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ORIGINAL RESEARCH ARTICLE

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Electrochemically precipitated struvite effects on extractable nutrients compared with other fertilizer-phosphorus sources

Ryder Anderson¹ | Kristofor Brye¹ | Laszlo Kekedy-Nagy² | Lauren Greenlee² | Edward Gbur²

Trenton Roberts^{1,3}

¹ Dep. of Crop, Soil, and Environmental Sciences, Univ. of Arkansas, Fayetteville, AR 72701, USA

² Dep. of Chemical Engineering, Univ. of Arkansas, Fayetteville, AR 72701, USA

³ Agricultural Statistics Laboratory, Univ. of Arkansas, Fayetteville, AR 72701, USA

Correspondence

Kristofor R. Brye, Dep. of Crop, Soil, and Environmental Sciences, Univ. of Arkansas, Fayetteville, AR 72701, USA. Email: kbrye@uark.edu

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Abstract

Recovery of struvite, or magnesium ammonium phosphate (MgNH₄PO₄ \cdot 6H₂O), from wastewater streams may provide an alternative to traditional P fertilizers. Little research has assessed the behavior of struvite relative to other commercially available, fertilizer-P sources in historically row-cropped soils in the United States. The objective of this study was to evaluate total extractable P and other nutrients from electrochemically (ECST) and chemically precipitated struvite (CPST) compared with triple superphosphate (TSP), diammonium phosphate (DAP), monoammonium phosphate (MAP), and rock phosphate (RP) in a moist-soil incubation without plants using varying soil textures (loam, silt loam, and silty clay loam). A uniform application rate of 24.5 kg total P ha⁻¹ was used for each fertilizer-P source. Soil sampling occurred six times over a 9-mo period (0.5, 1, 2, 4, 6, and 9 mo) to examine the change in soil pH and water-soluble (WS) and Mehlich-3-extractable nutrient concentrations (P, Ca, Mg, and Fe) from their initial levels over time. After 0.5 mo, WS-P concentrations increased the most in the ECST treatment (41.6 mg kg⁻¹), which did not differ from that of DAP. Throughout the remaining 8.5 mo of incubation, WS-P concentrations generally decreased in most treatments but were still greater than the initial level by 9 mo and were often similar among ECST, CPST, MAP, DAP, and TSP treatments. Comparable WS-P concentrations among ECST and MAP, DAP, and TSP under soil conditions near field capacity (~ 0.2 g s^{-1}) support struvite's potential as a sustainable fertilizer-P source, thus warranting further investigation of the plant response to struvite use as an alternative fertilizer-P source.

Abbreviations: CEC, cation exchange capacity; CPST, chemically precipitated struvite; DAP, diammonium phosphate; ECST, electrochemically precipitated struvite; ICAPS, inductively coupled, argon plasma spectrometry; M3, Mehlich-3; MAP, monoammonium phosphate; RP, rock phosphate; SiCL, Dardanelle silty clay loam; SiL 1, Calloway silt loam; SiL 2, Henry silt loam; SOM, soil organic matter; TR, total recoverable; TSP, triple superphosphate; UC, unamended control; WS, water-soluble; WWTP, wastewater treatment plant

INTRODUCTION 1

Phosphorus is a fundamental element that is essential to all forms of life. After N, P is the most common limiting nutrient in agricultural production. Phosphorus plays an essential role in the formation of nucleic acids, phosphoproteins, phosphate-containing energy compounds, and phospholipids

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(Shen et al., 2011; Smeck, 1985). However, only a small proportion of P is available to plants due to relatively low solubility and strong P-fixation capacity in many soils; thus, P fertilizers are often required to maintain optimal crop yields (Holford, 1997; Smil, 2000).

Food production has drastically increased over the recent century due to the technological advances of the Green Revolution, and the demand for P fertilizers has consequently increased (Cordell et al., 2011). Most commercially available, inorganic, fertilizer P sources are derived from rock phosphate (RP), which is limited in supply and is expected to be diminished in as little as 100–250 yr (Liu et al., 2012). Consequently, future food security depends on a sustainable source of fertilizer P to continue to feed a growing global population (Cordell et al., 2009).

Along with food and fiber production, clean water is an essential resource for irrigation, drinking water, and supporting many biologically diverse aquatic species. Although P is essential for plant growth, in aquatic systems, excess P can be problematic and lead to water quality degradation. Excess nutrient enrichment can lead to eutrophication, which can be detrimental to the balance of plants, fish, and other aquatic species (Syers et al., 2008). Eutrophication remains a widespread water quality issue throughout much of the United States and elsewhere and is largely caused by human development and manipulations of the landscape. In areas of impaired water, agriculture is the primary source of nutrients in lakes and rivers in the United States (Daniel et al., 1998; Elser & Bennett, 2011).

In addition to water quality, the current terrestrial P cycle is ineffective, where the majority of P in the food production system is lost to soil and waste flows (Suh & Yee, 2011). Furthermore, nearly 98% of P in the human diet ends up in sewage to be treated in wastewater treatment plants (WWTPs; Øgaard & Brod, 2016; Smil, 2000). Wastewater treatments plants are required by law in the United States to remove substantial amounts of P, N, and organic matter from wastewater effluent streams (De-Bashan & Bashan, 2004). However, considerable amounts of P and N removed from effluent streams in WWTPs are retained in sewage sludge (Plaza et al., 2007), which often ends up in landfills.

Recovering P from waste sources has been an area of active research over the last few decades to potentially provide a sustainable alternative to commercial P fertilizers derived from the finite supply of RP (Ahmed et al., 2018; Hertzberger et al., 2020; Huygens & Saveyn, 2018). One such potential alternative source is magnesium ammonium phosphate hexahydrate (MgNH₄PO₄ · 6H₂O), which is the mineral struvite. Struvite is a white crystalline material with approximately equal molar concentrations of Mg, NH₄, and PO₄ (Schoumans et al., 2015). Struvite has been shown to be a slow-release fertilizer P source (Tallboys et al., 2016) that can be recovered from both solid and liquid wastes. Numerous greenhouse potted-

Core Ideas

- Nutrient recovery from wastewater could provide a source of P in agricultural soils.
- Electrochemically precipitated struvite acts similar to other fertilizer-P sources.
- Electrochemically precipitated struvite can be an alternative fertilizer-P source.

plant studies have demonstrated plant response to be similar between struvite and other commercially available fertilizer P sources (Ahmed et al., 2018; González-Ponce et al., 2009; Hilt et al., 2016; Horta, 2017; Pérez et al., 2009; Plaza et al., 2007).

Although the agronomic effectiveness of struvite has been examined in several plant studies, soil-struvite interactions across soil textures have not been well studied, particularly in soils with a row-cropping history. In addition to P and N, struvite also contains Mg, which can affect plant growth. Furthermore, soil P behavior can be affected by interactions with Ca and Fe, depending on soil pH. The objective of this study was to evaluate temporal changes in total extractable P and other nutrients (i.e., inorganic N, Ca, Mg, and Fe) from two wastewater-derived struvite materials, electrochemically (ECST) and chemically precipitated struvite (CPST), compared with triple superphosphate (TSP), diammonium phosphate (DAP), monoammonium phosphate (MAP), and RP in a 9-mo, plantless incubation using moist soil of varying texture (loam, silt loam, and silty clay loam). Electrochemically precipitated struvite represents a new material that has not been studied in a soil environment previously, nor has ECST been compared with other fertilizer-P sources. It was hypothesized that both struvite sources will have similar water-soluble (WS) and Mehlich-3 (M3)-extractable P concentrations as MAP, DAP, and TSP after 2-4 mo, the approximate length of the growing season for a summer row crop. It was also hypothesized that the finer-textured soils will have smaller pH and nutrient concentration changes due to greater buffering capacity and soil retention, as initial chemical properties may differ between similar-textured soils with differing histories of exposure to oxidizing or reducing conditions to affect soil Fe concentrations that may affect P binding.

2 | MATERIALS AND METHODS

2.1 | Fertilizer-P sources and analyses

Two sources of struvite were used in this study: (a) a commercially available, CPST source (trade name Crystal Green,

TABLE 1 Summary of initial chemical properties among fertilizer-P sources used in the soil incubation

	Fertilizer-P source ^a						
Fertilizer property	MAP	DAP	TSP	CPST	ECST	RP	
pH	4.37	7.32	2.42	8.78	_b	6.67	
Electrical conductivity, dS m ⁻¹	84.6	105	32.8	226	-	514	
Water-soluble, mg kg ⁻¹							
Р	196,000	163,300	178,840	216	124,050	70.6	
Ca	2,252	153	121,296	11.6	12.7	148	
Mg	7,784	79.9	5,791	157	24,144	25.5	
Fe	68.8	63.6	473	1.22	5.80	4.20	
Mehlich-3-extractable, mg kg ⁻¹							
Р	181,919	164,349	171,493	24,479	158,798	638	
Ca	1,931	228	105,735	83	2.20	3,602	
Mg	6,767	507	4,715	21,444	27,197	338	
Fe	254	146	362	127	19.9	226	
Total recoverable , mg $\rm kg^{-1}$							
Р	209,215	183,365	182,187	116,556	227,658	75,956	
Ca	4,309	4,653	140,177	312	0.150	163,495	
Mg	14,535	6,734	6,205	83,234	57,023	3,219	
Fe	3,737	5,785	1,527	4,505	0.100	10,592	
Total N, g g^{-1}	0.107	0.181	0.0002	0.057	0.093	0.0004	

^aMonoammonium phosphate (MAP), diammonium phosphate (DAP), triple superphosphate (TSP), chemically precipitated struvite (CPST), electrochemically precipitated struvite (ECST), and rock phosphate (RP).

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

Ostara Nutrient Recovery Technologies) produced from raw, municipal wastewater, and (b) an ECST material produced from synthetic wastewater by researchers in the Department of Chemical Engineering at the University of Arkansas (Kékedy-Nagy et al., 2020). The ECST material was electrochemically precipitated from a prepared solution of known P and N concentrations with Mg being supplied by a Mg anode as an electrical current was imposed on the solution (Kékedy-Nagy et al., 2020). In addition to the two struvite sources, four additional commercially available fertilizer-P sources were used in this study: MAP, DAP, TSP, and RP.

Fertilizer particle sizes differed among fertilizer-P sources and varied from small pellets (i.e., prills) to crystals to powder forms. Monoammonium phosphate, DAP, TSP, and CPST were in pelletized form, whereas RP was in powder form, and ECST was in crystalline form. As reported in Anderson (2020), the average prill diameters were 2.9 mm for both DAP and CPST. Pelletized fertilizers were mechanically crushed and chemically characterized in powder form to facilitate comparisons of chemical compositions among fertilizer-P sources, but the raw forms of each fertilizer material, either pellet, powder, or crystalline, were applied to the soil for the incubation experiment described below.

Similar to Anderson et al. (2020), Anderson, Brye, Greenlee, et al. (2021), and Anderson, Brye, Kekedy-Nagy, et al.

(2021), for each fertilizer material, five subsamples were used for chemical analyses. Soil pH was measured potentiometrically in a 1:2 fertilizer mass/water volume ratio (Sikora & Kissel, 2014). Total N concentration was determined by high-temperature combustion using an Elementar VarioMax CN analyzer (Provin, 2014). Elemental concentrations of P, Ca, Mg, and Fe were quantified from water and M3 extractions and strong-acid digestions. Water-soluble concentrations were determined after extraction using a 1:10 fertilizer mass/water volume ratio, where the mixture was stirred for 1 h, filtered through a 0.45-µm screen, and analyzed by inductively coupled, Ar plasma spectrometry (ICAPS; Spectro Arcos ICP, Spectro Analytical Instruments; Zhang et al., 2014) to represent environmentally relevant concentrations after interaction with rainwater. Mehlich-3-extractable concentrations were determined after extraction with M3 extraction solution using a 1:10 fertilizer mass/extractant volume ratio (Tucker, 1992) and analyzed by ICAPS (Zhang et al., 2014) to represent plant-available nutrient concentrations. Total-recoverable (TR) concentrations were determined by ICAPS after strong-acid digestion (USEPA, 1996) to represent TR nutrient concentrations that could become environmentally available. Chemical compositions of the various fertilizer materials used in this study are summarized in Table 1.

2.2 | Soil collection and analyses

Four soils were collected from the top 10–15 cm in December 2017 from various agricultural settings throughout Arkansas. At the University of Arkansas Division of Agriculture's Vegetable Research Station in Kibler, AR, a Dardanelle silty clay loam (SiCL; fine-silty, mixed, superactive, thermic Typic Argiudolls; USDA, 2015) and a Roxana loam (loam; coarsesilty, mixed, superactive, nonacid, thermic Typic Udifluvent; USDA, 2015) were collected. The Dardanelle soil had been recently cropped to soybean [Glycine max (L.) Merr.], whereas the Roxana soil had been under recent vegetable production. From the Cotton Branch Experiment Station near Marianna, AR, a Calloway silt loam (SiL 1; fine-silty, mixed, active, thermic Aquic Fraglossudalfs; USDA, 2015), cropped to a wheat (Triticum aestivum L.)-soybean rotation for the previous 15 yr, was collected. From the Pine Tree Branch Experiment Station near Colt, AR, a Henry silt loam (SiL 2; coarse-silty, mixed, active, thermic Typic Fragiagualfs; USDA, 2015), cropped to a rice (Oryza sativa L.)-soybean rotation for at least the previous 5 yr, was collected. The SiL 1 soil was from a udic soil moisture regime, thus mostly experienced a history of aerobic, oxidizing soil conditions, whereas the SiL 2 soil experienced extensive anaerobic, reducing soil conditions due to the aquic soil moisture regime and history of rice production. Soils were sieved field-moist through a 7mm mesh screen and air dried for ~ 2 wk. The 7-mm mesh size was used to break up large clods from field collection, while retaining some degree of aggregation for subsequent use.

After procedures and the same soils used in Anderson et al. (2020), Anderson, Brye, Greenlee, et al. (2021), and Anderson, Brye, Kekedy-Nagy, et al. (2021) and procedures used for fertilizer material analyses, triplicate soil subsamples were characterized for physical (i.e., particle-size distribution) and chemical analyses (i.e., pH, EC, soil organic matter [SOM], total C, total N, and WS and M3 elemental concentrations [P, Ca, Mg, and Fe]). Soil subsamples were also extracted with 2 M KCl in a 1:5 soil mass/extractant volume ratio and analyzed spectrophotometrically to determine initial NO₃–N and NH₄–N concentrations by the cadmium-reduction and salicy-late methods, respectively (Mulvaney, 1996). Table 2 summarizes initial properties of the four soils used in this study.

2.3 | Soil incubation experiment

A moist-soil incubation was conducted over a 9-mo period from 6 Dec. 2018 to 15 Aug. 2019. Cylindrical, plastic soil cups, 4.5 cm tall and 10.5 cm in diameter at the top, were used. Two soil-cup replicates were prepared for each soil–fertilizer treatment combination for each of six planned sampling intervals over the 9-mo incubation period. Approximately 150 g of air-dried soil were added to each soil cup. One of the six fer**TABLE 2** Summary of initial soil properties among soils used in the soil incubation (adapted from Anderson et al., 2020)

	Soil ^a					
Soil property	SiL 1	SiL 2	Loam	SiCL		
рН	6.53b ^b	6.70a	6.17c	6.50b		
Electrical conductivity, $dS m^{-1}$	0.169b	0.164b	0.107c	0.273a		
Water-soluble, mg kg ⁻¹						
Р	5.47c	3.70d	11.9a	9.60b		
Ca	62.7b	62.0b	34.0c	74.3a		
Mg	23.3b	17.7c	21.7b	28.0a		
Fe	47.9a	47.9a	47.9a	47.9a		
Mehlich-3-extractable, mg kg ⁻¹						
Р	33.7c	19.7d	93.3b	143a		
Ca	1,842b	2,156b	933c	4,328a		
Mg	444b	365b	194c	774a		
Fe	186c	459a	201b	175d		
Sand, g g^{-1}	0.12b	0.10c	0.44a	0.07d		
Clay, g g^{-1}	0.14b	0.11c	0.09d	0.37a		
Silt, g g ⁻¹	0.75b	0.79a	0.46d	0.56c		
Soil organic matter, g g ⁻¹	0.024b	0.019c	0.007d	0.025a		
Total C, g g^{-1}	0.011b	0.009c	0.003d	0.012a		
Total N, g g^{-1}	0.0011a	0.0008b	0.0003c	0.0011a		
C/N ratio	9.68c	11.0ab	10.5b	11.4a		
NO_3 –N, mg kg ⁻¹	15.8a	12.2b	9.50c	6.30d ^a		
NH_4 –N, mg kg ⁻¹	8.20a	6.40b	3.90c	6.30b		

^aSilt loam (SiL) and silty clay loam (SiCL).

^bMeans in a row with different letters are different at p < .05.

tilizer treatments (i.e., pelletized MAP, pelletized DAP, pelletized TSP, powderized RP, pelletized CPST, and crystallized ECST) was applied to each soil cup replicate in each treatment combination. An unamended control (UC) treatment was also included. Fertilizers were applied at an agronomically relevant rate of 56 kg P_2O_5 ha⁻¹ (24.5 kg of P ha⁻¹), which was derived as an average from the recommended fertilizer-P rate for the dominant row crops produced in Arkansas, and based on the TR-P concentration of each fertilizer material. Despite varying in concentration among fertilizer-P sources, Ca, N, and Mg were not controlled for in this experiment due to the absence of plants and any potential rhizosphere effect. After the fertilizer was added, each soil cup was individually shaken for ~10 s in an up-down and circular motion to incorporate the fertilizers in the soil. In total, 336 soil cups were prepared for incubation. After mixing, soil cups were then randomly distributed among two three-level wooden shelf structures that were placed side by side in the laboratory. Soil cups were rotated among shelves every 2 wk over the course of the incubation.

Soil cups were periodically watered using identical procedures as described in Anderson et al. (2020), in which the target mass was determined for each soil based on a set target gravimetric water content (i.e., ~ 0.20 g g⁻¹). Soil cups were initially watered on the same day as the application of the fertilizers, in which soil cups were wetted to a specific target mass using tap water. The target watering mass was derived from estimating the field moisture capacity and bulk density for each soil based on measured sand, clay, and SOM using the Soil Water Characteristics subroutine of the Soil, Plant, Atmosphere, Water model (Saxton et al., 1986; USDA, 2017). Every 2 wk thereafter, soil cups were gravimetrically rewatered to the specific target mass using tap water.

Soil cups were prepared with a uniform mass of air-dried soil but achieved varying initial soil bulk densities due to the differences in soil particle-size distribution. Initial mean bulk densities ranged from 1.28 g cm⁻³ for the SiCL to 1.59 g cm⁻³ for the loam soil. Accounting for differences in soil particle-size distributions, estimated bulk densities, and measured SOM concentrations, target soil water contents for periodic rewetting the incubating soil varied only slightly among soils, ranging from 0.201 g g⁻¹ for the loam to 0.204 g g⁻¹ for the SiL 2 soil.

Over the 9-mo period, soil cups were destructively sampled after six incubation periods: 0.5, 1, 2, 4, 6, and 9 mo. At each sampling, soil was removed from the plastic cups, oven dried for 48 h at 70 °C, mechanically crushed, and sieved through a 2-mm mesh sieve. Any residual fertilizer material that was visible was not removed. Soil subsamples were analyzed for pH and WS- and M3-extractable P, Ca, Mg, and Fe, and inorganic NO₃- and NH₄-N concentrations following procedures previously described for fertilizer and soil analyses. Although no plants were used in this incubation study, the M3 was used along with the WS extraction of soils to relate to a situation when plants are grown and amended with the various fertilizer-P sources. Calcium was measured on account of Ca being a common carrier cation with inorganic P fertilizers and Ca's potential reactivity with P under alkaline soil conditions. Magnesium was measured on account of the additional Mg contained in the struvite materials. Iron was measured on account of Fe's potential reactivity with P under acidic soil conditions. Inorganic N forms were measured on account of the N concentration in several of the blended fertilizer materials (i.e., MAP, DAP, CPST, and ECST) used in the incubation.

The soil incubation was conducted in a climate-controlled laboratory location. Humidity and air temperature fluctuations were measured throughout the soil incubation using an Acurite thermometer model 0055 and 4SBDI (Chaney Instrument Company). Over the course of the 9-mo incubation period, the ambient air temperature ranged from 21.1 to 22.2 °C and averaged 21.6 °C. The ambient relative humidity ranged from 54 to 58% and averaged 56.5%.

2.4 | Statistical analyses

A one-factor ANOVA was conducted using PROC GLIM-MIX in SAS (version 9.4, SAS institute) to evaluate differences in initial soil properties among soils. Based on a splitsplit-plot, complete factorial, completely randomized design, a three-factor ANOVA was conducted using PROC GLIM-MIX in SAS to evaluate the effects of soil (loam, SiCL, SiL 1, and SiL 2), fertilizer treatment (MAP, DAP, TSP, RP, CPST, ECST, and UC), sampling time (0.5, 1, 2, 4, 6, and 9 mo), and their interactions on the change in soil pH and WS- and M3-extractable P, Ca, Mg, and Fe, and NO₃- and NH₄-N concentrations from their initial soil levels. The whole-plot factor was soil, the split-plot factor was fertilizer treatment, and the split-split-plot factor was sampling time. Soil, fertilizer treatment, and sampling time were fixed effects, whereas replication was treated as a random effect. Since a different set of replicate treatment combinations were sampled at each time point, all samples were assumed independent. To achieve the change in soil properties from their initial levels, which were the data that were statistically analyzed, the initial mean level of each soil property was subtracted from that measured at each sampling interval on a cup-by-cup basis. Since soilproperty-change data could be positive or negative, a normal data distribution was used for data analyses after visually assessing studentized residuals. When appropriate, means were separated by LSD at the .05 level.

3 | **RESULTS AN DISCUSSION**

3.1 | Electrochemically precipitated struvite properties

Several factors affect the formation of struvite that differ among waste products, wastewaters, and recovery techniques. The ECST material used in this experiment was derived from synthetic wastewater that contained only PO_4^{3-} and NH_4^{+} ions, in addition to the Mg anode through which an electrical current was applied to the solution (Kékedy-Nagy et al., 2020); thus, excluded impurities otherwise present in many other recovered-struvite sources. The result of the greater purity of the ECST material was numerically greater concentrations of total P (22.8%) and total N (9.3%) than for CPST (11.3 and 5.5%, respectively; Table 1). Although several studies have reported struvite containing in the range of 12-14% total P (Johnston & Richards, 2003; Kataki et al., 2016; Liu et al., 2012; Nongqwenga et al., 2017; Rahman et al., 2014), the greater total P concentration in the ECST material used in this study is still within the 11-26% total P range reported for struvite (Johnston & Richards, 2003; Kataki et al., 2016). In addition, the ECST material used in this study also had a

TABLE 3 Analysis of variance summary of the effects of soil (S), fertilizer treatment (F), sample time (T), and their interactions on the change in pH, NO_3 and NH_4 concentrations, and water-soluble (WS) and Mehlich-3 (M3)-extractable nutrient (P, Ca, Mg, and Fe) concentrations from initial soil levels

	<i>p</i> value										
Source of											
variation	∆pH ^a	$\Delta NO_3 - N$	ΔNH_4-N	Δ WS-P	∆WS-Ca	Δ WS-Mg	∆WS-Fe	∆М3-Р	∆M3-Ca	ΔM3-Mg	∆M3-Fe
S	<.01	.10	.05	.02	<.01	<.01	<.01	.39	<.01	<.01	<.01
F	<.01	<.01	<.01	<.01	<.01	<.01	.47	<.01	<.01	<.01	<.01
Т	<.01	<.01	<.01	<.01	<.01	<.01	<.01	.25	<.01	.17	<.01
$S \times F$	<.01	<.01	.09	.21	<.01	<.01	.73	.02	.82	.17	<.01
$S \times T$	<.01	<.01	<.01	<.01	<.01	<.01	<.01	.39	<.01	.52	<.01
$F \times T$	<.01	<.01	<.01	<.01	<.01	<.01	<.01	.60	.76	.49	.30
$S \times F \times T$	<.01	.01	<.01	.14	.99	.90	<.01	.54	.99	.59	.98

a Soil-property-change data were calculated by subtracting the initial mean level of each soil property from that measured at each sampling interval on a cup-by-cup basis.

numerically lower Mg concentration (5.7%) than that of CPST (8.0%; Table 1), although this was likely related to the differential recovery processes and is common among struvite sources.

3.2 | Change in soil pH

The change in soil pH from the initial value differed among soil-fertilizer treatment combinations over time (p < .05;Table 3). The change in soil pH was complex and a clear trend was not observed across fertilizer treatments and often differed among soils (Table 4). Over the duration of the incubation, soil pH generally decreased from the initial value in all soil-fertilizer combinations. In the loam soil, by 0.5 mo of incubation, soil pH increased the most from the initial value in the ECST (0.43 pH units), which did not differ from that for CPST, RP, and UC treatments (Table 4). Soil pH decreased from the initial value in the MAP treatment (-0.12 pH units), which did not differ from that for DAP (Table 4). As the incubation progressed, a net acidification effect was observed throughout all fertilizer treatments in the loam soil, which was likely caused by the dissolution of the fertilizers and the introduction of cations, such as Ca^{2+} , Mg^{2+} , and NH_4^+ , which have a greater affinity for exchange sites than do H⁺ ions (Anderson et al., 2020; do Nascimento et al., 2018; Montalvo et al., 2014). Additionally, another major source of soil acidity was likely caused by microbial nitrification of the NH_4 -containing fertilizers, such as MAP, DAP, CPST, and ECST (Vaneeckhaute et al., 2016). By 9 mo of incubation, soil pH had decreased from the initial value in all treatments and the greatest pH decrease occurred in the DAP treatment (-0.92 pH units; Table 4).

In contrast with the pH change in the loam soil, soil pH at least numerically increased from the initial value after 0.5 mo of incubation in all fertilizer treatments in the SiCL soil. By 0.5 mo of incubation, the CPST treatment had the greatest increase in soil pH (0.40 pH units) in the SiCL soil, which did not differ from that for the DAP, TSP, RP, and UC treatments (Table 4). After 1 mo of incubation, soil pH increased from the initial value and was similar among all fertilizer treatments (Table 4). By 2 mo of incubation and thereafter, soil pH generally decreased among all fertilizer treatments in the SiCL soil. Soil pH decreased later in the incubation in the SiCL soil due to the likely greater cation exchange capacity (CEC) of the SiCL, in which the SiCL soil had greater initial SOM and clay concentrations that have an increased capacity to resist change to additional H⁺ ions (Brady & Weil, 2002; Sposito, 2008). After 9 mo of incubation, soil pH had generally decreased from the initial value in all fertilizer treatments in the SiCL soil. Similar to the loam soil, by the 9-mo sampling interval, the greatest decrease in pH in the SiCL soil occurred in the DAP treatment (-0.50 pH units), which did not differ from that for MAP and ECST (Table 4). In addition, RP, CPST, and UC were the only treatments in which the change in pH from the initial value did not differ from a change of zero by the 9-mo sampling interval (Table 4).

Though not controlled for in this experiment on account of attempting to mimic what a producer would do in the field, the varying surface areas of the various forms of the original, raw fertilizer material (i.e., prill, crystalline, and powder forms) likely caused differential solubilities and reactivities to some degree among the fertilizer-P sources. However, results of this study would have been less agronomically relevant had the form and particle size been unified across the fertilizer materials than using the original form of each fertilizer material in this incubation experiment.

Similar to the pH change in the loam soil, fertilizer treatments in the SiL 1 soil (i.e., the well-drained SiL soil) displayed a similar trend in which soil pH decreased from the initial value earlier in the incubation. By 0.5 mo of incubation, soil pH decreased from the initial value and was

		Δ Soil pH							
Soil	Treat ^a	0.5 mo	1 mo	2 mo	4 mo	6 mo	9 mo		
Loam	MAP	-0.12P-a ^b	-0.32Y-j*	-0.67m-r*	-0.22T-e*	-0.57j-o*	-0.27V-h*		
	DAP	0.13H-P	-0.37a-k*	-0.77o-s*	-0.77o-s*	-1.02stu*	-0.92rst*		
	TSP	-0.07N-Y	-0.07N-Y	-0.07N-Y	0.03J-T	-0.27V-h*	-0.22T-e*		
	RP	0.18F-N*	0.23E-L*	0.18F-N*	0.23E-L*	-0.02L-V	-0.17R-c		
	CPST	0.28D-J*	0.23E-L*	0.08I-R	-0.12P-a	-0.32Y-j*	-0.37a-k*		
	ECST	0.43B-F*	-0.27V-h*	-0.37a-k*	-0.37a-k*	-0.52h-o*	-0.57j-o*		
	UC	0.23E-L*	0.23E-L*	0.13H-P	0.18F-N*	-0.07N-Y	-0.22T-e*		
SiCL	MAP	0.10H–Q	0.20E-M*	0.05I–S	-0.10O-Z	-0.30X-i*	-0.40c-l*		
	DAP	0.30D-I*	0.30D-I*	0.15G–O	0.00K-U	-0.25U-g*	-0.50g-n*		
	TSP	0.25D-K*	0.25D-K*	0.20E-M*	0.05I–S	0.00K-U	-0.20S-e*		
	RP	0.35C-H*	0.40B-G*	0.35C-H*	0.20E-M*	0.10H–Q	-0.10O-Z		
	CPST	0.40B-G*	0.45B-E*	0.40B-G*	0.30D-I*	0.05I–S	-0.10O-Z		
	ECST	0.10H–Q	0.25D-K*	0.15G–O	0.15G–O	-0.10O-Z	-0.35Z-k*		
	UC	0.30D-I*	0.45B-E*	0.30D-I*	0.25D-K*	0.10H–Q	-0.05M-X		
Sil 1	MAP	-0.33Z-k*	-0.38b-l*	-0.631-q*	-0.43d-m*	-0.73n-r*	-1.03tu*		
	DAP	-0.28W-i*	-0.43d-m*	-0.88q-t*	-0.73n-r*	-1.03tu*	-1.23u*		
	TSP	-0.33Z-k*	-0.33Z-k*	-0.38b-l*	-0.33Z-k*	-0.53i-o*	-0.68m-r*		
	RP	-0.13Q-b	-0.18S-d*	-0.23U-f*	-0.23U-f*	-0.48f-n*	-0.58k-p*		
	CPST	-0.23U-f*	-0.03M-W	-0.18S-d*	-0.28W-i*	-0.58k-p*	-0.83p-t*		
	ECST	-0.23U-f*	-0.43d-m*	-0.48f-n*	-0.48f-n*	-0.73n-r*	-0.83p-t*		
	UC	-0.18S-d*	-0.13Q-b	-0.28W-i*	-0.23U-f*	-0.43d-m*	-0.68m-r*		
Sil 2	MAP	0.05I–S	-0.05M-X	-0.30X-i*	-0.30X-i*	-0.40c-l*	-0.20S-e*		
	DAP	0.25D-K*	-0.15Q-c	-0.35Z-k*	-0.35Z-k*	-0.45e-m*	-0.40c-l*		
	TSP	0.25D-K*	0.40B-G*	0.25D-K*	0.15G–O	0.00K-U	0.05I–S		
	RP	0.50BCD*	0.60BC*	0.45B-E*	0.30D-I*	0.15G–O	0.25D-K*		
	CPST	0.20E-M*	0.65B*	0.50BCD*	0.25D-K*	0.05I-S	0.00K-U		
	ECST	1.15A*	0.20E-M*	0.40B-G*	0.15G–O	0.00K-U	0.00K-U		
	UC	0.65B*	0.60BC*	0.40B-G*	-0.10O-Z	0.20E-M	0.25D-K*		

TABLE 4 Summary of the combined effects of soil (loam, silty clay loam [SiCL], and silt loam [SiL]), fertilizer treatment, and sample time (0.5, 1, 2, 4, 6, and 9 mo) on the change in soil pH from initial soil levels (Table 2)

^aFertilizer treatments (Treat) used in this incubation included monoammonium phosphate (MAP), diammonium phosphate (DAP), triple superphosphate (TSP), rock phosphate (RP), chemically precipitated struvite (CPST), electrochemically precipitated struvite (ECST), and an unamended control (UC).

^bAll means for soil-fertilizer treatment combinations followed by different letters are different at p < .05. Due to the large number of treatment combinations, letter notations started with a set of uppercase letters and continued to a set of lowercase letters if necessary.

*Significant at the .05 probability level.

similar among all treatments (Table 4). Like the loam and SiCL soils, soil pH generally decreased over time in all fertilizer treatments and decreased from the initial value in all treatments by 9 mo of incubation. After 9 mo of incubation, the pH had decreased the most from the initial in the DAP treatment (-1.23 pH units), which did not differ from that for MAP (Table 4). Soil pH decreased the least in the RP treatment (-0.58 pH units), which did not differ from that for TSP, CPST, ECST, and UC (Table 4). Soil pH decreased the most in the SiL 1 out of the four soils, which was likely caused by a low CEC of the initial SiL 1 soil that would have reduced the ability of the soil to buffer a pH change caused by the dissolution of the P fertilizers (Brady & Weil, 2002).

Early in the incubation, the pH change in fertilizer treatments in the SiL 2 soil (i.e., the poorly drained SiL soil) exhibited a similar trend as the pH change in the SiCL soil. By 0.5 mo of incubation, soil pH from all fertilizer treatments increased from the initial value, except for MAP, which did not differ from a change of zero (Table 4). Soil pH increased the most from the initial value in the ECST treatment (1.15 pH units) at the 0.5-mo sampling (Table 4). In contrast with the other soils, soil pH did not considerably change over time in



FIGURE 1 Soil (loam, silty clay loam [SiCL], and silt loam [SiL]) effects, averaged over fertilizer amendments, on the change in water-soluble (WS) soil P concentrations from the initial value (Table 2) over time. Means within a panel with different letters are different at p < .05. An asterisk (*) indicates the mean change is different than zero (p < .05)

most of the fertilizer treatments in the SiL 2 soil. After 9 mo of incubation, the change in soil pH in MAP, TSP, RP, and CPST was similar to the pH change in each individual fertilizer treatment that occurred after only 0.5 mo of incubation (Table 4). However, by the 9-mo sampling interval, soil pH decreased from the 0.5-mo sampling in only the UC, ECST, and DAP treatments. Overall, soil pH increased from the initial value in RP and UC (0.25 pH units), decreased from the initial value in MAP and DAP (-0.20 and -0.40 pH units, respectively), and did not differ from a change of zero in TSP, CPST, and ECST (<0.05 pH units) at the 9-mo sampling (Table 4). The greater variability of soil pH change in the fertilizer treatments in the SiL 2 soil over time was likely related to the lower buffering capacity of the SiL 2 soil, in which the low initial SOM concentration and low concentration of exchangeable cations from the history of rice production decreased the soil's ability to resist a major change in soil pH (Brady & Weil, 2002).

3.3 | Changes in water-soluble nutrient concentrations

The change in WS-P concentrations, averaged across fertilizer amendments, differed among soils over time (p < .05; Table 3). Over the first 2 mo of the incubation, WS-P increased from the initial value in all soils, but increased more in the loam (34.7 and 30.7 mg kg⁻¹, respectively) than in any other soil (Figure 1). The substantially greater WS-P concentration in the loam soil was likely related to the greater initial WS-P concentration in the loam than in the other soils (Table 2). By the 6- and 9-mo sampling intervals, WS-P concentrations continued to numerically decrease from earlier (Figure 1). However, WS-P concentrations were still greater than the initial value at the 6- and 9-mo sampling intervals in the loam (17.7 and 14.6 mg kg⁻¹, respectively, which did not differ) and SiCL (9.28 and 10.6 mg kg⁻¹, respectively, which did not differ) soils, but no longer differed from the initial value in SiL 1 and SiL 2 soils (Figure 1). The change in WS-P concentrations at least numerically decreased over time due to the decreasing solubility of the fertilizers later in the experiment and the transformation of WS-P into less available forms (Nongqwenga et al., 2017).

The change in WS-P concentrations also differed among fertilizer amendments, averaged across soils, over time (p < .05; Table 3). By 0.5 mo of incubation, WS-P concentrations had increased from the initial value in the MAP, DAP, TSP, CPST, and ECST treatments (20.1, 35.2, 21.3, 30.8, and 41.6 mg kg⁻¹, respectively) and did not differ from the initial value in RP and the UC (Figure 2). Out of all fertilizer treatments, WS-P concentration increased more from the initial value in the ECST treatment, which did not differ from that for DAP, than in any other fertilizer treatment at the 0.5-mo sampling (Figure 2). The greater WS-P concentration in the ECST after 0.5 mo was likely caused by the smaller particle size and greater fertilizer-to-soil contact, which likely led to faster dissolution and redistribution of P into the soil (Degryse et al., 2016; Everaert et al., 2018), associated with the ECST material, which also had a relatively large WS-P concentration (Table 1). After 1 and 2 mo, WS-P concentration at least numerically decreased among fertilizer treatments, but the WS-P concentration had increased the most in the CPST treatment (29.2 and 21.5 mg kg⁻¹, respectively), which did not differ from MAP, DAP, TSP, and ECST. The consistency in the CPST treatment was likely related to the gradual dissolution of the CPST material, which likely has a reduced



FIGURE 2 Fertilizer amendment (monoammonium phosphate [MAP], diammonium phosphate [DAP], triple super phosphate [TSP], rock phosphate [RP], chemically precipitated struvite [CPST], electrochemically precipitated struvite [ECST], and unamended control [UC]) effects, averaged over soils, on the change in water-soluble (WS) soil P concentration from the initial value (Table 2) over time. Means within a panel with different letters are different at p < .05. An asterisk (*) indicates the mean change is different than zero (p < .05)

P fixation effect, as the fertilizer is slowly water soluble over time (Degryse et al., 2016). After both 6 and 9 mo, WS-P concentration continued to decrease among fertilizer treatments, but the WS-P concentration was again similar among all fertilizer treatments and greater than the initial value, except for RP and UC, which did not differ from the initial value (Figure 2). Water-soluble P concentrations likely did not change from the initial value in the RP treatment throughout the incubation due to RP's very low solubility, which limited the WS-P concentration.

Averaged across sampling times, the change in WS-Ca and -Mg concentrations differed among fertilizer treatments across soils (p < .05; Table 3). Water-soluble Ca concentrations increased from the initial value in all soil-fertilizer treatment combinations but were at least numerically greater in all fertilizer treatments in the SiL 1 and SiL 2 than in either the loam or SiCL soils (Figure 3), which was likely related to lower CECs of the SiL 1 and SiL 2 soils. In all soils, WS-Ca concentrations increased more from the initial value in the DAP treatment than in any other fertilizer treatment, which was somewhat unexpected since DAP had an initial WS-Ca concentration that was approximately 70 and 15 times less than that for the TSP and MAP treatments, respectively (Table 1, Figure 3). However, the WS-Ca concentration difference among treatments could have been a result of the substantial pH change that generally occurred in the DAP treatment, which likely affected the proportion of acids and bases

on exchange sites (Mengel, 1993). Within the loam and SiCL soils, WS-Ca concentration increased from the initial value and was intermediate in TSP (52.2 and 42.2 mg kg⁻¹, respectively), MAP (70.0 and 56.4 mg kg⁻¹, respectively), CPST (59.0 and 50.8 mg kg⁻¹, respectively), and ECST (64.4 and 59.1 mg kg⁻¹, respectively) and increased from the initial value and was smallest in the RP (27.2 and 25.3 mg kg⁻¹, respectively) and UC treatments (26.5 and 25.6 mg kg⁻¹, respectively), which did not differ (Figure 3). In the SiL 2 soil, WS-Ca concentrations increased from the initial value more in MAP (99.1 mg kg⁻¹) than for TSP, CPST, and ECST treatments (60.9, 74.3, and 78.0 mg kg⁻¹, respectively), which did not differ (Figure 3). Water-soluble Ca concentrations were likely at least numerically lower in the CPST and ECST treatments among all soils than in the MAP or DAP treatments due to the initial WS-Ca concentrations, which were considerably lower in the two struvite sources (Table 1). Like the loam and SiCL soils, WS-Ca concentrations increased from the initial value and were smallest in the RP and UC treatments in both SiL 1 (44.8 and 48.0 mg kg⁻¹, respectively) and SiL 2 (41.0 and 46.0 mg kg⁻¹, respectively) soils (Figure 3).

Water-soluble Mg concentrations were affected more by the different soils used in the incubation than the different fertilizer amendments. Like the change in WS-Ca, the change in WS-Mg concentrations were substantially lower in all fertilizer amendments in the loam and SiCL soils than that in the SiL 1 and SiL 2 soils, which was again likely related to the



FIGURE 3 Fertilizer amendment (monoammonium phosphate [MAP], diammonium phosphate [DAP], triple super phosphate [TSP], rock phosphate [RP], chemically precipitated struvite [CPST], electrochemically precipitated struvite [ECST], and unamended control [UC]) effects, averaged over time, on the change in water-soluble (WS) soil Ca and Mg concentrations from the initial value (Table 2) among soils (loam [L], silty clay loam [SiCL], and silt loam [SiL]). Means within a panel with different letters are different at p < .05. An asterisk (*) indicates the mean change is different than zero (p < .05)

greater CEC of the loam and SiCL soils (Figure 3). Within the loam soil, WS-Mg concentrations increased from the initial value in the MAP, DAP, CPST, and ECST treatments, decreased from the initial value in RP and UC treatments, and did not change from the initial value in the TSP treatment. Water-soluble Mg concentrations were greatest in the CPST treatment in the loam soil (15.3 mg kg⁻¹), which was expected due to the greater initial concentration of Mg in the CPST fertilizer (Table 1). In the SiCL soil, WS-Mg concentrations did not change from the initial in the MAP, DAP, CPST, and ECST treatments and had decreased from the initial value in the TSP, RP, and UC treatments. Within the SiL 1 and SiL 2 soils, the WS-Mg concentration increased from the initial value in all fertilizer treatments but increased the most in the DAP treatment (33.7 and 27.8 mg kg⁻¹, respectively) compared with any other treatment (Figure 3). In the DAP treatment, a more complete dissolution of the fertilizer pellet was observed over time and likely released a greater concentration of cations, which resulted in greater WS-Mg concentration increase than that in the other P fertilizers, despite having a low initial WS-Mg concentration in the DAP fertilizer (Table 1).

Averaged across fertilizer treatments, WS-Ca and -Mg concentrations also differed among soils over time (p < .05; Table 3). In all soils, WS-Ca concentrations generally increased over time and were roughly four times greater in each soil by the end of the incubation (i.e., after 9 mo; Figure 4). The general increase in WS-Ca concentrations was likely caused by the influx of cations by the dissolving fertilizers, which replaced Ca ions on soil exchange sites. After 0.5 mo, WS-Ca concentrations increased from the initial value and were similar among all soils (Figure 4). However, by 1 mo, WS-Ca concentrations had increased from the initial value in the loam (28.6 mg kg⁻¹), SiL 1 (25.2 mg kg⁻¹), and SiL 2 (31.9 mg kg⁻¹) soils and had not changed from the initial value in the SiCL soil (Figure 4). After 2 and 4 mo, WS-Ca concentrations continued to increase in all soils and increased more in the loam, SiL 1, and SiL 2 soils, which did not differ,



FIGURE 4 Soil (loam, silty clay loam [SiCL], and silt loam [SiL]) effects, averaged over fertilizer amendments, on the change in water-soluble (WS) soil Ca and Mg concentrations from the initial value (Table 2) over time. Means within a panel with different letters are different at p < .05. An asterisk (*) indicates the mean change is different than zero (p < .05)

than in the SiCL soil (Figure 4). The greater CEC of the SiCL soil likely limited the impact of the fertilizers on WS-Ca concentrations in the SiCL soil and reduced the overall increase in WS-Ca concentrations that was observed in the other soils. By 9 mo, WS-Ca concentrations had increased from the initial value in all soils, but the greatest increase occurred in the SiCL (148 mg kg⁻¹), SiL 1 (165 mg kg⁻¹), and SiL 2 (164 mg kg⁻¹) soils, which did not differ (Figure 4).

Like the change in WS-Ca, WS-Mg concentrations also generally increased across soils over time. By 0.5 mo, WS-

Mg concentrations decreased from the initial value in the loam $(-10.6 \text{ mg kg}^{-1})$ and SiCL $(-10.5 \text{ mg kg}^{-1})$ soils and had not changed from the initial value in SiL 1 and SiL 2 soils (Figure 4). After 1 mo, WS-Mg concentrations had increased from the initial value in SiL 1 and SiL 2 soils and had decreased from the initial value in the loam and SiCL soils (Figure 4). Water-soluble Mg concentrations were likely lower in the loam and SiCL soils due to increased CEC in both soils, in which a greater concentration of cations was adsorbed by the soil and ultimately would buffer an increase caused by the



FIGURE 5 Fertilizer amendment (monoammonium phosphate [MAP], diammonium phosphate [DAP], triple super phosphate [TSP], rock phosphate [RP], chemically precipitated struvite [CPST], electrochemically precipitated struvite [ECST], and unamended control [UC]) effects, averaged over soils, on the change in water-soluble (WS) soil Ca and Mg concentrations from the initial value (Table 2) over time. Means within a panel with different letters are different at p < .05. An asterisk (*) indicates the mean change is different than zero (p < .05)

influx of cations from dissolving fertilizers (Brady & Weil, 2002; do Nascimento et al., 2018; Mengel, 1993; Montalvo et al., 2014). After 6 and 9 mo, WS-Mg concentrations had increased the most from the initial value in the SiL 1 (34.5 and 48.4 mg kg⁻¹, respectively) compared with both loam and SiL 2 soils.

Averaged over soils, the change in WS-Ca and -Mg concentrations also differed among fertilizer treatments over time (p < .05; Table 3). Similar to the change in soils over time, the change in WS-Ca concentrations generally increased among all fertilizer amendments over time (Figure 5). After 0.5 mo, WS-Ca concentrations increased from the initial value and were similar among all fertilizer treatments, except for RP and UC treatments, which did not differ from the initial value (Figure 5). After 1 mo, WS-Ca concentrations had increased from the initial value and were greatest in the DAP treatment (40.0 mg kg⁻¹), which did not differ from MAP, CPST, and ECST treatments. The change in WS-Mg concentration also had increased from the initial value and was intermediate for TSP, and the WS-Ca concentrations did not change from the initial value in the RP and UC treatments (Figure 5). After 9 mo of incubation, WS-Ca concentrations were still greater than the initial value in all fertilizer treatments and had increased more in the DAP (197 mg kg⁻¹) than in any other treatment. Additionally, after 9 mo, WS-Ca concentration had increased more in the MAP (170 mg kg⁻¹) than in the TSP, CPST, and ECST treatments, which did not differ (Figure 5), likely due to a more complete dissolution of the fertilizer pellets, which was not observed for all fertilizers. The smallest increase in WS-Ca concentrations, once again, occurred in the RP (109 mg kg⁻¹) and UC (115 mg kg⁻¹) treatments after 9 mo (Figure 5). Although RP had the largest WS-Ca concentration among all fertilizers (Table 1), the low dissolution rate of RP likely limited the WS-Ca response in the RP treatment.

Like WS-Ca, WS-Mg concentrations generally increased among fertilizer amendments over time. After 0.5 mo, WS-Mg concentrations had not changed from the initial value in CPST and ECST treatments and decreased from the initial value in MAP (-5.2 mg kg^{-1}), DAP (-4.8 mg kg^{-1}), TSP $(-4.0 \text{ mg kg}^{-1})$, RP $(-9.8 \text{ mg kg}^{-1})$, and UC $(-10.7 \text{ mg kg}^{-1})$ treatments (Figure 5). After 1 mo, WS-Mg concentrations had increased from the initial value in the ECST, decreased from the initial value in the RP and TSP, and did not change from the initial value in all other fertilizer treatments (Figure 5). A greater concentration of WS-Mg was expected in the ECST treatment due to the composition of the struvite, which contained the greatest concentration of WS-Mg of all the P fertilizers (Table 1). After 9 mo, WS-Mg concentrations had increased from the initial value in all fertilizer treatments, except for the UC, which did not differ from the initial value (Figure 5).

The change in WS-Fe concentrations differed among fertilizer-soil treatment combinations over time (p < .05;Table 3). The change in WS-Fe concentrations was complex, and a clear trend was not observed across fertilizer treatments and commonly differed among soils over time. The WS-Fe concentrations generally decreased among fertilizer treatments over time in all soils (Table 5), which was likely caused by precipitation reactions, in which available P and Fe ions would precipitate into less available iron phosphates (Holford, 1997; Plaza et al., 2007; Tiessen et al., 1984). Despite the similar initial WS-Fe concentrations among soils (Table 2), WS-Fe concentrations generally decreased more from the initial value in the loam and SiL 2 soils, which was likely caused by the different CECs of the soils and the different WS-P response in each soil. In the loam soil, after 0.5 mo, WS-Fe concentrations had decreased the most from the initial value in the UC $(-46.5 \text{ mg kg}^{-1})$, which did not differ from that for DAP, TSP, RP, and ECST (Table 5). After 1 mo of incubation, WS-Fe concentrations had decreased from the initial value and were similar among all treatments (Table 5). Generally, WS-Fe concentrations continued to decrease in the loam soil over time and typically varied only slightly $(\pm 2.9 \text{ mg kg}^{-1})$ among treatments. After 9 mo, WS-Fe concentrations decreased the most from the initial value in MAP $(-48.4 \text{ mg kg}^{-1})$, which did not differ from that for DAP, CPST, ECST, and UC (Table 5). In the SiCL soil, after 0.5 mo, WS-Fe concentrations had decreased the most from the initial value in the CPST $(-37.5 \text{ mg kg}^{-1})$, which did not differ from that for the MAP, DAP, TSP, and ECST treatments. Similar to the loam soil, the change in WS-Fe concentrations slightly decreased over time and only slightly varied $(\pm 2.9 \text{ mg})$ kg^{-1}) among fertilizer treatments between 1 and 6 mo of incubation. After 9 mo, WS-Fe concentrations had decreased the most in the ECST $(-39.7 \text{ mg kg}^{-1})$, which did not differ from the MAP, DAP, TSP, CPST, and UC treatments (Table 5).

The change in WS-Fe concentrations in the SiL 1 and SiL 2 soils generally followed a similar trend, where, after 0.5 mo, WS-Fe concentrations had decreased the most from the initial value in RP (-37.0 and -55.3 mg kg⁻¹, respectively; Table 5), which did not differ from that for the MAP, DAP, and UC treatments in each individual soil. In both the SiL 1 and SiL 2 soils, after 1 mo, WS-Fe concentrations had decreased from the initial value and were similar among all treatments in each individual soil (Table 5). In the SiL 1 soil, after 4, 6, and 9 mo, WS-Fe concentrations had numerically decreased the most in the DAP treatment (-40.3, -40.5, and -40.1 mg kg⁻¹, respectively). In the SiL 2 soil, after 6 and 9 mo, WS-Fe concentrations had numerically decreased the MAP treatment (-58.8 and -58.9 mg kg⁻¹, respectively).

3.4 | Changes in Mehlich-3-extractable nutrient concentrations

The changes in all M3 soil nutrient concentrations were generally numerically larger than those in their WS soil nutrient concentrations due to the greater extractability with the moderately acidic M3 extraction solution. The change in M3-P concentrations, averaged over time, differed among fertilizer treatments across soils (p < .05; Table 3). In the loam, SiL 1, and SiL 2 soils, M3-P concentrations at least numerically increased from the initial value and were similar among MAP, DAP, TSP, CPST, and ECST treatments (Figure 6). Despite the slower dissolution rate of the two struvite sources (i.e., ECST and CPST), similar P availability was observed among the struvite sources and MAP, DAP, and TSP, where a similar result has been reported in several previous studies (Cabeza et al., 2011; Hall et al., 2020; Katanda et al., 2016; Tallboys et al., 2016). In the SiCL soil, the change in M3-P concentrations was much more complex. The M3-P concentrations increased the most from the initial value in the CPST treatment (223 mg kg⁻¹), which was

IADLL	5 Summary	of the combined e	inects of son (loani,	, sinty ciay loant [Sit	LJ, and shi toam [Si	LJ), lefulizer treatile	and sample time		
0.5, 1, 2, 4, 6, and 9 mo) on the change in water-soluble (WS) Fe from initial soil concentrations (Table 2)									
		Δ WS-Fe							
Soil	Treat ^a	0.5 mo	1 mo	2 mo	4 mo	6 mo	9 mo		
		mg kg ⁻¹							

Soil	Treat ^a	0.5 mo	1 mo	2 mo	4 mo	6 mo	9 mo
				mg k	cg ⁻¹		
Loam	MAP	-42.6Fg*b	-45.2h-k*	-45.2h-k*	-47.6m-s*	-47.51-s*	-48.4qrs*
	DAP	-45.0hij*	-45.9i-n*	-45.6h-n*	-48.7s*	-48.6rs*	-48.2p-s*
	TSP	-45.2h-k*	-46.2i-q*	-46.4i-q*	-46.0i-p*	-46.7j-s*	-45.5h-m*
	RP	-46.0i-p*	-46.3i-q*	-46.5j-s*	-46.5i-r*	-46.2i-q*	-46.0i-p*
	CPST	-44.2ghi*	-45.3h-l*	-45.6h-n*	-46.5j-s*	-47.3k-s*	-47.8n-s*
	ECST	-45.0hij*	-45.0hij*	-45.0hij*	-46.1i-q*	-48.10-s*	-47.8n-s*
	UC	-46.5j-s*	-45.9i-o*	-43.6gh*	-46.3i-q*	-47.0j-s*	-46.2i-q*
SiCL	MAP	-36.6C-S*	-36.8D-T*	-36.9D-T*	-37.5I-X*	-37.5I-X*	-39.3U-e*
	DAP	-36.4B-R*	-36.6C-S*	-34.6A-D*	-37.0E-T*	-38.3P-e*	-39.4W-e*
	TSP	-37.1E-U*	-36.9E-T*	-37.5I-X*	-37.7K-Z*	-37.8K-b*	-38.1N-d*
	RP	-34.6A-D*	-36.3B-Q*	-37.2E-W*	-37.5I-Z*	-38.0M-c*	-36.4C-S*
	CPST	-37.5I-X*	-36.1B-O*	-36.5C-S*	-38.1M-c*	-37.5I-Y*	-39.7X-e*
	ECST	-36.4B-R*	-36.9E-T*	-37.3G-W*	-37.9L-c*	-38.4P-e*	-39.7Y-e*
	UC	-34.5ABC*	-36.7D-S*	-35.7A-L*	-37.6J-Z*	-37.9L-b*	-39.0T-e*
Sil 1	MAP	-35.1A-G*	-36.0A-N*	-37.4H-W*	-39.0T-e*	-40.2cde*	-37.3F-W*
	DAP	-35.1A-F*	-36.3B-Q*	-39.4V-e*	-40.3de*	-40.5ef*	-40.1cde*
	TSP	-34.2AB*	-37.2E-W*	-35.8A-M*	-37.7K-a*	-38.5Q-e*	-39.8Z-e*
	RP	-37.0E-T*	-37.2F-W*	-35.0A-E*	-37.8K-b*	-37.9L-b*	-39.7Y-e*
	CPST	-35.6A-K*	-35.4A-J*	-35.3A-I*	-38.30-e*	-40.0b-e*	-40.0a-e*
	ECST	-33.8A*	-36.0A-N*	-36.2B-P*	-39.3U-e*	-40.0a-e*	-40.0a-e*
	UC	-36.4B-R*	-37.2E-V*	-35.2A-H*	-38.6R-e*	-38.7S-e*	-39.7X-e*
Sil 2	MAP	-54.4t-z*	-55.0u-aa*	–57.0aa–jj*	–58.5iijj*	-58.8iijj*	–58.9jj*
	DAP	-54.0t-w*	-56.2x-hh*	-58.4iijj*	–58.9jj*	-56.6y-ii*	-58.0ff-jj*
	TSP	-53.9tuv*	-54.9u-aa*	-55.0u-aa*	-56.1w-hh*	-56.1w-gg*	-58.3gg-jj*
	RP	-55.3u-cc*	-55.8v-ee*	-54.3t-x*	-56.0v-ff*	–57.5bb–jj*	-55.7v-ee*
	CPST	-52.4t*	-54.6u-z*	-54.4t-y*	-55.7v-ee*	-57.6dd-jj*	–58.5iijj*
	ECST	-54.5t-z*	-54.5t-z*	-53.2tu*	-56.7z-jj*	–57.9ee–jj*	-58.4hh-jj*
	UC	-55.2u-aa*	-55.5v-dd*	-55.2u-bb*	-56.1v-gg*	-54.5t-z*	–57.5cc–jj*

^aFertilizer treatments (Treat) used in this incubation included monoammonium phosphate (MAP), diammonium phosphate (DAP), triple superphosphate (TSP), rock phosphate (RP), chemically precipitated struvite (CPST), electrochemically precipitated struvite (ECST), and an unamended control (UC).

^bAll means for soil-fertilizer treatment combinations followed by different letters are different at p < .05. Due to the large number of treatment combinations, letter notations started with a set of uppercase letters and continued to a set of lowercase letters if necessary.

*Significant at the .05 probability level.

approximately four times greater than for any other fertilizer-P source (Figure 6). However, the unexpected M3-P concentration increase in the CPST treatment was caused by a substantially greater M3-P concentration in the 6-mo sampling interval (227 mg kg $^{-1}$), which was greater than the theoretical maximum P increase from fertilizer-P addition (134 mg kg⁻¹) and affected the overall mean M3-P concentration in the SiCL soil. However, the SiCL soil also had the largest initial M3-P concentration among the four soils (Table 2). Furthermore, as recently reported in Anderson et al. (2020), CPST pellets often remained intact in each replication throughout the incubation,

and one or more pellets were likely ground and analyzed with the soil as the soil cup was destructively sampled, as would have occurred had this study been conducted in the field.

Averaged over fertilizer treatments, M3-Ca and -Fe concentrations differed among soils over time (p < .05; Table 3). The change in M3-Ca concentrations generally decreased and was relatively stable across soils over time (Figure 7). Mehlich-3-extractable Ca concentrations likely decreased due to soil fixation processes, although a decrease in M3-Ca concentration was somewhat unexpected due to the influx of Ca ions into the soil solution by the dissolving fertilizers. However,



FIGURE 6 Fertilizer amendment (monoammonium phosphate [MAP], diammonium phosphate [DAP], triple super phosphate [TSP], rock phosphate [RP], chemically precipitated struvite [CPST], electrochemically precipitated struvite [ECST], and unamended control [UC]) effects, averaged over time, on the change in Mehlich-3 (M3)-extractable soil P and Fe concentrations from the initial value (Table 2) among soils (loam [L], silty clay loam [SiCL], and silt loam [SiL]). Means within a panel with different letters are different at p < .05. An asterisk (*) indicates the mean change is different than zero (p < .05)

since M3-Ca concentrations were generally much greater than other M3 nutrient concentrations, a slight increase in M3-Ca concentrations by the dissolving fertilizers may not have been quantified due to the inherent variability among initial soil samples. The change in M3-Ca concentrations was affected more by the different soils than time (Figure 7). Mehlich-3extractable Ca concentrations decreased more from the initial value in the SiCL soil after 0.5 mo $(-1,095 \text{ mg kg}^{-1})$ than in any other soil and were substantially lower than in all soils in the subsequent sampling times (Figure 7), which was likely related to the greater clay concentration and Ca fixation in the SiCL soil. The relatively large decrease in M3-Ca concentration, which was ~25% of total M3-Ca concentration in the SiCL, was somewhat unusual, and the cause was not immediately clear. After 9 mo, M3-Ca concentrations again decreased the most from the initial value in the SiCL soil



FIGURE 7 Soil (loam [L], silty clay loam [SiCL], and silt loam [SiL]) effects, averaged over fertilizer amendments, on the change in Mehlich-3 (M3)-extractable soil Ca and Fe concentrations from the initial value (Table 2) over time. Means within a panel with different letters are different at p < .05. An asterisk (*) indicates the mean change is different than zero (p < .05)

 $(-1,009 \text{ mg kg}^{-1})$ and decreased the least in the loam soil $(-74.2 \text{ mg kg}^{-1})$; Figure 7).

Averaged across soils and time, the change in M3-Ca concentration also differed among fertilizer treatments (p < .05; Table 3). Mehlich-3-extractable Ca concentrations decreased from the initial value in all fertilizer treatments but decreased the most in the CPST treatment $(-554 \text{ mg kg}^{-1})$ and decreased the least in the TSP treatment $(-454 \text{ mg kg}^{-1})$. The general decrease in M3-Ca concentration in all fertilizer treatments was likely related to the fixation of M3-Ca to clays that occurred throughout the experiment. The M3-Ca concentrations likely decreased the least in the TSP treatment because of the rapid dissolution of the TSP fertilizer pellets, which also had the greatest initial concentration of M3-Ca compared with other fertilizers (Table 1). Unlike TSP, the initial M3-Ca in the CPST material was the lowest among all fertilizers and likely was not fully solubilized over the course of the incubation, even after 9 mo (Table 1).

The change in M3-Mg concentration, averaged across soils and time, also differed among fertilizer treatments (p < .05; Table 3). Mehlich-3-extractable Mg concentrations increased from the initial value in CPST (39.2 mg kg⁻¹) and decreased from the initial value in all other fertilizer treatments, with the greatest decrease occurring in the RP treatment (-87.3 mg kg⁻¹). The greater M3-Mg concentration in the CPST treatment was expected due to Mg-containing composition of struvite. The lower M3-Mg concentration in the RP throughout the incubation was also expected due to the lowest initial M3-Mg fertilizer concentration in the RP treatment and the relatively low dissolution rate of RP (Table 1).

The change in M3-Mg concentrations, averaged over fertilizer treatments and time, also differed among soils (p < .05; Table 3). The change in M3-Mg concentrations varied substantially among soils. Mehlich-3-extractable Mg concentrations decreased from the initial value in the SiCL and SiL 2 soils and did not differ from the initial value in loam and SiL 1 soils. The greatest decrease in M3-Mg concentrations occurred in the SiL 2 soil (-135 mg kg^{-1}), and the greatest numerical increase in M3-Mg concentration occurred in SiL 1 (16.2 mg kg⁻¹). The variable M3-Mg response was somewhat unexpected since the initial M3-Mg concentration was intermediate in the SiL 1 and SiL 2 soils (Table 3);

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thus, the direct cause of the variation was not immediately clear.

Like M3-P concentrations, the change in M3-Fe concentrations, averaged over time, differed among fertilizer-soil treatment combinations (p < .05; Table 3). The change in M3-Fe concentrations was complex and varied considerably across fertilizer treatments among soils. The majority of the variation in M3-Fe concentrations was likely caused by the variable initial M3-Fe concentrations of the soils used in the incubation, where the initial M3-Fe concentrations for both the SiCL and SiL 1 soils were lower than those for the loam and SiL 2 soils (Table 2). Mehlich-3 Fe concentrations generally increased from the initial value among soil-fertilizer combinations in the SiCL 1 and SiL 1 soils, with a few exceptions (Figure 6). The M3-Fe concentrations generally did not differ from the initial value in the loam soil, except for DAP, which was greater than the initial value (15.6 mg kg⁻¹) and M3-Fe concentrations decreased from the initial value in the SiL 2 soil (Figure 6). In all soils, M3-Fe concentrations were lower in the RP and CPST, which did not differ from the UC, than in any other fertilizer treatment. The lower M3-Fe concentrations may have been caused by the slower dissolution rate of RP and CPST that would have prolonged the precipitation reactions of soil-solution P and exchangeable Fe, leading to a shift in the equilibrium concentration to the solution phase rather than the solid phase of the soil.

Like M3-Ca and -Mg concentrations, the change in M3-Fe concentrations, averaged over fertilizer treatments, differed among soils over time (p < .05; Table 3). The change in M3-Fe concentrations generally increased over time but varied substantially among soils. The variable M3-Fe response in all soils was likely greatly affected by the initial M3-Fe concentrations, where the initial M3-Fe concentrations for both the SiCL and SiL 1 soils were significantly lower than in the loam and SiL 2 soils (Table 2). After 0.5 mo, M3-Fe concentrations had increased from the initial value in the SiCL $(44.3 \text{ mg kg}^{-1})$, had not changed from the initial value in the SiL 1, and had decreased from the initial value in both the loam and SiL 2 (-16.0 and -135 mg kg⁻¹, respectively) soils (Figure 7). After 1 mo, M3-Fe concentrations had increased from the initial value in the SiCL and had decreased from the initial value in all other soils (Figure 7). After 6 mo, M3-Fe concentrations had increased from the initial value in the SiCL $(84.5 \text{ mg kg}^{-1})$, loam (23.6 mg kg⁻¹), and SiL 1 (52.0 mg kg^{-1}) soils and decreased from the initial value in the SiL 2 soil $(-54.7 \text{ mg kg}^{-1}; \text{Figure 7}).$

3.5 | Changes in soil inorganic N concentrations

The change in soil NO₃–N and NH₄–N concentrations differed among soil-fertilizer treatment combinations over time

(p < .05; Table 3). The change in soil NO₃–N concentration generally increased over time across soil-fertilizer treatment combinations, except between 6 and 9 mo, in which soil NO₂-N concentrations at least numerically decreased in the loam, SiL 1, and SiL 2 soils. The general gradual increase in soil NO₃–N was likely caused by microbial nitrification that converted applied NH4+ from the NH4+-containing fertilizers (i.e., MAP, DAP, CPST, and ECST) and the existing NH_4^+ in the soil into NO₃⁻ over time, particularly given the constant moist-soil conditions used for in the incubation. The change in soil NO₂-N concentration was at least numerically greater in the fertilizer treatments in both the SiL 1 and SiL 2 soils than in either the loam or SiCL soils (Table 6), which was likely related to the greater initial soil NO3-N concentration in the SiL 1 and SiL 2 soils (Table 2), as well as the different CECs of the soils.

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After 0.5 mo, soil NO₃–N concentrations increased from the initial value in the DAP-loam, CPST-loam, ECST-SiCL, and all fertilizer treatments in the SiL 1 and SiL 2 soils, whereas soil NO₃–N concentrations did not change from the initial value in all other treatment combinations (Table 6). Soil NO₃–N increased more in the DAP-SiL 2 and CPST-SiL 2 combinations than in any other soil–fertilizer treatment combination after 0.5 mo. In the loam, SiL 1, and SiL 2 soils, soil NO₃–N increased more from the initial value from DAP than from all other fertilizer treatments in every subsequent sampling time, with the exception of the ECST-SiL 1 combination after 1 mo and the MAP-loam combination after 9 mo, which did not differ from that for DAP (Table 6).

In the SiCL soil, soil NO₃-N changes from the initial value were similar among all treatments after 1 and 2 mo and averaged 7.0 and 11.9 mg kg⁻¹, respectively (Table 6). Although similar among all fertilizer treatments after 2 mo, soil NO₃-N concentrations increased from the initial value only in the MAP, DAP, CPST, ECST, and UC treatments, whereas soil NO₃-N concentrations did not change from the initial value in TSP or RP treatments (Table 6). The greater CEC of the SiCL soil was likely the reason that soil NO₃-N concentrations were relatively stable among fertilizer treatments earlier in the incubation due to the soil's ability to attract cations, such as NH_4^+ , and limit nitrification (Fenn & Kissel, 1976). Similar to the other soils, after 9 mo, soil NO₃-N concentrations increased the most from the initial value in the DAP treatment (98.8 mg kg⁻¹) compared with any other treatment in the SiCL soil (Table 6).

In contrast with the general increase in soil NO_3-N from initial concentrations in all fertilizer-soil combinations over time, soil NH_4-N concentrations (Table 7) generally decreased over time among fertilizer-soil combinations. Although a general decrease was observed among fertilizer treatments, the primary decrease occurred in the NH_4 containing fertilizers (i.e., MAP, DAP, CPST, and ECST) and generally did not substantially change among other fertilizer

		ΔNO_3-N								
Soil	Treat ^a	0.5 mo	1 mo	2 mo	4 mo	6 mo	9 mo			
				m	g kg ⁻¹					
Loam	MAP	9.7qq-xx ^b	30.5u-pp*	43.1m-dd*	79.10-b*	78.7O-b*	78.4O-b*			
	DAP	19.0hh-xx*	87.6L-T*	76.5Q–c*	125.2C-G*	135.9CD*	94.8J–Q*			
	TSP	7.6ss-xx	16.0ii–xx*	25.6bb-vv*	44.0m-dd*	41.4n-ff*	28.4x-rr*			
	RP	6.5uu–xx	11.500-xx	14.9kk-xx*	26.4z-uu*	29.4w-qq*	26.2aa-uu*			
	CPST	15.5jj–xx*	31.1t-pp*	53.8d-q*	70.2S-h*	85.2M-V*	73.2R-d*			
	ECST	13.2mm-xx	45.9k-bb*	52.3e-s*	75.5R–c*	75.2R-c*	66.4U-k*			
	UC	8.9rr-xx	16.2ii-xx*	21.8ee-xx*	28.5x-rr*	33.6q-ll*	27.3z-tt*			
SiCL	MAP	7.4tt-xx	11.800-xx	25.5bb-vv*	43.4m-dd*	53.2d-r*	63.2X-m*			
	DAP	5.3vv-xx	13.6ll-xx	29.7w-qq*	46.9j–aa*	68.7T-i*	98.8H–O*			
	TSP	3.9Xx	7.7ss-xx	11.700-xx	19.2hh-xx*	19.8gg-xx*	28.0y-ss*			
	RP	4.8wwxx	7.5ss-xx	12.6nn-xx	15.0kk-xx*	19.1hh-xx*	29.0w-rr*			
	CPST	4.5wwxx	10.6pp-xx	19.7gg-xx*	24.8cc-ww*	35.4q-kk*	61.8Y–n*			
	ECST	16.3ii-xx*	20.4gg-xx*	32.1s-oo*	41.2o-ff*	50.6h-v*	64.8V–l*			
	UC	6.7tt-xx	11.700-xx	17.2hh-xx*	17.6hh-xx*	24.4cc-xx*	29.1w-rr*			
Sil 1	MAP	28.1y-ss*	48.9i-x*	81.6N-Y*	113.4E–J*	137.7C*	117.9D–I*			
	DAP	36.3q-ii*	72.2R-f*	116.2D–I*	170.5B*	195.3A*	164.4B*			
	TSP	24.9cc-ww*	41.2n-ff*	51.6g-t*	80.5N-Z*	91.7K-R*	73.3R-d*			
	RP	24.3cc-xx*	35.8q-jj*	47.0j–z*	57.0c-p*	77.8P-b*	80.1N-a*			
	CPST	32.7r-nn*	45.8k-bb*	51.4g-t*	90.8K-S*	123.6C-G*	127.3C-F*			
	ECST	30.0v-qq*	63.5W-m*	72.8R–e*	118.6D–H*	137.8C*	108.3G–L*			
	UC	23.6dd-xx*	40.1o-gg*	49.0i-x*	73.0R-e*	89.6K-S*	70.4S-h*			
Sil 2	MAP	33.3q-mm*	60.5Z-o*	84.1N-W*	114.2E–J*	125.5C-G*	115.8D–I*			
	DAP	42.0n-ee*	90.8K-S*	130.4CDE*	174.8AB*	177.3AB*	160.0B*			
	TSP	19.5gg-xx*	33.3q-mm*	48.1i–y*	67.4T–j*	71.7R-g*	59.7a–o*			

TABLE 6 Summary of the combined effects of soil (loam, silty clay loam [SiCL], and silt loam [SiL]), fertilizer treatment, and sample time (0.5, 1, 2, 4, 6, and 9 mo) on the change in soil NO₃-N from initial soil concentrations (Table 2)

^aFertilizer treatments (Treat) used in this incubation included monoammonium phosphate (MAP), diammonium phosphate (DAP), triple superphosphate (TSP), rock phosphate (RP), chemically precipitated struvite (CPST), electrochemically precipitated struvite (ECST), and an unamended control (UC).

33.1r-nn*

45.0l-cc*

49.4i-w*

50.7h-u*

50.8h-u*

82.8N-X*

100.8H-N*

51.7f-t*

^bAll means for soil-fertilizer treatment combinations followed by different letters are different at p < .05. Due to the ¹arge number of treatment combinations, letter notations started with a set of uppercase letters and continued to a set of lowercase letters if necessary.

*Significant at the .05 probability level.

RP

CPST

ECST

UC

treatment combinations over time (Table 7). After 0.5 mo, soil NH₄–N concentration increased from the initial value in all MAP, DAP, CPST, and ECST treatments in all soils, aside from the CPST-SiL 2 combination, which not differ from a change of zero (Table 7). After 0.5 mo, soil NH₄–N concentrations increased from the initial value more in DAP than in any other fertilizer treatment among all soils (Table 7). After 1 month, the soil NH₄–N concentration had increased from the initial value in ECST-SiCL combination and in all MAP, DAP, and CPST treatments, with the exception of the MAP-SiL 2 combination, which did not change from the initial value in ECST-SiCL combination the initial value in ECST-SiCL combination and in all MAP.

17.2hh-xx*

41.8n-ee*

21.7ff-xx*

14.9kk-xx*

24.6cc-xx*

37.0p-hh*

65.3V-k*

26.7z-uu*

tial value, whereas in all other treatment combinations, soil NH_4 –N concentration did not change from the initial value (Table 7). After 9 mo, soil NH_4 –N concentration increased from the initial value in the DAP-SiCL and CPST-SiCL combinations and in all fertilizer treatment combinations in the SiL 1 soil, except for the RP-SiL 1 combination, which did not change from the initial value, whereas soil NH_4 –N concentration in all other treatment combinations also did not change from the initial value (Table 7). The greater solubility of DAP and the continued slow-release dissolution of CPST, particularly in the absence of organic acids from plant roots

76.5Q-c*

109.0F-K*

86.8M-U*

31.9s-oo*

58.7b-o* 97.3I-P*

102.7H-M*

58.8b-o*

		ΔNH_4-N					
Soil	Treat ^a	0.5 mo	1 mo	2 mo	4 mo	6 mo	9 mo
				mg l	kg ⁻¹		
Loam	MAP	35.0G-L* ^b	25.5K-T*	7.2V-n	1.6c-o	-0.9g-o	2.2b–o
	DAP	120.8A*	49.6DEF*	36.5F-K*	32.1H-N*	15.0R-d*	5.5Y–o
	TSP	-1.2h-o	-3.1j-o	-3.2j-o	-1.3h-o	-0.8f-o	2.8а–о
	RP	-2.3j-o	-3.3j-o	-3.3j-o	-0.7f-o	-1.6h-o	3.7Z–o
	CPST	44.7D-H*	26.5J-T*	18.3N-Y*	5.5Y–o	-0.1f-o	3.8Z-o
	ECST	35.7F-K*	9.4U–l	0.3e-o	-1.4h-o	-2.1j-o	2.1b–o
	UC	-1.7h-o	-3.1j-o	-3.2j-o	-1.6h-o	-1.2h-o	3.3Z-o
SiCL	MAP	25.1K-T*	23.4K-U*	20.0M-X*	6.3W–o	5.4Ү-о	10.6U–j
	DAP	80.9BC*	75.9BC*	70.3C*	54.6D*	44.7D-H*	22.6K-U*
	TSP	-2.6j-o	-5.0l-o	-4.3k-o	-2.9j-o	-4.3k-o	6.1X–o
	RP	-1.8i–o	-5.2mno	-4.4k-o	-2.4j-o	-3.6j-o	5.1Y–o
	CPST	44.1D–I*	21.0L-V*	35.6F-K*	31.5H–O*	12.8S-h*	14.7R–e*
	ECST	18.3N-Y*	14.6R-e*	12.4T-i*	5.1Y–o	2.6а–о	6.4W–o
	UC	-2.7j-o	-4.5l-o	-4.0k-o	-2.6j-o	-4.2k-o	5.1Ү-о
Sil 1	MAP	40.0E–J*	27.1J–S*	5.9X–o	1.9с-о	3.9Ү-о	15.8R–c*
	DAP	86.8B*	47.2D–G*	17.7N–Z*	17.50–Z*	7.5V–n	16.2Q-b*
	TSP	-1.4h-o	-3.8j-o	-3.5j-o	-0.7f-o	1.5d–o	13.7R-f*
	RP	2.7а–о	-2.9j-o	-2.3j-o	-1.1h-o	1.8c–o	9.4U–l
	CPST	30.0I–Q*	40.1E–J*	29.9I–Q*	20.6L-W*	15.7R–c*	13.5R-g*
	ECST	30.3H-P*	1.8c–o	–3.7ј–о	-2.0i-o	-0.3f-o	12.8T-h*
	UC	-0.4f-o	-2.9j-o	-2.2j-o	-0.3f-o	2.6а–о	17.50–Z*
Sil 2	MAP	27.7J-R*	10.1U-k	-4.1k-o	-3.1j-o	-2.4j-o	7.8V–m
	DAP	52.7DE*	17.0P–a*	-5.7mno	-2.2j-o	-4.2k-o	4.2Y–o
	TSP	-4.7l-o	-7.30	-6.4no	-3.7j-o	-4.4l-o	8.0V-m
	RP	-4.7l-o	-7.30	-6.0mno	-4.8l-o	-3.6j-o	4.5Y–o
	CPST	6.8V–n	17.7N–Z*	20.2M-X*	6.9V–n	-1.8i–o	10.0U-k
	ECST	33.8G-M*	-5.4mno	–5.4mno	-3.5j-o	-3.7j-o	4.0Y–o
	UC	-4.6l-o	-7.30	-5.9mno	-4.1k-o	-3.4j-o	6.2W–o

TABLE 7 Summary of the combined effects of soil (loam, silty clay loam [SiCL], and silt loam [SiL]), fertilizer treatment, and sample time (0.5, 1, 2, 4, 6, and 9 mo) on the change in soil NH₄–N from initial soil concentrations (Table 2)

^aFertilizer treatments (Treat) used in this incubation included monoammonium phosphate (MAP), diammonium phosphate (DAP), triple superphosphate (TSP), rock phosphate (RP), chemically precipitated struvite (CPST), electrochemically precipitated struvite (ECST), and an unamended control (UC).

^bAll means for soil–fertilizer treatment combinations followed by different letters are different at p < .05. Due to the large number of treatment combinations, letter notations started with a set of uppercase letters and continued to a set of lowercase letters if necessary.

*Significant at the .05 probability level.

(Degryse et al., 2016), were likely the major factors contributing to the continued greater soil NH_4 –N concentration in the DAP and CPST treatments over time. The general increase in soil NH_4 –N concentration observed in all fertilizer treatments in the SiL 1 soil at the 9-mo sampling interval was unexpected and the cause was unknown.

3.6 | Practical implications

Considering the fertilizer materials used in this study and despite no plants being present in the incubation experiment,

there would likely be few negative effects on crop productivity or soil quality unless soil pH and WS or M3 concentrations changed to beyond optimum ranges or threshold levels for optimal growth for a particular crop. However, with the lowest initial soil pH, the loam soil resulted in a large enough pH decrease in numerous fertilizer treatments at various times in the incubation such that soil pH dropped below 6.0, which, if not corrected with a liming material, could negatively affect nutrient availability, and thus negatively affect plant productivity. The low soil pH could also promote greater co-precipitation of P with Fe and minimize the potential co-precipitation of P with Ca. Although similar levels of pH decrease occurred from various fertilizers in the other soils as well, the initial soil pH of the other three soils was larger, such that the resulting pH decrease, on average, maintained soil pHs above 6.0. Furthermore, though M3-P concentrations were generally stable and WS-P concentrations generally numerically decreased, both P forms increased from their initial concentrations over the course of the 9-mo incubation, which showed that even the slow-release struvite materials could be viable fertilizer-P sources.

Intentional struvite recovery has the potential to decrease the global dependence for RP-derived fertilizers and provides an opportunity to recycle P from a number of different waste sources from various sectors. Results from this study provided evidence of the behavior of recovered struvite, in the absence of plants, in various soil textures historically managed for agricultural production. Despite the slow-release properties of struvite in general and the differing particle form, but similar to that concluded recently by Ahmed et al. (2018), results generated from this study demonstrated that ECST has comparable fertilizer-P behavior to commercially available CPST and conventional P fertilizers (i.e., MAP, DAP, and TSP) in multiple soil textures. As a recovered, fertilizer-P source, struvite has the potential to provide an alternative to traditional fertilizer-P sources (Ahmed et al., 2018) in the global agricultural production system, thus contributing to food security for future generations. Struvite also has the added benefit of providing Mg, which, as an essential plant nutrient, can become deficient in some soils.

In addition to global benefits of struvite recovery, the controlled recovery of struvite on a local scale can lead to a reduction in P and N loads of wastewater effluent and sewage sludge in WWTPs (Doyle & Parsons, 2002; Jaffer et al., 2002). Reduced P and N loads have the potential to decrease eutrophication, leading to cleaner surface waters nationwide. Although the benefits of intentional struvite recovery appear to be extensive, further research is needed to better understand the plant response to struvite fertilization and the economic feasibility of struvite recovery.

4 | CONCLUSIONS

Although the agronomic applications of recovered struvite have been observed in a few small plant studies, fewer studies have evaluated struvite dissolution dynamics. Furthermore, virtually no studies have examined soil–struvite interactions in soils in the United States with a history of row-crop agriculture, particularly for the new ECST material that has not been compared with other fertilizer-P sources nor previously studied in any soil environment.

Despite the limitations of this study, results supported the hypothesis that both struvite sources would have a similar WS- and M3-P response as MAP, DAP, and TSP under artifi-

cial incubation conditions and with differential granule size. Despite the slower dissolution rate of struvite, the change in WS-P concentration was not significantly lower in either struvite source over the duration of incubation, which was somewhat unexpected. The WS-P concentration differed among soil textures over time, and the greatest WS-P concentration change occurred in the loam soil followed by the SiCL soil throughout the entire incubation. The M3-P concentrations were generally similar among MAP, DAP, TSP, CPST, and ECST in each individual soil, except for the SiCL soil. The comparable WS- and M3-P concentrations among struvite sources and other fertilizer-P sources further support the findings of previous studies that demonstrated struvite's potential as a sustainable, alternative P fertilizer. The comparable WS- and M3-P concentrations of struvite and conventional fertilizer-P sources and the slower dissolution of struvite suggest a potential benefit of the utilization of struvite in agronomic applications due to the likely prolonged P availability over multiple plant growth stages.

Results showed that not only the chemical and physical properties of the different soils and fertilizers, but also the previous management histories in similar-textured soils (i.e., SiL 1 and SiL 2), affected the fertilizer response of the various fertilizer-P sources. The decision for agricultural producers regarding which fertilizer-P source to use will clearly need to consider field management history (i.e., upland or lowland cropping), soil texture, and timing of fertilizer applications to best tailor the most appropriate fertilizer-P source for optimal crop production results. To accurately assess the applicability of struvite, chemically or electrochemically precipitated, as an alternative fertilizer-P source, additional in-depth research is still necessary to better understand struvite behavior in additional soil environments (i.e., under floodedsoil conditions), soil textures, and in field studies with plants.

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AUTHOR CONTRIBUTIONS

Ryder Anderson: Data curation; Formal analysis; Investigation; Methodology; Writing-original draft. Kristofor Brye: Conceptualization; Funding acquisition; Project administration; Supervision; Writing-review & editing. Laszlo Kekedy-Nagy: Methodology. Lauren Greenlee: Funding acquisition; Writing-review & editing. Edward Gbur: Formal analysis; Writing-review & editing. Trenton Lee Roberts: Writingreview & editing.

CONFLICT OF INTEREST

The authors declare no conflict of interest.

ORCID

Kristofor Brye https://orcid.org/0000-0001-9353-4009 Trenton Roberts https://orcid.org/0000-0001-5558-7257

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