 59% of TP in feces.

Studies comparing the water solubility with the source of P in soils in order to better understand the effect of P source on solubility and consequently the potential for P movement from the site of application are limited. The objective of this study was to evaluate how TP and soil test P (M-1 and M-3) relate to WSP for manure, fertilizer-applied soils, and soils forming in phosphate-rich parent material (phosphatic soils). The results are pertinent to the potential effects of P source differences on the sensitivity of agronomic and environmental soil P assessments.

Materials and methods

Site selection and soil sampling

Sampling sites were selected based on three general soil categories: phosphatic, inorganically fertilized, and manure-amended soils. Samples analyzed were collected from a total of seven Florida counties. Phosphatic sites were native areas of North Central Florida, where soils were known to be likely influenced by phosphate-rich parent material (Cathcart 1986; Ramnarine, Harris, and Grunwald 2015) and judged to be minimally affected by contemporary anthropogenic activities. These included four sites along the Cody Escarpment on or in the vicinity of the University of Florida campus and five sites in Northern Marion County, FL, including four sites on the University of Florida Plant Science Research and Education Unit. These soils were inferred to be Udufts or Udalfs based on morphology and county soil survey data. Presence of phosphate-cemented nodules, total P concentration ($>1000 \text{ mg kg}^{-1}$) and sustained high P concentrations with depth were the criteria used to confirm the presence of naturally occurring P. These criteria indicated that elevated P concentrations at the phosphatic sites were primarily attributable to geologic (parent material) rather than anthropogenic P sources. Additionally, 30 samples from 5 counties on the Florida peninsula, meeting the criteria stated above for inferring a naturally phosphatic soil, were available in the sample archive of the former Florida Cooperative Soil Survey (FCSS) (<https://soils.ifas.ufl.edu/flsoils/>). Data from these samples are also included and presented in this study. These samples tracked closely with the data for samples collected in this study and helped amplify the source-solubility relationships found in this study. The phosphatic soils selected exclusively for this study were sampled at 0–5, 5–15 and 80–100 cm depth; deeper samples were collected to confirm that P had not declined with depth. The FCSS archived samples ranged in depth from 0 to $>200 \text{ m}$. They included Udufts, Udalfs, Aqualfs, and Aquods.

Manure-amended and inorganically fertilized soil sites were selected based on farm records obtained from the Suwannee River Water Management District, Florida, indicating that the areas had received exclusively either inorganic fertilizers or dairy manure as nutrient sources for crop production. All sites were within the Middle Suwannee River Basin. Years of either manure or inorganic fertilizer application ranged from 12 to 30 years. Samplings were not done around manure application times to minimize any impacts on sample analyses and results. The frequency and rate of fertilizer application in each field varied according to the crop needs. Composite samples at 0–5 and 5–15 cm depths were taken. Six sites for each category were sampled, and 10 samples per field were collected based on a field transect.

Soil chemical and mineralogical analysis

Soil pH, Mehlich soil extractions and analyses, extraction of WSP from soils was done at the IFAS Analytical Services Laboratories, University of Florida following the standard methods (Mylavarapu, Bergeron, and Wilkinson 2021). Soil pH was determined with a 1:2 (v/v) soil-to-water ratio using a glass electrode. Mehlich-1 P was extracted at a soil:solution (0.0125 M H_2SO_4 + 0.05 M HCl) ratio of 1:4 w/v with 5-min reaction time, and filtered using Whatman #42 filter paper. Mehlich-3 (a solution

containing 0.015 M NH_4F + 0.25 M NH_4NO_3 + 0.001 M EDTA + 0.2 M CH_3COOH + 0.13 M HNO_3) was used to extract P at a 1:10 w/v ratio, with a 5-min reaction time, and then filtered with Whatman #41 filter paper. Water-soluble P (WSP) was determined using a 1:10 (w/v) soil to water ratio and 1-h shaking time followed by vacuum filtration through a 0.45- μm Millipore membrane. From the recovered filtrate, P was analyzed on ICP-AES (method 200.7- U.S. Environmental Protection Agency 1994). From both Mehlich extractions, the collected supernatant solution was analyzed for P, Ca, Mg, Fe and Al using Inductively Coupled Plasma Atomic Emission Spectroscopy using EPA method 200.7. For TP determination, all FCSS samples were determined using the method described by Dick and Tabatabai (1977). For all the other samples, 1 g of soil was placed for 1 h in a muffle furnace at 350°C, followed by raising the temperature to 500°C for 4 h. Once the sample was cooled, the ash was moistened with a few drops of DI water and 20 mL of 6 M HCl were added. The solution was allowed to evaporate slowly on a hot plate. When the residue was dry, the temperature was raised to 120°C to dehydrate the silica. Upon cooling, 25 mL of 0.5 M HCl were added to the contents of beaker. The beaker was warmed again to dislodge any residue. The samples were then allowed to sit in acid solution for approx. 30 minutes before filtration using Whatman 41 filter paper and transferring quantitatively into 20-mL scintillation vials for TP analysis on the ICP (Mylavarapu, Bergeron, and Wilkinson 2021).

Total carbon (TC) was determined by an automated combustion procedure at 1020°C using a Carlo-Erba (Milan, Italy) NA-1500 CNS Analyzer. For the procedure, 0.05 g of soil sample was weighed on aluminum cups and then ignited in the combustion chamber. Total carbon was calculated based on known weights of the standard. The total carbon for the soils of this study can be inferred to be organic carbon given the acidic nature of the soils in the study region.

Dry powder mounts of ground nodules found in samples from 3 of the naturally phosphatic soils (80–100 cm depth) were prepared for X-ray diffraction (XRD) to confirm the presence of P-minerals (Figure 1). Nodule samples were from two soils in Alachua County, FL and one soil from Marion County, FL. The soils and nodules were considered morphologically representative of the phosphatic soils (as confirmed by sustained TP with depth) sampled in the study. The purpose of sampling nodules from three representative soils was to gain confidence that nodules being identified morphologically as phosphatic indeed contained phosphate minerals. Samples were scanned from 2° to 60° 2 θ with Cu K α radiation using a computer-controlled x-ray diffractometer equipped with a stepping motor and graphite crystal monochromator.

Particle-size distribution was determined by the pipet method (Gee and Bauder 1986), with sodium hexametaphosphate as the dispersing agent, following organic matter removal using hydrogen peroxide.

Statistical analysis

Slopes for relationships between WSP and the variables M-1, M-3 and TP were statistically compared for the P-source sample sets: inorganically-P-fertilized soils, manure-amended soils, and naturally phosphatic soils. A general linear mixed model was used because the variance was not constant across treatments and depths. Also, a multivariate regression analysis was performed using the SAS JMP v.15 (SAS Institute 2019).

Results and discussion

Samples collected to represent all three P sources, excluding FCSS samples, were relatively sandy, with sand content ranging from 86% to 98%. The FCSS samples had a broader textural distribution, with sand content ranging from 39% to 92% and mean clay content of 28%. Finer textures for the FCSS samples were due to a greater proportion of finer-textured subsurface horizons being represented. The

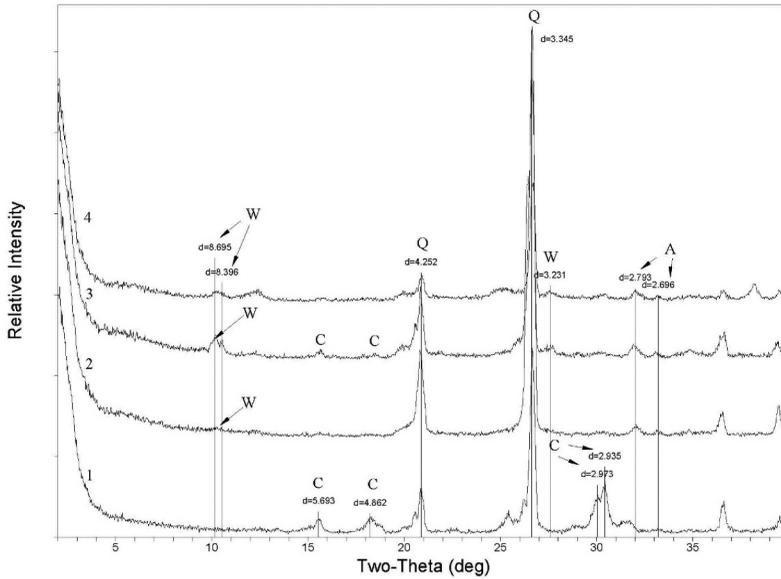


Figure 1. X-ray diffraction scans for ground phosphate-cemented nodules collected from phosphatic soils analyzed in the study. Sample #1 was from a soil located on the University of Florida Plant Science Research and Education Unit in Northern Marion County, FL. Samples #2 and #3 were from the Cody Encarpment in the vicinity of the University of Florida campus in Gainesville, FL. Sample #4 was from the same soil as sample #3 but was elutriated to collect the finer components of the nodule. The minerals identified are apatite (A), wavellite (W), crandallite (C), and quartz (Q).

overwhelming majority of samples analyzed in this study were acidic; the pH ranged from 4.1 to 7.5. Descriptive statistics on P solubilities with water-, M-1 and M-3 extractions and the TP for the three sources of soils are provided in [Table 1](#).

All soil profiles at the sampling sites that were anticipated to have phosphatic parent materials met the pre-set criteria for naturally phosphatic soils. All had P concentrations $>1000 \text{ mg kg}^{-1}$ to 100 cm depth with no tendency to decline with depth. They also contained coarse fragments with morphologies typical of P-cemented nodules that commonly occur in phosphatic soils (Wang, Harris, and Yuan 1989). Nodules from three selected sites were confirmed using XRD to contain phosphate minerals commonly found in phosphatic soils, to include apatite (3/3 sites), crandallite (2/3 sites), and wavellite (3/3 sites) ([Figure 1](#)).

All these minerals have been previously reported for various phosphatic soils in Florida (Wang, Harris, and Yuan 1989, 1991; Ramnarine, Harris, and Grunwald 2015). Mean TP was significantly higher for phosphatic soils (3334 mg kg^{-1}) than for inorganically-fertilized (252 mg kg^{-1}) and manure-amended (661 mg kg^{-1}) soils.

The manure-amended soils showed a steeper increase in WSP with increases in these measures than was the case for the fertilizer-amended or phosphatic soils ([Figures 2,3 and 4](#)). The range and maximum accumulation of P in manure-amended soils, based on TP and soil test P measures, were much greater than for fertilizer-amended soils as shown in [Table 1](#). The high solubility of commercial P fertilizers likely precludes appreciable P accumulation in the sandy soils of the study area. However, the P in manure is likely to be in sparingly soluble forms as associated with calcium (Ca) or magnesium (Mg) (Eghball et al. 2002; Herrera et al. 2010; Nair, Graetz, and Portier 1995) which can tend to build up in soils with repeated application such as in dairy sprayfields.

The naturally phosphatic soils were distinct from the manure-amended soils in that their WSP values remained relatively low over their broad range of TP and soil test P values. As a comparison for perspective, the phosphatic soil sample with highest TP value ($34,505 \text{ mg kg}^{-1}$) had a WSP value of $<20 \text{ mg kg}^{-1}$, whereas the manure-amended samples with highest TP

Table 1. Descriptive statistics for Mehlich-1 P, Mehlich-3 P, water-soluble P, and total P for each of the three soil P sources and for all sampling depths.

Statistic	Soil P Source		
	Phosphatic (including 30 FCSS samples*)	Inorganic Fertilized	Manure Amended
-----mg kg ⁻¹ -----			
Mehlich-1 P			
Mean	177	64	279
Range	3–2340	22–128	35–1350
SD	357	25	276
N =	62	120	120
Mehlich-3 P			
Mean	206	128	336
Range	70–650	60–230	100–1000
SD	120	39	213
N =	62	120	120
Water Soluble P			
Mean	5	6	31
Range	0–20	1–10	2–150
SD	5	2	29
N =	62	120	120
Total P			
Mean	7916	256	659
Range	1230–34500	120–400	260–2270
SD	7780	74	379
N =	53	106	119

*FCSS = Florida Cooperative Soil Survey.

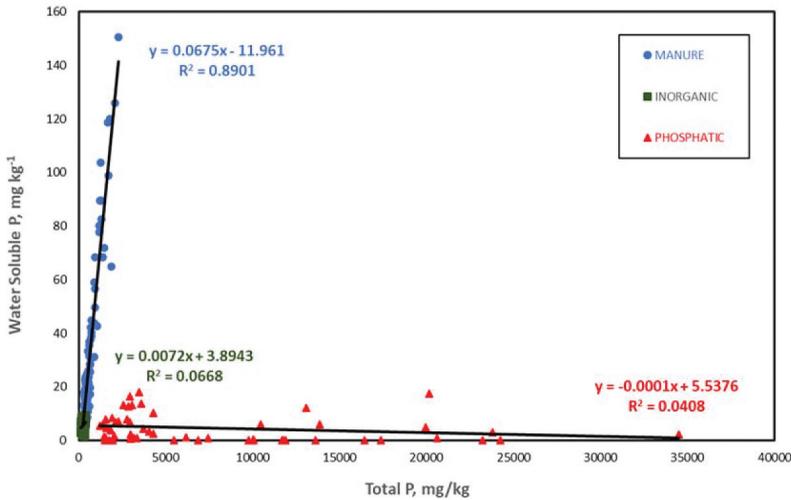


Figure 2. Water-soluble P (WSP) as a function of total P for phosphatic, manure-amended, and inorganically-fertilized soils including the 30 Florida Cooperative Soil Survey samples. Differences in slope for the relations were statistically significant (Table 2).

(2,275 mg kg⁻¹) had a WSP value of 151 mg kg⁻¹. In effect, very high values of soil-test or TP are apparently not a reliable indicator of high P leachability for phosphatic soils, to the extent that WSP is a gauge of the latter. The P in phosphatic soils is mainly geologic rather anthropogenic. Weathering of these soils on old geomorphic surfaces has fostered the in-situ accumulation of P forms that are more recalcitrant than those of anthropogenic sources. Further research

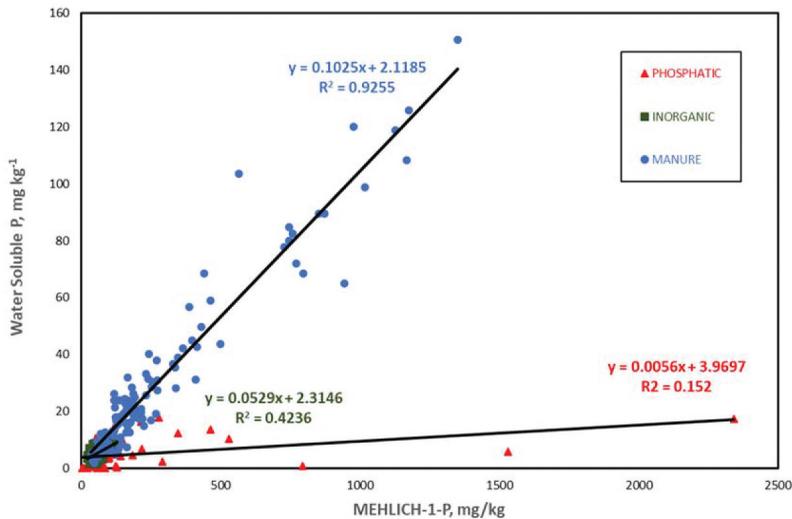


Figure 3. Water-soluble P (WSP) as a function of Mehlich-1 P for phosphatic (including 30 Florida Cooperative Soil Survey samples), manure-amended, and inorganically-fertilized soils. Differences in slope for the relations were statistically significant (Table 2).

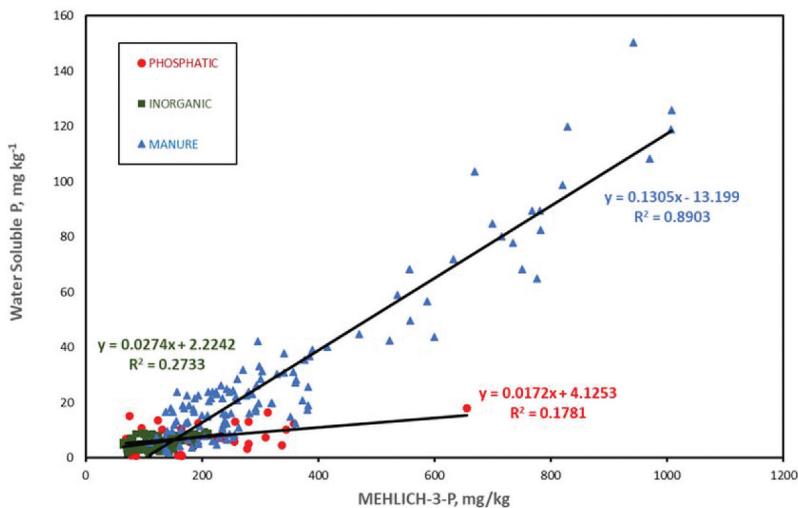


Figure 4. Water-soluble P (WSP) as a function of Mehlich-3 P for phosphatic (including 30 Florida Cooperative Soil Survey samples), manure-amended, and inorganically-fertilized soils. Differences in slope for the relations were statistically significant with exception of the inorganically-fertilized versus phosphatic comparison (Table 2).

is necessary to determine the extent to which these native P sources provide sufficient P for agronomic crops. That sufficiency may relate to the mineral form of phosphate in these geologically phosphatic soils and to the crop in question.

Several states in the USA in general and the southeastern states in particular, use M-1 and M-3 as common soil extractants in the labs (Zhang et al. 2014). The M1 extractant is especially suited to acidic and low organic matter soils of the southeastern US (Mylavarapu et al. 2002) but in recent years a number of laboratories in the southeastern and other parts of the USA have adopted M3 as the official method of extraction, replacing M1 and other methods, because M3 is suitable for a wider soil

Table 2. Slope comparison between P sources and soluble or extractable P.

Extraction Comparison*	Slope Comparison by P Source	Estimate	Standard Error	t Value	Pr > t
WSP vs. M-1	Fert vs Manure	-0.0495	0.0203	-2.44	0.0155
	Fert vs Phosphatic	0.0473	0.0203	2.33	0.0205
	Manure vs Phosphatic	-0.0968	0.0027	35.24	<0.0001
WSP vs. M-3	Fert vs Manure	-0.1031	0.0160	-6.42	<0.0001
	Fert vs Phosphatic	0.0101	0.0188	0.54	0.5913
	Manure vs Phosphatic	0.1132	0.0106	10.65	<.0001
WSP vs. TP	Fert vs Manure	-0.0603	0.0087	-6.91	<0.0001
	Fert vs Phosphatic	0.0073	0.0086	0.85	0.3956
	Manure vs Phosphatic	0.0676	0.0016	-41.55	<0.0001

*WSP = water soluble phosphorus; M-1 = Mehlich-1; M-3 = Mehlich-3; TP = Total Phosphorus

pH range when compared to M1, and can also be used to simultaneously extract multiple elements (Mylavarapu, Mitchell, and Savoy 2014), using inductively coupled plasma (ICP) techniques for efficiency.

Mehlich-1 and M-3 for manure-amended and inorganically fertilized soils showed a close relation between P release and release of other elements (Tables 3 and 4). However, the relations differed between the two sources. Manure-amended soils were distinguished in showing Mg to have the strongest associated release with P in the case of both M-1 and M-3 extractions (Table 3). Magnesium is used as a dietary supplement in dairy cow nutrition (Herrera et al. 2010; Schonewille 2013) and has been reported for dairy manure amended Florida soils (Josan et al. 2005). Manure-derived Mg-associated P is likely to be much more soluble than the P associated with indigenous Al and Fe soil components. This is consistent with the result that inorganically fertilized soils showed P release to be more strongly associated with Al and Fe release. Aluminum- and Fe-oxides (to included hydroxides and oxy-hydroxides) are components with a high affinity for P sorption and that would tend to be more prevalent than Ca-bearing minerals in most weathered humid region soils. Humphreys and Pritchett (1971) working with seven sandy soils, described the reaction products after phosphate application. Under field conditions, they found Fe-P and Al-P to be the dominant fractions of all P retained by the soil 7–11 years after the application of inorganic fertilizer.

Table 3. Multivariable regression analysis for $P = \text{fn}(\text{Al}, \text{Fe}, \text{OC})$ for Mehlich-1 (M-1) and Mehlich-3 (M-3) extractions of inorganically fertilized soils.*

Variable	Partial R-Square	Model R-Square	Pr > F
M-1 Mg	0.78	0.78	<0.0001
M-1 Al	0.07	0.85	<0.0001
M-1 Ca	0.03	0.88	<0.0001
M-1 Fe	0.01	0.89	0.0005
M-3 Mg	0.69	0.69	<0.0001
M-3 Al	0.08	0.77	<0.0001
M-3 Ca	0.03	0.80	<0.0001
TC	0.02	0.82	0.002
M-3 Fe	0.01	0.83	0.003

*No other variable met the 0.5 significance level for entry into the model.

Table 4. Multivariable regression analysis for $P = \text{fn}(\text{Al}, \text{Fe}, \text{Ca}, \text{Mg} \text{ and } \text{OC})$ for Mehlich-1 (M-1) and Mehlich-3 (M-3) extractions of manure-amended soils.*

Variable	Partial R-Square	Model R-Square	Pr > F
M-1 Al	0.57	0.57	<0.0001
M-1 Fe	0.04	0.61	0.0005
M-3 Al	0.66	0.66	<0.0001
M-3 Fe	0.11	0.77	<0.0001

*No other variable met the 0.5 significance level for entry into the model.

Mean M-1and M-3 P concentration observed in each source type was well above the agronomic optimum value of 30- and 45 mg kg⁻¹, respectively, according to UF-IFAS interpretations for agronomic crops (Mylavarapu et al. 2020; SERA-6 2013). This was the consequence of a site selection strategy intended to assure effects of the 3 P sources targeted for study.

Conclusions

The relationships between WSP and soil P extraction procedures for making agricultural management decisions vary with the following P sources: manure-amended-, inorganically fertilized-, and naturally phosphatic soils. Total P, soil test P, and WSP ranged to much higher levels for manure-amended soils than for fertilizer-amended soils. This buildup is probably due to the lower solubility of P forms in manure as compared to highly soluble commercial P fertilizers. Although P contents were multiple-fold higher, for phosphatic soils the corresponding WSP showed no significant increase with increasing TP and soil test P values, in contrast to manure-amended soils. The phosphate minerals in phosphatic soils do not likely constitute the same extractable P pool as for agriculturally amended soils. Hence, soil test extractions may not be well calibrated for phosphatic soils. Further research is necessary to determine the extent to which these native P sources provide sufficient P for agronomic crops, which may relate to the mineral forms of phosphate (e.g. Ca-P vs. Al-P forms) in these geologically phosphatic soils and to the crop in question. Very high soil-test- and TP values are apparently not a reliable indicator of high P leachability for phosphatic soils, to the extent that WSP is related to leaching risk.

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Disclosure statement

No potential conflict of interest was reported by the author(s).

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