

Total extractable phosphorus in flooded soil as affected by struvite and other fertilizer-phosphorus sources

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Abstract

A sustainable P source is imperative to ensure that food production can supply a growing global population. Wastewater-recovered struvite ($\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$) has emerged as an attractive option because of the ability to recover P from waste streams. This study aimed to evaluate total extractable soil P from electrochemically precipitated struvite (ECST) compared with other fertilizer-P sources [chemically precipitated struvite (CPST), diammonium phosphate (DAP), and rock phosphate] in two soil textures (two different silt loams and a silty clay loam) over time in a flooded soil environment. An equivalent fertilizer rate of $24.5 \text{ kg P ha}^{-1}$ was used. The change in water-soluble (WS) and Mehlich-3 (M3)-extractable nutrient concentrations (P, K, Ca, Mg, and Fe) from their initial concentrations was determined five times over a 4-mo period. After 0.5 mo, WS-P increased the most from the initial value with DAP (27.6 mg kg^{-1}), which did not differ from CPST or ECST. After 0.5 mo, M3-P increased the most in ECST (82 mg kg^{-1}), which did not differ from DAP. After 1 mo and thereafter under flooded conditions, M3-P increased the most from the initial value and was similar among ECST, CPST, and DAP. After 3 and 4 mo, WS-P was greater than the initial value in DAP only, but remained similar to CPST, ECST, and rock phosphate, which did not differ from the initial value. Comparable WS- and M3-P concentrations among ECST, CPST, and DAP under flooded conditions support struvite's agronomic potential as a prospective sustainable fertilizer-P source.

1 | INTRODUCTION

Despite the well-established potential consequences of excess phosphorus (P) in aquatic systems, P remains one of the most important nutrients for plant growth and agricultural

production, as plants use P to perform many physiological and biochemical functions. In upland terrestrial ecosystems, many soil properties affect the concentration and form of P in the soil, such as soil pH, clay and organic matter concentrations; cation exchange capacity; initial P concentrations; and exchangeable soil Fe, Ca, and Al concentrations (Nascimento, Pagliari, Faria, & Vitti, 2018), which often rapidly limits P availability to plants when fertilizer-P additions are made. Low plant accessibility, often coupled with low solubility, frequently renders P a growth-limiting nutrient in the environment, particularly in soils managed for optimal crop

Abbreviations: CPST, chemically precipitated struvite; DAP, diammonium phosphate; ECST, electrochemically precipitated struvite; ICAPS, inductively coupled argon-plasma spectrometry; M3, Melich-3; RP, rock phosphate; SiCL, silty clay loam; SiL 1, Calloway silt loam; SiL 2, Henry silt loam; SOM, soil organic matter; TR, total recoverable; UC, unamended control; WS, water-soluble; WWTP, wastewater treatment plant.

productivity (Cordell & White, 2011; Le Corre, Valsami-Jones, Hobbs, & Parsons, 2009). Consequently, to overcome the limited availability and low solubility of soil P, synthetic fertilizer-P sources are used maximize crop production.

The majority of synthetic fertilizer-P sources are derived from mined rock phosphate (RP), which is limited in supply in the environment. Rock phosphate reserves are unevenly distributed around the world, with approximately 80% of RP deposits existing in Morocco, China, South Africa, and the United States. Additionally, roughly 95% of the global phosphate production is currently used in agricultural applications (Desmidt et al., 2015). Increased food production throughout the 20th century has increased the demand for fertilizer-P sources and it is predicted that the amount of cost-effective, feasibly mined RP could be exhausted in as little as 100 yr (Liu, Kumar, Kway & Ra, 2012). Because of the potential food security concerns with the current global P system, alternative sustainable fertilizer-P sources have recently become an area of ongoing research.

Wastewaters have long been considered a potential nutrient source, as ancient civilizations used wastewater in agriculture for thousands of years (Jaramillo & Restrepo, 2017). Because of the large concentrations of P and N in wastewater effluent streams, wastewater treatment plants (WWTPs) remain a potential nutrient source that could be utilized in new P-recovery technologies. Additionally, government-mandated standards in the United States require WWTPs to remove no less than 90% of P from wastewater effluent (USEPA, 2011). However, the majority of the P removed from wastewater effluent is incorporated into the solid fraction via sewage sludge, which, for a variety of reasons, is undesirable for land application and use for food production (Desmidt et al., 2015). Introducing P recovery technology in WWTPs has the potential to considerably reduce the volume of sewage sludge, thus significantly reducing operational costs for WWTPs and providing a potential process for improved P extraction and reuse (Doyle & Parsons, 2002; Woods, Sock & Daiger, 1999).

Intentional struvite precipitation has become one popular method of P recovery in recent decades (Hertzberger, Cusick, & Margenot, 2020; Huygens & Saveyn, 2018). Struvite ($\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$) is a white, crystalline, solid material comprised of equal molar concentrations of magnesium (Mg^{2+}), ammonium (NH_4^+), and phosphate (PO_4^{3-}) (Hertzberger et al., 2020; Schoumans et al., 2015). Struvite recovery has gained popularity because of the ability to simultaneously remove both P and N from solid and liquid wastes, and because of struvite's slow-release fertilizer properties under aerobic soil conditions (Degryse, Baird, da Silva, & McLaughlin, 2016; Nascimento et al., 2018; Tallboys et al., 2016). Struvite recovery has been applied to a number of waste sources, including industrial wastewater (Diwani, Rafie, Ibiari, & El-Aila, 2007), dairy wastewater (Massey, Davis, Sheffield, & Ippolito, 2007), sewage sludge (Münch

Core Ideas

- Phosphorus and N recovery from wastewater could provide nutrients to agricultural soils
- Electrochemically precipitated struvite behaved similarly to other P sources in flooded soil
- Electrochemically precipitated struvite could be an alternative P source in flooded soil

& Barr, 2001), and semiconductor wastewater (Ahmed, Shim, won, & Ra, 2018; Ryu, Lim, Kim, Kim & Lee, 2012). Furthermore, on the basis of struvite's chemical composition, struvite has been considered a potential alternative fertilizer-P source, which could provide relief to the global dependence on traditional fertilizer-P sources derived from RP.

Though rapid fixation and low solubility often render P a limiting nutrient for optimal crop production in upland soils, P's behavior differs substantially in lowland settings, such as with flood-irrigated rice (*Oryza sativa* L.) production. Lowland rice production, particularly in the mechanized United States, is accompanied by establishment of a flood, often 10 to 15 cm thick, which is maintained for 3 to 4 mo during the growing season. Early in the flooded period, the soil water content quickly achieves saturation, decreasing the oxygen concentration in the soil, hence shifting the oxidation–reduction potential to reducing conditions (Brye, Rogers, Smartt, & Norman, 2013). Achievement of reducing conditions in flooded soils causes oxidized iron (Fe^{+3}), which is ubiquitous in soils and is responsible for abundant P binding in acidic soil conditions, to be reduced to Fe^{+2} . Reduced Fe^{+2} , which is highly soluble, consequently releases once chemically bound P into the saturated, reduced soil environment, thereby increasing ambient soil-P concentrations and P availability and mobility in soils used for rice production. The different behavior of P between lowland and upland conditions requires an evaluation of alternative fertilizer-P sources as they are developed.

Although several studies have examined struvite's fertilizer potential in potted-plant and greenhouse studies (Ackerman, Zvomuya, Cicek, & Flaten, 2013; Bonvin et al., 2015; Hilt et al., 2016; Gong et al., 2018), fewer studies have examined struvite–soil interactions in fine-textured, agricultural soils with a history of row cropping and, to our knowledge, no studies have examined struvite–soil interactions under flooded soil conditions. The objective of this study was to evaluate net changes in total water-soluble (WS) and Mehlich-3 (M3)-extractable soil P over time from varying soil textures (two silt loams and a silty clay loam) amended with an electrochemically precipitated struvite (ECST) compared with other fertilizer-P sources [chemically precipitated struvite (CPST), diammonium phosphate (DAP), and rock phosphate (RP)] in a plant-free, flooded soil environment. Since much of the

previous research on struvite's slow-release characteristics has been conducted under aerobic soil conditions, it was hypothesized that ECST would have similar WS and M3-extractable P concentrations to the CPST material and DAP, and greater WS and M3-P concentrations than RP after 3 mo of flooded soil conditions. It was also hypothesized that the two struvite sources would have greater WS and M3-Mg concentrations than other fertilizer-P sources over time because of the initially greater Mg concentration in the struvite sources.

2 | MATERIALS AND METHODS

2.1 | Soil collection and characterization

Similar to Anderson, Brye, Gbur, Roberts, and Greenlee (2020a) and Anderson, Brye, Greenlee, & Gbur (2020b), several soils were collected to capture a range of chemical and physical soil properties from representative row-crop agricultural sites throughout Arkansas. A Dardanelle silty clay loam (SiCL)[fine-silty, mixed, superactive, thermic Typic Argiudolls; USDA-NRCS (2015)] was collected from the Vegetable Research Station near Kibler, AR. The Dardanelle soil had a previous history of soybean [*Glycine max* (L.) Merr.] production. A Calloway silt loam (SiL 1) [fine-silty, mixed, active, thermic Aquic Fraglossudalfs; USDA-NRCS (2015)], which had a recent history of a wheat (*Triticum aestivum* L.)–soybean rotation for the previous 15 yr, was collected from the Cotton Branch Experiment Station near Marianna, AR. A Henry silt loam (SiL 2)[coarse-silty, mixed, active, thermic Typic Fragiaqualfs; USDA-NRCS (2015)], which was cropped to a rice–soybean rotation for at least the previous 5 yr, was collected from the Pine Tree Research Station close to Colt, AR. After collection, the soils were manually moist-sieved through a 7-mm mesh sieve, air-dried in a greenhouse for nearly 2 wk, and stored in 20-L buckets.

Soil subsamples were prepared in triplicate for each soil for physical and chemical property analyses. Soil subsamples were oven-dried at 70 °C for 48 h, mechanically crushed, and sieved through a 2-mm mesh screen. Particle-size analyses were performed in triplicate via a modified 12-h hydrometer method to determine the distribution of clay, sand, and silt (Gee & Bauder, 1986). The soil organic matter (SOM) concentration was determined gravimetrically by weight-loss-on-ignition (Zhang, Hardy, Mylavarapu, & Wang, 2014). Electrical conductivity and soil pH were measured potentiometrically in a 1:2 (mass/volume) soil-to-water paste ratio (Sikora & Kissel et al., 2014). Total N and total C concentrations were measured by high-temperature combustion (VarioMax CN analyzer, Elementar Americas, Inc., Mt. Laurel, NJ)(Provin, 2014). Undisturbed bulk density was estimated for each soil by multiple regression analyses, as detailed by Saxton, Rawls, Romberger, & Papendick (1986), taking

measured clay, sand, and SOM concentrations and applying the soil water characteristics subroutine of the Soil–Plant–Atmosphere–Water Field and Pond Hydrology model (version 6.02.75)(USDA-NRCS, 2017). Subsamples were also used to determine the gravimetric water content of the air-dried soils. Air-dried subsamples were weighed, oven-dried at 70 °C for 48 h, and reweighed.

The initial extractable soil elemental concentrations were determined in triplicate for each of the three soils. A water extraction was performed with a 1:10 soil mass/water volume ratio, where soil suspensions were stirred for 1 hour, filtered through a 0.45-μm filter, and analyzed by inductively coupled argon-plasma spectrometry (ICAPS)(Spectro Arcos ICP, Spectro Analytical Instruments, Inc.)(Zhang et al., 2014) to determine the WS elemental concentrations (i.e., P, K, Mg, Ca, and Fe). A M3 extraction (Tucker, 1992) was conducted with a 1:10 (mass/volume) soil/extractant solution proportion to quantify the M3-extractable nutrient concentrations (i.e., P, K, Ca, Mg, and Fe) by ICAPS. The resulting ICAPS-measured WS and M3 concentrations represented total (i.e., organic plus inorganic) elemental concentrations in their respective extracts, as it was beyond the scope of this study to differentiate between organic and inorganic elemental fractions. A strong-acid digestion was also performed via USEPA Method 3050B (USEPA, 1996), with concentrated nitric acid, followed by heating, and samples were analyzed by ICAPS to determine total recoverable (TR) elemental concentrations (i.e., P, K, Mg, Ca, and Fe). Table 1 summarizes the initial physical and chemical properties of the three soils used.

2.2 | Fertilizer-P sources and analyses

Two sources of struvite were used in this study: (a) ECST generated from artificial wastewater by researchers in the Department of Chemical Engineering at the University of Arkansas (Kékedy-Nagy, Teymouri, Herring, & Greenlee, 2020) and (b) CPST referred to as Crystal Green (Ostara Nutrient Recovery Technologies, Inc.). Despite having similar basic chemical compositions constituting struvite, the CPST material was created from a wastewater treatment plant near Atlanta, GA, and thus was expected to contain a much more diverse composition that could potentially contribute to variations in dissolution dynamics than the much cleaner ECST that was created from synthetic wastewater. In addition to the two sources of struvite, two additional commercially available P fertilizer sources were used in the soil incubation experiment: RP and DAP.

Particle sizes of the various fertilizers in their raw forms differed among fertilizer-P sources and varied from powder to crystals to small pellets (i.e., prills). Diammonium phosphate and CPST were in pelletized form, whereas RP was in powdered form and ECST was in crystalline form. The

TABLE 1 Summary of initial soil properties ($n = 3$) among soils used in the soil incubation (adapted from Anderson et al., 2020a, 2020b)

Soil property	Soil		
	Dardanelle silty clay loam	Calloway silt loam	Henry silt loam
Sand, g g ⁻¹	0.07c ^a	0.12a	0.10b
Clay, g g ⁻¹	0.37a	0.14b	0.11c
Silt, g g ⁻¹	0.56c	0.74b	0.79a
pH	6.50b	6.53b	6.70a
Electrical conductivity, dS m ⁻¹	0.273a	0.169b	0.164b
Soil organic matter, g g ⁻¹	0.025a	0.024b	0.019c
Total C, g g ⁻¹	0.012a	0.011a	0.009b
Total N, g g ⁻¹	0.0011a	0.0011a	0.0008b
C/N ratio	11.4a	9.68c	11.0 b
Water-soluble, mg kg ⁻¹			
P	9.60a	5.47b	3.70c
K	44.7a	25.3c	28.3b
Ca	74.3a	62.7b	62.0b
Mg	28.0a	23.3b	17.7c
Fe	47.9a	47.9a	47.9a
Weak-acid-extractable, mg kg ⁻¹			
P	143a	33.7b	19.7c
K	485a	143b	158b
Ca	4,328a	1,842c	2,156b
Mg	774a	444b	365c
Fe	175c	186b	459a
Total-recoverable, mg kg ⁻¹			
P	672a	568b	297c
K	5,828a	1,525b	892c
Ca	4,463a	1,757c	2,006b
Mg	8,544a	2,429b	1,236c
Fe	27,880a	18,230b	14,297c

^aDifferent letters in a row indicate differences among soils at $P < .05$.

average diameters of the pelletized fertilizer-P sources were 2.9 ($\pm$ 0.60) mm for DAP and 2.9 ($\pm$ 0.57) mm for CPST (Anderson, 2020). Pelletized fertilizers and ECST were mechanically crushed and chemically characterized in powdered form to enable direct comparisons among fertilizer-P sources.

Similar to recent procedures by Anderson et al. (2020b), chemical analyses were conducted for each of the fertilizer-P sources. Five subsamples of each fertilizer-P source were used for chemical analyses. Fertilizer pH and electrical conductivity were determined potentiometrically in a 1:2 fertilizer mass/water volume ratio (Sikora & Kissel, 2014) for each fertilizer material except for ECST because of the relatively large mass required for these analyses and the limited supply of ECST that was available to use. Total N and total C concentrations were measured by high-temperature combustion (Elementar VarioMax CN Analyzer)(Provin, 2014).

Water-soluble elemental concentrations (i.e., P, K, Mg, Ca, and Fe) were determined in a 1:10 fertilizer mass/water volume ratio, where the mixture was agitated for 1 h, filtered through a 0.45- μ m filter, and analyzed by ICAPS to represent the relevant concentrations that would become environmentally available after interaction with rainwater. Plant-available nutrient concentrations were determined, in which, elemental concentrations (i.e., P, K, Ca, Mg, and Fe) were evaluated after M3 extraction using a 1:10 fertilizer mass/extractant volume ratio (Tucker, 1992) and analyzed by ICAPS (Zhang et al., 2014). A strong-acid digestion (USEPA, 1996) was conducted, with analysis by ICAPS, to evaluate the TR elemental concentrations (i.e., P, K, Ca, Mg, and Fe) that represented the maximum nutrient concentration that could become environmentally available. The chemical composition of the various fertilizer-P sources used in this study is summarized in Table 2.

TABLE 2 Summary of the initial chemical properties among fertilizer-P used in the incubation (adapted from Anderson et al., 2020b)

Fertilizer property	Fertilizer-P source			
	DAP	CPST	ECST	RP
pH	7.32	8.78	N/A ^b	6.67
EC, dS m ⁻¹	105	226	N/A	514
Organic matter, g g ⁻¹	0.321	0.259	0.227	0.021
Total C, g g ⁻¹	0.005	0.002	0.001	0.004
Total N, g g ⁻¹	0.181	0.057	0.033	0.0004
Water-soluble, mg kg ⁻¹				
P	163,300	216	3,680	70.6
K	1,173	1.50	3.03	28.5
Ca	153	11.6	4.08	148
Mg	79.9	157	1317	25.5
Fe	63.6	1.22	0.72	4.20
Weak-acid-extractable, mg kg ⁻¹				
P	164,349	24,479	39,701	638
K	1,244	230	45.7	139
Ca	228	83	0.3	3,602
Mg	507	21,444	33,683	338
Fe	146	127	0.2	226
Total recoverable, mg kg ⁻¹				
P	183,365	116,556	184,510	75,956
K	1,510	842	0.01	2,762
Ca	4,653	312	0.01	163,495
Mg	6,734	83,234	133,150	3,219
Fe	5,785	4,505	0.1	10,592

^aDAP, diammonium phosphate; CPST, chemically precipitated struvite; ECST, electrochemically precipitated struvite; RP, rock phosphate; EC, electrical conductivity; N/A, not applicable.

^bThe limited supply of ECST material prohibited the analysis of fertilizer pH and EC.

2.3 | Soil incubation experiment

The behavior of fertilizer-P sources was evaluated under flooded soil conditions. To isolate soil property–fertilizer interactions, plants and roots were excluded from this incubation experiment, as fertilizer-P dynamics are known to change to some extent depending on the rhizosphere environment, such as the pH changes that could affect P solubility and mobility. The soil incubation was conducted in a climate-controlled laboratory setting. Humidity and air temperature fluctuations were measured throughout the duration of the experiment with an Acurite thermometer (Model 00554SBDI, Chaney Instrument Co.). Similar to Anderson et al. (2020a), the flooded soil incubation was conducted over a 4-mo period from 20 June to 12 Sept. 2019.

For each of the five planned sampling intervals (i.e., 0.5, 1, 2, 3, and 4 mo) during the 4-mo incubation period, soil

cups were prepared in triplicate for each soil–fertilizer treatment combination. Approximately 150 g of air-dried soil were added to each plastic cup. One of the five fertilizer treatments [i.e., pelletized DAP (106.9 ± 10 mg), powderized RP (257 ± 10 mg), pelletized CPST (170.7 ± 10 mg), crystallized ECST (88.5 ± 10 mg), or an unamended control (UC)] was applied to each soil cup. Fertilizers were applied at a uniform total P rate of $56 \text{ kg P}_2\text{O}_5 \text{ ha}^{-1}$ ($24.5 \text{ kg P ha}^{-1}$). Though differing in concentration among fertilizer-P sources, Ca, N, and Mg concentrations were not controlled for in the incubation on account of there being no plant response to evaluate and no potentially confounding rhizosphere effects to account for. Furthermore, though the two struvite sources had the numerically largest Mg concentrations, even DAP and RP contained measurable extractable Mg (Table 2) to minimize the potential effects of nonuniform Mg concentrations.

After fertilizer addition, each soil cup was individually shaken for approximately 10 s in a circular and up-and-down motion to mix the fertilizers into the soil. After the fertilizers were incorporated, the soil cups were randomly distributed between a pair of three-level wooden shelf structures that were placed side-by-side in the laboratory. Soil cups were randomly distributed among the three levels of the two wooden shelves. The soil cups were loosely covered with lids with holes and were rotated among shelves every 2 wk to ensure that all soil cups underwent uniform environmental conditions (i.e., air-flow exposure and light) over the course of the 4-mo incubation period. In total, 225 soil cups were prepared for the soil incubation.

The soil cups were watered with two watering schemes, similar to those described in Anderson et al. (2020a). Over the first month of the incubation, a target mass was determined for each soil on the basis of a set gravimetric water content. The soil cups were initially watered on the same day as fertilizer incorporation, where the soil cups were moistened gravimetrically to a specific target mass with tap water. The target watering mass was derived from the estimated field moisture capacity for each soil from the Soil–Plant–Atmosphere–Water Field and Pond Hydrology model. The soil cups were rewetted to their target mass via the identical process after 2 wk of incubation. At 1 mo of incubation and thereafter, a flood was imposed on the remaining soil cups with tap water, where ~ 1 cm of ponded water was imposed and maintained in each soil cup. After the flood was imposed, one drop of algicide (API POND ALGAEFIX, Mars Fishcare North America, Inc.) was applied to each soil cup to prohibit algal growth, which was achieved, as no algal growth was observed throughout the duration of the incubation. Water levels were monitored regularly and soil cups were rewetted every 2 wk to maintain a 1-cm flood depth in each soil cup.

After the flood was imposed, the soil oxidation–reduction potential (mV) was measured in four random flooded

soil cups at 2.5 and 3.5 mo of incubation with a VWR Symphony SB80PC benchtop pH/conductivity meter and electrode (VWR International) to determine if reducing conditions were present in the soil cups. The millivolt measurements were made via an electrode that was positioned in the flood water, as close to the soil surface as possible. However, because of the variability between soil cups, measurements were highly variable and ranged from -69.4 to 62.4 mV.

Over the 4-month incubation period, the soil cups were destructively sampled after five incubation periods: 0.5, 1, 2, 3, and 4 mo. Water was poured out of each cup and discarded, and the soil was removed. The soil was oven-dried for 48 h in a forced-draft oven at 70°C , mechanically crushed, and sieved through a 2-mm mesh screen. Water-soluble and M3 analyses were performed, as previously described for the initial soil properties, to evaluate the extractable total nutrient concentrations (i.e., P, K, Ca, Mg, and Fe) over time. Soil pH was also measured, as previously described, after each sampling time. Because of struvite's initial Mg concentration, soil Mg was evaluated over time. Considering their reactivity with P, soil Ca and Fe concentrations were quantified over time. Because of K's large solubility and lower valence state, which allow K^{+} ions to potentially be replaced by other polyvalent cations (i.e., Ca, Mg, and Fe) on the exchange sites, soil K was also evaluated over time.

Over the course of the 4-mo incubation period, the ambient air temperature in the climate-controlled laboratory ranged from 21.0 to 22.5°C and averaged 21.6°C . The ambient relative humidity ranged from 52 to 58% and averaged 56.2%. In the laboratory, incubation cups received regular sunlight through a window in the laboratory and fluorescent lighting while lights were on during the day.

2.4 | Statistical analyses

Based on a completely random design, a one-factor ANOVA was conducted with PROC GLIMMIX in SAS (Version 9.4, SAS Institute Inc.) to evaluate the effect of soil (i.e., SiCL, SiL 1, and SiL 2) on WS, M3, and TR soil elemental (i.e., P, K, Mg, Ca and Fe), total C, total N, and SOM concentrations, and pH. Based on a split-split-plot, randomized experimental design, a three-factor ANOVA was conducted with PROC GLIMMIX to evaluate the effects of soil (i.e., SiCL, SiL 1, and SiL 2), fertilizer treatment (i.e., DAP, RP, CPST, ECST, and UC), time (i.e., 0.5, 1, 2, 3, and 4 mo), and their interactions on the change in soil pH and WS and M3 elemental concentrations (i.e., P, K, Mg, Ca, and Fe) from their initial magnitudes. The split-split-plot factor was time, the split-plot factor was fertilizer amendment, and the whole-plot factor was soil. When appropriate, means from all analyses were separated by LSD at the 0.05 level.

3 | RESULTS AND DISCUSSION

3.1 | Change in soil pH

It is well known that soil pH often dictates nutrient availability. The change in soil pH, averaged over time, differed among fertilizer amendments across soils ($P < .05$; Table 3). Although the change in soil pH differed between soils and among fertilizer amendments, the change in soil pH was generally impacted more by the different soils used in the incubation and, subsequently, the different initial chemical properties and soil management histories.

Soil pH generally increased from the initial pH in all fertilizer treatments in the SiCL and SiL 2 soils (Figure 1). However, the magnitude of change in soil pH was approximately two times greater in all fertilizer treatments in SiL 2 than in SiCL (Figure 1). In the SiL 2 and SiCL soils, soil pH increased from the initial pH the most in the UC treatment (0.71 and 0.22 pH units, respectively), which generally did not differ from the RP, CPST, and ECST treatments, except for in the CPST–SiL 2 combination, in which the soil pH increased from the initial pH but the measured increase was less than that in the UC treatment (Figure 1). The overall greater pH increase in the SiL 2 soil was probably related to lower SOM and clay concentrations, which were less effective at buffering a change in pH in combination with the previously flooded history of SiL 2. The greater concentration of M3-Fe associated with SiL 2 was likely to be reduced to the more soluble Fe^{2+} , which has been shown to increase soil pH (Mitsch & Gosselink, 2000).

An acidifying effect was observed with the DAP treatment in the SiL 1 and SiCL soils, where soil pH decreased from the initial value by 0.47 and 0.08 pH units, respectively (Figure 1). The pH increased from the initial value in the SiL 2 soil, although the change in pH was considerably less than under all other treatments (Figure 1). The acidifying effect of DAP was probably related to the greater N concentration in the DAP fertilizer, in which more microbial nitrification probably occurred in the SiL 1 and SiCL soils than in the SiL 2 soil because of the lower SOM concentration of the SiL 2 soil (Anderson et al., 2020b; Vaneeckhaute, Janda, Vanrolleghem, Tack, & Meers, 2016).

The change in soil pH, averaged across soils, also differed among fertilizer amendments over time ($P < .05$; Table 3). Similar to the results of Anderson et al. (2020a), the change in soil pH was significantly affected by the imposition of saturated or flooded conditions after 1 mo of incubation. After 0.5 mo of incubation, the soil pH had increased from the initial value in all treatments, but increased numerically the most in the CPST and UC treatments (0.32 and 0.31 pH units, respectively; Figure 1). After 1 mo of incubation, soil pH generally decreased from the initial value in all treatments but decreased

TABLE 3 ANOVA summary of the effects of soil (S), fertilizer amendment (A), sample time (T), and their interactions on the change in soil test pH, water-soluble (WS), and Mehlich-3 (M3)-extractable nutrient (P, K, Ca, Mg, and Fe) concentrations from the initial soil values for the soil incubation

Source of variation	<i>P</i> value										
	Δ pH	Δ WS P	Δ WS K	Δ WS Ca	Δ WS Mg	Δ WS Fe	Δ M3-P	Δ M3-K	Δ M3-Ca	Δ M3-Mg	Δ M3-Fe
S	<.01	.57	<.01	.96	.03	<.01	<.01^a	.03	.01	.04	<.01
A	<.01	<.01	<.01	<.01	<.01	.56	<.01	.66	.32	.10	.68
T	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01	<.01
S $\times$ A	<.01[†]	1.00	.22	.94	.94	.06	.11	.89	.83	.98	.76
S $\times$ T	<.01	.80	<.01	<.01	<.01	<.01	.49	<.01	<.01	<.01	<.01
A $\times$ T	<.01	<.01	.86	.75	.07	.57	.05	.45	.34	<.01	<.01
S $\times$ A $\times$ T	.92	.89	1.00	1.00	.99	.30	.33	.98	.46	.81	.09

^aBold values are considered significant and are reported at $P < .05$.

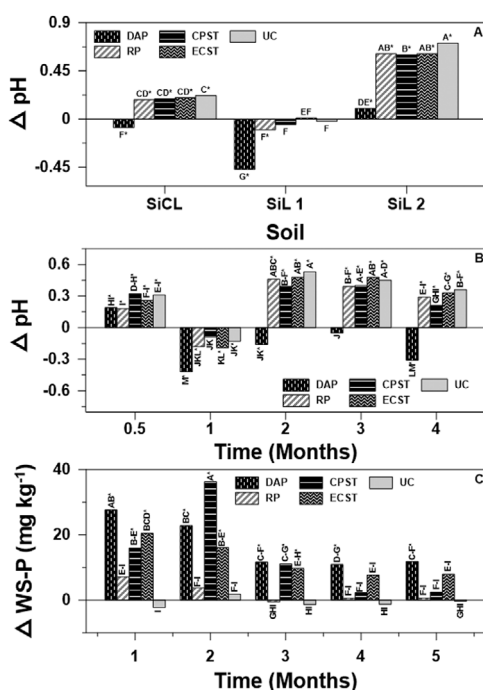


FIGURE 1 Effects of fertilizer amendment [diammonium phosphate (DAP), rock phosphate (RP), chemically precipitated struvite (CPST), electrochemically precipitated struvite (ECST), and the unamended control (UC)], averaged over time, on the change in soil pH from the initial value among soils (a) (SiCL, silty clay loam; SiL, silt loam) and among the fertilizer amendment effects, averaged over soils, on the change in (b) soil pH and (c) water-soluble (WS) P concentrations from the initial values (Table 1) over time. Means within a panel with different letters are different ($P < .05$). An asterisk (*) indicates that the mean value is different from zero ($P < .05$)

more in DAP (0.42 pH units) than in any other treatment (Figure 1). The general acidifying effect that was present by 1 mo of incubation was probably caused by the dissolution of fertilizers and the influx of various cations that caused a

displacement of H^+ ions on exchange sites in the moist soil conditions (Montalvo, Degryse, & McLaughlin, 2014; Nascimento et al., 2018). Moreover, the less drastic pH change in CPST, ECST, and RP was probably related to the slower dissolution rate of all three fertilizer materials than that of DAP, which, according to visual observations during sample cup deconstruction, exhibited more complete dissolution earlier in the incubation. Although the original prill diameters differed slightly between DAP and CPST, which probably had a minimal effect on prill dissolution, the powder, crystalline, and pellet forms of the raw fertilizer materials used had varying surface areas, which could have differentially affected their solubilities and reactivities. However, unifying the form and particle size across fertilizer materials for the purposes of this incubation experiment would have rendered the results even less agronomically relevant than using the fertilizer materials in their original form, which is what would be done in the field by a producer.

After 1 mo of flooded conditions (i.e., 2 mo of incubation), soil pH generally increased from the initial pH in all treatments, except for DAP, which decreased from the initial pH (Figure 1). After 3 mo of incubation, soil pH generally increased from the initial value, but the increase was generally less after 3 mo than after 2 mo of incubation. After 4 mo of incubation, soil pH increased the most from the initial pH in the UC treatment (0.36 pH units), which did not differ from the ECST (0.33 pH units) and RP (0.29 pH units) treatments (Figure 1). Additionally, after the 4-mo sampling point, soil pH was again lower from the initial value in the DAP treatment by 0.31 pH units (Figure 1).

Fertilizers such as monoammonium phosphate and triple superphosphate, which contain P in the form of phosphate ($H_2PO_4^-$), can have an acidifying effect in alkaline soils (Fertiliser Technology Research Centre, 2015), which probably did not occur because of the slightly acidic soils used in the incubation (Table 1). Consequently, a more likely, and at least

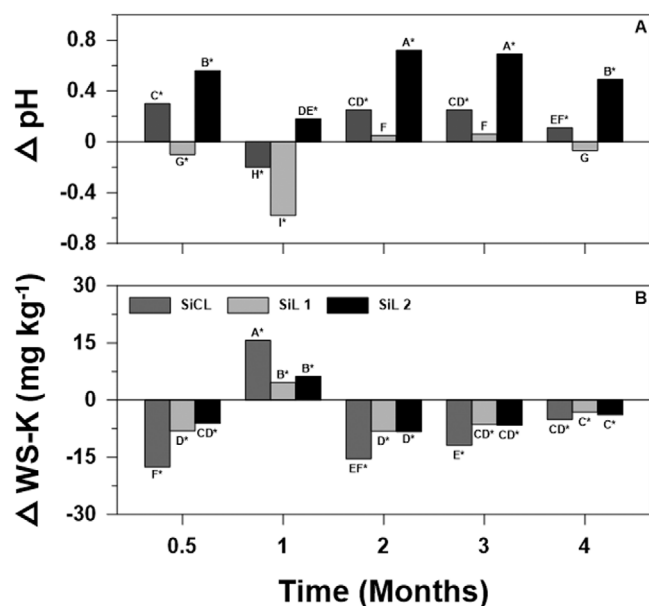


FIGURE 2 Effects of soil (SiCL, silty clay loam; SiL, silt loam), averaged over fertilizer amendments, on the change in (a) soil pH and (b) water-soluble (WS) soil K concentrations from the initial value (Table 1) over time. Means within a panel with different letters are different ($P < .05$). An asterisk (*) indicates that the mean value is different from zero ($P < .05$).

partial, explanation for the soil pH differences among fertilizers was related to the different carrier cation (i.e., Ca^{2+} , Mg^{2+} , and NH_4^+) affinities of the fertilizers. The fertilizer carrier cations could all displace H^+ from the exchange sites and at potentially different concentrations (Anderson et al., 2020b; Montalvo et al., 2014; Nascimento et al., 2018). Furthermore, soil pH was also probably, at least partly, affected by the different fertilizer compositions and forms of P in the fertilizer themselves (i.e., H_2PO_4^- and HPO_4^{2-} ; Fertiliser Technology Research Centre, 2015). Though not controlled for in the experiment, Ca, N, and Mg concentrations varied among fertilizer-P sources (Table 2), probably affecting exchange-site equilibria with the soil solution, which, in turn, influenced the measured soil pH trends. Though beyond the scope of this study to address, fertilizer-induced changes in soil pH may have also led to differences in organic and inorganic P fractionation, which may have further influenced soil pH trends and differences among fertilizer-P sources.

The change in soil pH, averaged across fertilizer amendments, also differed across soils over time ($P < .05$; Table 3). After 0.5 mo of incubation, soil pH increased from the initial value in the SiCL (0.30 pH units) and SiL 2 (0.56 pH units) soils and decreased from the initial value in the SiL 1 soil by 0.10 pH units (Figure 2), which may have been caused by differences in M3- and TR Fe concentrations among the soils (Table 1). After 1 mo of incubation, soil pH decreased from the initial value in the SiCL and SiL 1 soils, whereas the soil

pH had increased from the initial value in the SiL 2 soil (Figure 2). The imposition of flooding after 1 mo of incubation led to a drastic change in soil pH in all soils in the subsequent sampling intervals, which was probably related to the partly reduced conditions of the flooded soil cups. After 2 mo of incubation, soil pH increased from the initial value in the SiCL and SiL 2 soil, but was similar to the initial pH in SiL 1 (Figure 2). The pH increase was probably caused by the general effect of flooding previously drained soils, which causes an increase in pH in acidic soils through the reduction of Fe^{3+} in the soil (Mitsch & Gosselink, 2000). After 2 mo of incubation, soil pH decreased slightly in all soils but was still greater than the initial pH in the SiCL (0.11 pH units) and SiL 2 (0.49 pH units) soils at the 4-mo sampling point, whereas soil pH did not differ from the initial value in the SiL 1 soil (Figure 2).

3.2 | Change in WS soil nutrient concentrations

The change in WS P concentrations, averaged across soils, differed among fertilizer amendments over time ($P < .05$; Table 3). After 0.5 mo of incubation, WS P concentrations had generally increased from the initial value in all treatments, but increased the most in the DAP treatment (27.6 mg kg⁻¹), which did not differ from CPST and ECST (15.9 and 20.4 mg kg⁻¹, respectively; Figure 1). Despite the reported slow-release properties of struvite, both CPST and ECST had similar WS P concentration increases to DAP over the first 0.5 mo of the incubation, which was somewhat unexpected because of the greater solubility and P availability of DAP. After 1 mo of incubation, WS P concentrations generally decreased numerically in all treatments, except for CPST, which had approximately double the WS P increase from the 0.5-mo sampling point (Figure 1). The large WS P increase in the CPST treatment at the 1-mo sampling point was probably caused by the incorporation of one or more undissolved fertilizer pellets in the soil sample upon destructive sampling, which was also reported by Anderson et al. (2020b). After 2 mo of incubation, WS P concentrations had at least numerically decreased in all treatments from earlier in the incubation, as the WS P was converted to less soluble P forms, but were still greater than the initial value and were similar among the DAP, CPST, and ECST treatments (Figure 1). The decrease in WS P concentrations at the 2-mo sampling time point may have been partly related to the flooded soil conditions that were imposed and the dilution effect caused by the flood water in each soil cup, which was ultimately removed upon destructive sampling. The WS P concentrations generally remained relatively consistent between 2 and 4 mo of incubation, except for CPST, in which WS P concentrations decreased after the 2-mo interval. After 4 mo of incubation, WS P

concentrations were still greater than the initial value in DAP (11.8 mg kg^{-1}) and did not differ from the initial value in RP, CPST, and ECST (0.57 , 2.40 , and 7.86 mg kg^{-1} , respectively) treatments; however, the change in WS P was similar among all fertilizer treatments after 4 mo of incubation (Figure 1).

The change in WS K, Ca, Mg, and Fe concentrations, averaged across fertilizer amendments, differed among soils over time ($P < .05$; Table 3). After 0.5 mo of incubation, WS K concentrations had decreased from the initial value in all soils, but decreased the most in the SiCL soil (-17.6 mg kg^{-1} ; Figure 2). After 1 mo of incubation, soil WS K concentrations had increased from the initial value in all soils and had increased more in the SiCL soil than in either SiL soil. The increase was probably caused by the simultaneous addition of the various carrier cations from each P fertilizer material, such as Mg^{2+} , Ca^{2+} , and NH_4^+ , which may have affected exchangeable K ions in the solid phase of each soil (Montalvo et al., 2014; Nascimento et al., 2018). Additionally, the greater WS K concentration increase in the SiCL soil between 0.5 and 1 mo of incubation was probably caused by a possibly greater cation exchange capacity and the greater concentration of K ions that could become available. After imposition of the flood, WS K concentrations decreased from the initial value in all soils after 2 mo of incubation (Figure 2). The dilution effect of the additional water in the flooded soil cups was again likely to have reduced the WS K concentrations in all soils throughout the duration of the flood. Between 2 and 4 mo of incubation, WS K concentrations increased only slightly but remained below the initial value in all soils by the 4-mo sampling point (Figure 2). After 4 mo of incubation, WS K concentrations had decreased the least in the SiL 1 and SiL 2 soils (-3.24 and -3.86 mg kg^{-1} , respectively), which did not differ from each other.

The change in WS Ca concentrations was affected the most by the imposition of the flood after 1 mo of incubation (Figure 3). After 0.5 mo of incubation, WS Ca concentrations increased from the initial value in the SiL 2 soil (23.7 mg kg^{-1}), did not differ from the initial value in the SiL 1 soil (19.5 mg kg^{-1}), and decreased from the initial value in the SiCL soil (-4.15 mg kg^{-1} ; Figure 3). After 1 mo of incubation, WS Ca concentrations increased and were greater than the initial value in all soils, yet the greatest numeric increase occurred in the SiL 1 soil (Figure 3). The increase in WS Ca concentration among all soils over the first month of the incubation was probably caused by the influx of Ca from the Ca-containing fertilizers in combination with other cations from the fertilizers that may have displaced lower-valence cations from the exchange sites, thus influencing the available WS Ca (Anderson et al., 2020b). After the imposition of the flood, WS Ca concentrations decreased significantly and did not differ from the initial value in all soils after the 2-mo sampling point (Figure 3). The decrease in WS Ca after imposition of the flood corresponded with at least numerically decreased

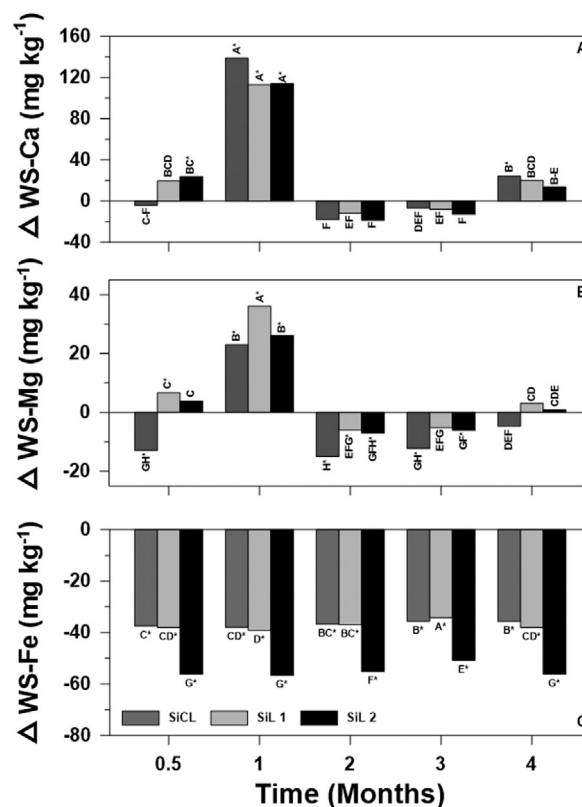


FIGURE 3 Effects of soil, averaged over fertilizer amendments, on the change in water-soluble (WS) soil (a) Ca, (b) Mg, and (c) Fe concentrations from the initial values (Table 1) over time. Means within a panel with different letters are different ($P < .05$). An asterisk (*) indicates that the mean value is different from zero ($P < .05$)

WS P and increased pH (Figure 1), suggesting the formation of insoluble calcium phosphates. Similar to the change in WS K concentrations, WS Ca concentrations increased only slightly after 3 mo of incubation but did not differ from the 2-mo sampling point in each soil. After 4 mo of incubation, WS Ca concentrations did not differ from the initial value in the SiL 1 or SiL 2 soils (20.1 and 13.7 mg kg^{-1} , respectively), yet had increased from the initial value in the SiCL soil (24.2 mg kg^{-1} ; Figure 3).

The change in WS Mg concentrations generally followed a similar trend to the change WS K and Ca concentrations. After 0.5 mo of incubation, WS Mg concentrations increased from the initial value in the SiL 1 soil (6.55 mg kg^{-1}), did not differ from the initial value in the SiL 2 soil (3.82 mg kg^{-1}), and decreased from the initial value in the SiCL soil (-12.9 mg kg^{-1} ; Figure 3). After 1 mo of incubation, WS Mg concentrations increased in all soils because of the influx of more Mg from the ECST and CPST materials, as well as the probably greater concentration of available base cations resulting from the pH decrease that occurred in all treatments at the 1-mo sampling point (Mengel, 1993). After the imposition of the flood at 1 mo of incubation, WS Mg concentrations decreased

substantially by 2 mo of incubation and decreased from the initial value in all soils at least partly because of the dilution effect of the additional water in the soil cups (Figure 3). Additionally, after 2 mo of incubation, the greatest decrease in WS Mg concentration occurred in the SiCL soil (-15.0 mg kg^{-1} ; Figure 3). Similar to WS Ca, WS Mg concentrations did not differ between 2 and 3 mo of incubation in each soil. Compared with after 3 mo, a slight increase in WS Mg concentrations occurred in all soils after 4 mo of incubation; however, the WS Mg concentrations were similar among soils and did not differ from the initial value in all soils (Figure 3).

Unlike the change in other WS elements, the change in WS Fe concentration across soils was not substantially impacted by the imposition of saturated or flooded conditions and only marginally ($\pm 6 \text{ mg kg}^{-1}$) changed over time (Figure 3). After 0.5 mo of incubation, WS Fe concentrations decreased from the initial value in all soils but decreased the most in the SiL 2 soil (-56.2 mg kg^{-1} ; Figure 3). The general decrease in WS Fe concentrations was probably caused by the continued fixation of Fe^{3+} to clays and the relatively insoluble reaction with phosphate from the fertilizers over time, which was similar to what was reported by Anderson et al. (2020a). The WS Fe concentrations slightly increased after 2 and 3 mo of incubation but still decreased from the initial value in all soils and decreased the most in the SiL 2 soil at each sampling interval (Figure 3). The slight increase in WS Fe concentration between the 2- and 3-mo sampling points was probably related to small concentrations of Fe^{3+} being reduced to Fe^{2+} , as reducing conditions were often measured during the same time interval; however, large variability ($\pm 60 \text{ mV}$) existed in the redox potential measurements. After 4 mo of incubation, the WS Fe concentrations generally decreased and were similar to each soil's 0.5-mo sampling values in both the SiL 1 and SiL 2 soils (-37.9 and -56.2 mg kg^{-1} , respectively; Figure 3). Additionally, after 4 mo of incubation, WS Fe concentrations did not change from the 3-mo sampling point in the SiCL soil (-35.6 mg kg^{-1}). One of the intended responses of the imposition of the flood was to create reducing conditions in the soil. However, it is likely that complete reducing conditions were not achieved, as the redox potential measurements did not consistently demonstrate reduced conditions across all soil cups and treatment replications. In truly anaerobic soil environments, Fe concentrations have been shown to increase as a result of Fe^{3+} reducing to the soluble Fe^{2+} form (Banach et al., 2009), yet only a small concentration of Fe^{3+} was probably reduced.

The change in WS K, Ca, and Mg concentrations, averaged across soil and time, also differed among fertilizer amendments ($P < .05$; Table 3). The WS K concentrations did not differ from the initial value in DAP (-1.4 mg kg^{-1}) and decreased from the initial value in CPST (-4.2 mg kg^{-1}) and ECST (-5.4 mg kg^{-1}), which did not differ from each other. In addition, WS K concentrations decreased the most from

the initial value in the UC treatment (-7.2 mg kg^{-1}), which did not differ from RP and ECST. The WS Ca concentrations generally increased from the initial value in all fertilizer treatments but increased the most from the initial value in DAP (44.3 mg kg^{-1}), followed by CPST and ECST (28.0 and 24.8 mg kg^{-1} ; respectively), which did not differ from each other. Similar to WS K and Ca, WS Mg soil concentrations increased the most from the initial value in DAP (6.4 mg kg^{-1}), which did not differ from the CPST and ECST (5.4 and 5.4 mg kg^{-1} , respectively) treatments, and did not differ from the initial value in the RP treatment (-2.9 mg kg^{-1}). The greater (i.e., more positive) WS K, Ca, and Mg concentrations in DAP were probably related to greater solubility and greater concentrations of NH_4^+ and K^+ ions in the initial DAP fertilizer, which could have influenced cation displacement from the exchange sites, specifically K, Ca, and Mg. Furthermore, if we consider Mg in particular, for which CPST and ECST had greater WS, M3, and TR Mg concentrations than DAP (Table 1), the smaller amount of Mg contained in the DAP material may have been released more quickly than the larger amount of Mg in the CPST and ECST materials, producing a numerically greater overall increase in WS Mg.

One limitation of this study was that, upon destructive sampling of incubation cups over time, the ponded water was discarded and not chemically analyzed, which probably removed some amount of P, Ca, K, Mg, and Fe that was not captured in the WS soil extracts and potentially confounded a clear interpretation of results. However, the volume of ponded water was relatively small and was uniform across all treatments. Thus, there was probably little to no differential effect of fertilizer-P source on floodwater nutrient concentrations had they been quantified, rendering the relative differences among fertilizer-P sources still valid. Furthermore, all fertilizer material was mixed into the soil and not left surface-applied, which would have had a potentially more substantial effect on floodwater nutrient concentrations.

3.3 | Change in M3-extractable soil nutrient concentrations

The M3 elemental concentrations were generally numerically greater than their respective WS concentrations because of the greater extractability of the acidic M3 extraction solution. However, M3 soil concentrations typically followed similar trends to their respective WS concentrations.

The change in M3-P concentrations, averaged across soils, differed among fertilizer treatments over time ($P < .05$; Table 3). After 0.5 mo of incubation, M3-P concentrations increased from the initial value in all fertilized treatments but increased the most from the initial value in the ECST treatment (82.0 mg kg^{-1}), which did not differ from DAP (66.2 mg kg^{-1} ; Figure 4). After 1 mo of incubation, M3-P

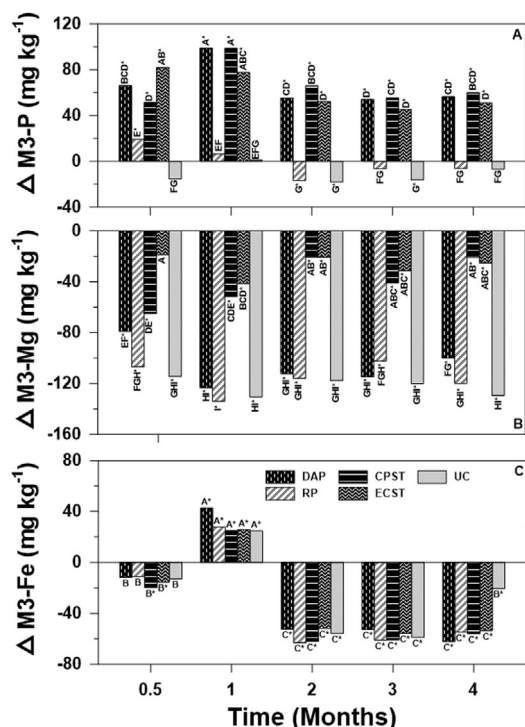


FIGURE 4 Effects of fertilizer amendment effects (DAP, diammonium phosphate; RP, rock phosphate; CPST, chemically precipitated struvite; ECST, electrochemically precipitated struvite; UC, and unamended control), averaged over soils, on the change in Mehlich-3 (M3)-extractable soil (a) P, (b) Mg, and (c) Fe concentrations from the initial values (Table 1) over time. Means within a panel with different letters are different ($P < .05$). An asterisk (*) indicates that the mean value is different from zero ($P < .05$).

concentrations increased the most from the initial value in the DAP, CPST, and ECST treatments, which did not differ from each other (Figure 4). After 2 mo of incubation, M3-P concentrations decreased slightly in all fertilizers compared with after 1 mo of incubation but were still greater than the initial value and were similar among the DAP, CPST, and ECST treatments (Figure 4). The slight decrease in M3-P concentrations at the 2-mo sampling point was probably caused by the dilution effect when additional water was added to flood the soil cups. After 3 mo of incubation, M3-P concentrations decreased numerically in all fertilizer treatments, yet were greater than from the initial value and were similar among the DAP, CPST, and ECST treatments. After 4 mo of incubation, M3-P concentrations increased numerically in all fertilizer treatments and increased the most in CPST (59.9 mg kg⁻¹), which did not differ from the DAP (56.3 mg kg⁻¹) and ECST (51.0 mg kg⁻¹) treatments (Figure 4). The slight numerical increase in M3-P concentrations in all treatments at the 4-mo sampling point was somewhat unusual, as the M3-P concentrations typically declined over time because of the decreased solubility of all fertilizer treatments later in the experiment, which was similar to the observation made by Anderson et al.

(2020b) in a nonflooded soil incubation experiment. However, because of the partly reduced conditions of the flooded soil environment in the soil incubation, it is possible that some concentration of Fe³⁺ was reduced to soluble Fe²⁺, resulting in more previously bound phosphate transferring to the soil solution (Banach et al., 2009). Figure 4 shows that, with the exception of after 1 mo of incubation, M3-Fe concentrations decreased relative to the initial value, whereas M3-P concentrations increased relative to the initial value, supporting the contention that the saturated, partly reduced soil conditions affected both the M3-P and -Fe concentrations over the duration of the incubation.

Averaged across fertilizers and time, M3-P concentrations also differed among soils ($P < .05$; Table 3). The M3-P concentrations increased the most from the initial value in the SiL 1 (50.4 mg kg⁻¹) and SiL 2 (45.3 mg kg⁻¹), which did not differ from each other. Additionally, M3-P soil concentrations increased from the initial value but less so in the SiCL soil (13.5 mg kg⁻¹) than in either SiL soil. The substantially lower change in M3-P concentration in the SiCL soil was probably related to an approximately fourfold greater initial M3-P concentration and a greater SOM concentration in the SiCL soil, which was likely to have buffered a change by the dissolving fertilizer-P sources.

The change in M3-K, -Ca, -Mg, and -Fe concentrations, averaged across fertilizer amendments, differed among soils over time ($P < .05$; Table 3). After 0.5 mo of incubation, M3-K concentrations decreased from the initial value in all soils, but decreased by approximately threefold more in the SiCL soil (−159 mg kg⁻¹) than in either the SiL 1 (−47.1 mg kg⁻¹) or SiL 2 (−53.4 mg kg⁻¹) soils, which did not differ from each other (Figure 5). Although M3-K concentrations decreased considerably more in the SiCL soil, the immediate cause was unknown. After 1 mo of incubation, M3-K concentrations increased slightly but remained less than the initial value in all soils as a result of the dissolving fertilizers (Figure 5). After the flood was imposed, M3-K concentrations were again lower than the initial value in all soils after 2 mo of incubation because of the dilution effect of the added flood water. Although a numerical increase was observed, M3-K concentrations did not change significantly in either the SiL 1 or SiL 2 soil between 2 and 4 mo of incubation (Figure 5). However, a similar trend was not observed in the SiCL soil. After 3 mo of incubation, M3-K concentrations continued to decrease considerably in the SiCL soil. In contrast, after 4 mo of incubation, an increase in M3-K concentrations was observed in the SiCL soil, although M3-K concentrations remained substantially less than in both the SiL 1 and SiL 2 soils (−47.2 and −53.4 mg kg⁻¹, respectively) and less than the initial value (−130 mg kg⁻¹; Figure 5). The M3-K concentrations increased at least numerically in all soils at the 4-mo sampling point, probably because of the continued dissolution of fertilizers under flooded soil conditions and the influx of car-

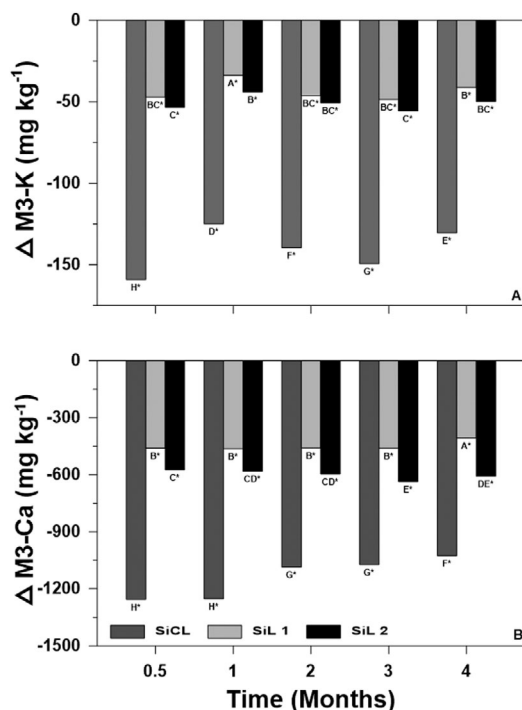


FIGURE 5 Effects of soil (SiCL, silty clay loam; SiL, silt loam) effects, averaged over fertilizer amendments, on the change in Mehlich-3 (M3)-extractable soil (a) K and (b) Ca concentrations from the initial values (Table 1) over time. Means within a panel with different letters are different ($P < .05$). An asterisk (*) indicates that the mean value is different from zero ($P < .05$).

rier cations that affected cations on the exchange sites and the soil K concentration (Hoeft, Nafziger, Johnson, & Aldrich, 2000; Korb, Jones, & Jacobsen, 2005).

Similar to the change in M3-K soil concentrations, M3-Ca soil concentrations decreased from the initial value in all soils throughout the entire duration of the soil incubation. Although the M3-Ca had decreased from the initial value at every sampling point, the change in M3-Ca concentration varied considerably among soils over time. After 0.5 mo, M3-Ca concentrations decreased from the initial value in all soils but again decreased more in the SiCL soil ($-1,255 \text{ mg kg}^{-1}$) than in either the SiL 1 or SiL 2 soils (-460 and -574 mg kg^{-1} , respectively; Figure 5). The M3-Ca soil concentrations generally remained unchanged in each soil over the first 2 mo of the incubation, except for an increase in the SiCL soil that occurred at the 2-mo sampling point. After 3 mo of incubation, the change in M3-Ca concentrations increased numerically from earlier in the incubation in the SiCL soil, remained unchanged in the SiL 1 soil, and decreased from earlier in the incubation in the SiL 2 soil (Figure 5). The variable M3-Ca response among soils at the 3-mo sampling point was probably the result of different initial M3-Ca soil concentrations and the likely different cation exchange capacities of each soil. Between the 3- and 4-mo sampling points, M3-Ca concentra-

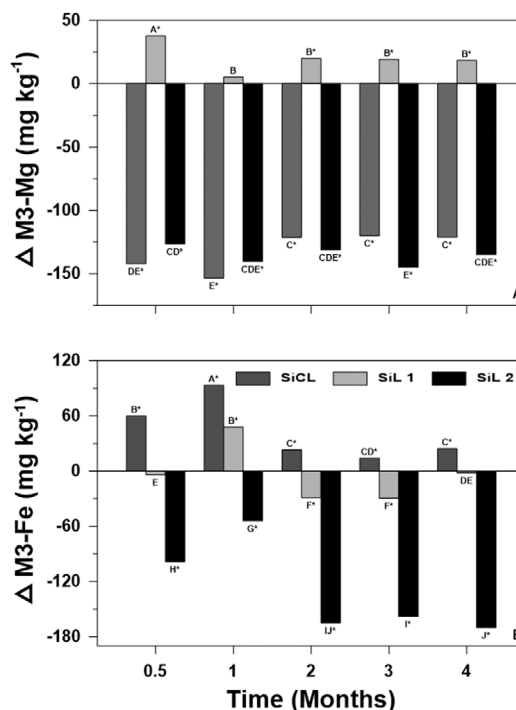


FIGURE 6 Effect of soil (SiCL, silty clay loam; SiL, silt loam) effects, averaged over fertilizer amendments, on the change in Mehlich-3 (M3)-extractable soil (a) Mg and (b) Fe concentrations from the initial values (Table 1) over time. Means within a panel with different letters are different ($P < .05$). An asterisk (*) indicates that the mean value is different from zero ($P < .05$).

tions increased slightly in all soils, except in the SiL 2 soil, which only increased numerically. In addition, after 4 mo of incubation, M3-Ca concentrations were still lower than from the initial value in all soils, but decreased the most from the initial value in the SiCL soil ($-1,026 \text{ mg kg}^{-1}$) and decreased the least in the SiL 1 soil (-407 mg kg^{-1} ; Figure 5).

The change in M3-Mg concentrations was complex and a clear trend was not observed among soils over time. After 0.5 mo of incubation, M3-Mg concentrations increased from the initial value in the SiL 1 soil (37.7 mg kg^{-1}) and decreased from the initial value in both the SiCL and SiL 2 soils (-142 and -126 mg kg^{-1} , respectively), which did not differ from each other (Figure 6). Though the initial WS, M3-, and TR Mg concentrations differed among soils (Table 1), the considerable variability in M3-Mg concentrations among soils, specifically in the SiL 1 and SiL 2 soils, was somewhat unexpected. However, considering that the SiL 1 soil had the lowest initial WS, M3-, and TR Mg concentrations among the soils (Table 1), the addition of the Mg contained in the fertilizer materials, particularly the two struvite sources, probably had a greater positive effect in SiL 1 than in the other two soils that had significantly greater initial soil Mg (Table 1). After 1 mo of incubation, M3-Mg concentrations decreased in all soils and decreased from the initial value in the SiCL and SiL

2 soils, whereas the M3-Mg concentration did not differ from the initial value in the SiL 1 soil. The decrease in M3-Mg concentrations across all soils was again somewhat unexpected because of the addition of Mg ions from all fertilizer sources, particularly the two struvite materials (i.e., ECST and CPST), and the addition of other carrier cations from the dissolving fertilizers under moist soil conditions that should have made M3-Mg more available; however, this was not observed. After 1 mo of saturated or flooded soil conditions (i.e., 2 mo of incubation), M3-Mg concentrations increased slightly in all soils, but remained less than the initial value in the SiCL and SiL 2 soils. Similar to M3-K concentrations, M3-Mg concentrations generally did not change in any soil between 2 and 4 mo of incubation. After 4 mo of incubation, M3-Mg concentrations still remained lower than the initial values in SiCL and SiL 2 soils (-121 and -126 mg kg $^{-1}$, respectively) and was greater from the initial value in the SiL 1 soil (18.4 mg kg $^{-1}$; Figure 6).

Similar to M3-Mg concentrations, the change in M3-Fe concentrations varied considerably among the different soils used in the incubation. After 0.5 mo of incubation, M3-Fe concentrations increased from the initial value in the SiCL soil (60.1 mg kg $^{-1}$), did not differ from the initial value in the SiL 1 soil (-4.06 mg kg $^{-1}$), and decreased from the initial value in the SiL 2 soil (-98.5 mg kg $^{-1}$; Figure 6). The variability in M3-Fe concentrations among soils after 0.5 mo of incubation was probably caused by the considerably different initial M3-Fe concentrations of the soils used in the incubation (Table 1). After 1 mo of incubation, M3-Fe concentrations had generally increased across all soils from earlier in the incubation, and had increased from the initial value in both the SiCL and SiL 1 soils, whereas M3-Fe concentrations remained lower than the initial value in the SiL 2 soil. After 1 mo of saturated or flooded soil conditions (i.e., 2 mo of incubation), M3-Fe concentrations had decreased by more than 60 mg kg $^{-1}$ in every soil, and only the M3-Fe concentration in the SiCL remained greater than the initial value, whereas the M3-Fe concentration in both the SiL 1 and SiL 2 soils were less than the initial value. The M3-Fe concentrations remained relatively stable (i.e., ± 10 mg kg $^{-1}$) in both the SiCL and SiL 2 soils between 2 and 4 mo of incubation. However, after 4 mo of incubation, a slight increase in M3-Fe concentration was observed in the SiL 1 soil from earlier in the incubation, but the M3-Fe concentrations did not differ from the initial value. Additionally, after 4 mo of incubation, M3-Fe concentrations had still decreased the most from the initial value in the SiL 2 soil (-170 mg kg $^{-1}$) and were still greater than the initial value in the SiCL soil (24.4 mg kg $^{-1}$; Figure 6).

The general decrease in M3-Ca, -K, -Mg, and -Fe relative to their initial soil concentrations was probably the result of a combination of two processes. First, since the floodwater was poured off and discarded prior to processing the soil for subsequent analyses at each sampling time after impos-

ing the flood, it is likely that some concentration of each of these elements was contained in the discarded floodwater that was not quantified. However, it is also likely that there were not large differences in floodwater concentrations among fertilizer amendments because all the amendments were mixed into the soil initially and not simply surface-applied. Second, though the TR concentrations were not measured after each sampling point, it is possible that some concentration of Ca, K, Mg, and Fe became more tightly bound to the soil than what the M3 extraction could recover, which would also have contributed to the general decrease in M3 concentrations over time. Consequently, if Ca, K, Mg, and Fe became more tightly bound to the soil than what the M3 extraction could recover, the ramification would be that these nutrients became non-plant-available, as the M3 extraction is meant to represent the plant-available soil fraction.

The change in M3-Mg and -Fe concentrations, averaged across soils, also differed among fertilizer treatments over time ($P < .05$; Table 3). After 0.5 mo of incubation, M3-Mg concentrations generally decreased from the initial among fertilizer amendments but decreased the most from the initial value in the UC treatment (-114 mg kg $^{-1}$), which did not differ from RP (-107 mg kg $^{-1}$; Figure 4). In addition, after 0.5 mo of incubation, M3-Mg concentrations did not differ from the initial value in the ECST treatment (-18.8 mg kg $^{-1}$; Figure 4). After 1 mo of incubation, M3-Mg concentrations increased slightly in CPST from the 0.5-mo sampling point and decreased from earlier in the incubation in the ECST, RP, and DAP treatments (Figure 4). After 2 mo of incubation, the M3-Mg concentration increased in both the CPST and ECST treatments, but remained less than the initial value and generally did not change significantly in all other treatments. After 2, 3, and 4 mo of incubation, M3-Mg concentrations remained relatively stable, with only minor differences among fertilizer treatments. After 4 mo of incubation, M3-Mg concentrations decreased the most from the initial value in the UC treatment (-129 mg kg $^{-1}$), which did not differ from RP (-120 mg kg $^{-1}$), and decreased the least from the initial in the CPST treatment (-20.9 mg kg $^{-1}$), which did not differ from ECST (-25.4 mg kg $^{-1}$; Figure 4). The generally less negative M3-Mg concentrations in both struvite sources throughout the soil incubation period was expected, given the substantially greater initial M3-Mg concentrations of the struvite materials (Table 2).

Unlike the change in M3-Mg concentrations, the change in M3-Fe concentrations among fertilizer treatments was greatly affected by the imposition of saturated or flooded soil conditions. After 0.5 mo of incubation, M3-Fe concentrations did not differ among fertilizer treatments, but decreased from the initial value in the CPST (-19.9 mg kg $^{-1}$) and ECST (-15.5 mg kg $^{-1}$) treatments and did not differ from the initial value in all other treatments (Figure 4). The CPST and ECST materials had lower M3-Fe concentrations than DAP

and RP (Table 2); thus, the contribution of Fe from CPST and ECST was lower than that from DAP and RP, allowing early M3-Fe concentrations to change less than with CPST and ECST. After 1 mo of incubation, M3-Fe concentrations increased in all treatments and were similar among all fertilizer treatments (Figure 4), which may have been caused by the conversion of WS Fe into less available M3-Fe as secondary phosphates precipitated out of the soil solution (Nascimento et al., 2018; Smeck, 1985; Tiessen, Stewart, & Cole, 1984). After 1 mo of flooded conditions (i.e., 2 mo of incubation), M3-Fe concentrations decreased in all treatments because of the dilution effect of saturated or flooded soil conditions. They were lower than the initial value and were similar among all fertilizer treatments (Figure 4). Throughout the entire duration of the flooded conditions (i.e., 2, 3, and 4 mo of incubation), M3-Fe concentrations did not change among fertilizers. They remained less than the initial value and similar among all fertilizer treatments, despite differences in the initial M3-Fe concentrations among the four fertilizer materials (Table 2).

3.4 | Implications

Because of the relatively limited supply of RP (Liu et al., 2012), long-term use of conventional RP-derived fertilizer-P sources presents a potential food security risk. Thus, important research into applications of recovered fertilizer-P sources remains an ongoing pursuit in sustainable development. Struvite recovery is one attractive option for a sustainable source of P because of the ability to recover both P and N from liquid and solid wastes (Schoumans et al., 2015). Additionally, struvite recovery has been shown to be applicable to a number of different waste sources from industrial and agricultural sectors (Diwani et al., 2007; Jaffer, Clark, Pearce, & Parsons, 2002; Massey et al., 2007; Münch & Barr, 2001; Rahman et al., 2014). Intentional recovery of struvite from WWTPs has the potential to reduce the volume of sewage sludge produced by reducing nutrients from secondary waste streams and, subsequently, to reduce the operational costs associated with the disposal of sewage sludge (Doyle & Parsons, 2002; Woods et al., 1999). Struvite, as a fertilizer-P source, has been shown to have slow-release characteristics (Massey, Davis, Ippolito, & Sheffield, 2009; Tallboys et al., 2016), which could potentially benefit rice production systems, since excess P in released floodwater can cause eutrophication in local surface waters (Carpenter & Bennett, 2011; Tian, Zhang, Zhao, Zhang, & Huang, 2017). Struvite is also a potential source of Mg, which could be used to help alleviate soil Mg deficiencies in certain circumstances. However, there is also the potential for the large Mg load provided with struvite applications to disrupt the Ca/Mg ratio in a soil and thus could negatively affect plant responses, which would have to be monitored through soil testing.

Although plants were not grown in this study, there would probably be no adverse effects on soil quality or crop production through the application of ECST or CPST with the soil textures and the total P rate used in this study, unless soil pH or WS and M3 nutrient concentrations changed substantially beyond the optimal limits. However, explicitly in this study, M3-Ca in all soils and M3-Mg concentrations in the SiL 2 and SiCL soils decreased by more than 25% from the initial concentrations, which could lead to a reduction in plant productivity if not corrected.

4 | CONCLUSIONS

The results from this study partly supported the hypothesis that WS and M3-P concentrations were similar among ECST, CPST, and DAP, which were greater than those from RP. The hypothesis was only partly supported because, generally, WS P concentrations were at least numerically greater in the CPST, ECST, and DAP treatments than under the RP treatment, although, at the 3- and 4-mo sampling points, WS P concentrations were similar in CPST, ECST, and RP. The results supported the hypothesis of a similar M3-P concentration among CPST, ECST, and DAP treatments. Even under the complex and often dynamic conditions of flooded soils, WS and M3-P concentrations were similar among ECST, CPST and other commercial P fertilizers. The comparable WS- and M3-P concentrations over the course of the incubation in the two struvite materials and other commercial P fertilizers under flooded conditions further support evidence from previous studies that have shown struvite to be a viable alternative to traditional RP-derived P fertilizers in several soil environments.

The results from the soil incubation also partly supported the hypothesis that WS and M3-Mg concentrations would be greatest in the two struvite sources. The hypothesis was only partly supported because, averaged over time and soils, WS Mg concentrations increased the most from the initial value in the DAP treatment, which did not differ from CPST and ECST. However, the results supported the hypothesis of greater M3-Mg concentrations in the two struvite sources (i.e., CPST and ECST) than in DAP or RP.

Overall, despite several procedural limitations, the results from this study provide important information on struvite-soil interactions in agronomic soils, specifically in a flooded soil environment. Additionally, the results from this study document the effects that P fertilizers can have on select soil chemical properties in a flooded soil environment. A generally similar response was observed in WS and M3-P concentrations among struvite and traditional fertilizer-P sources, despite differences in the physical and chemical composition of the different P fertilizers used in the study. The similar fertilizer response between fertilizer-P sources further

supports the potential utilization of wastewater-recovered struvite in future agronomic applications. Although potential rhizosphere interactions were not addressed in this study because of the absence of plants, the results provided a baseline of soil P behavior over time from struvite as an alternative fertilizer-P source that may potentially be applicable to a wide variety of upland and lowland crops. Additional research on struvite's effectiveness as a fertilizer-P source, particularly in a flooded soil environment, will need to include plants to ascertain struvite's solubility in the presence of organic acids over time in the rhizosphere, which, in turn, could affect plant nutrient uptake.

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AUTHOR CONTRIBUTIONS

Ryder Anderson: Data curation, Formal analysis, Methodology, Writing-original draft. Kristofor R. Brye: Conceptualization, Funding acquisition, Methodology, Project administration, Supervision, Writing-review & editing. Laszlo Kekedy-Nagy: Methodology. Lauren Greenlee: Funding acquisition, Writing-review & editing. Edward Gbur: Formal analysis, Writing-review & editing. Trent L. Roberts: Formal analysis, Writing-review & editing.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interest.

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