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Soil Chemical Property Changes over Time from Struvite Compared to Other Fertilizer-Phosphorus Sources in Multiple Soils

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Abstract

Studies have shown that phosphorus (P) recovered from wastewater as the mineral struvite [$MgNH_4PO_4 \cdot 6(H_2O)$] may be a viable alternative fertilizer-P source. This study aimed to compare the effectiveness of electrochemically precipitated struvite (ECST), reclaimed from synthetic wastewater, to other commercial fertilizer-P sources in cultivated soils from Arkansas [AR; silt loam (SiL) and loam (L)], Missouri (MO; SiL), and Nebraska [NE; SiL and sandy loam (SL)]. A plant-less, moist-soil incubation experiment, including ECST, chemically precipitated struvite (CPST), monoammonium phosphate (MAP), triple superphosphate (TSP), and an unamended control (UC), was conducted to quantify soil pH, nitrate (NO_3-N), ammonium (NH_4-N), and Mehlich-3 (M3)-P, -Ca, -Mg, and -Fe concentrations at 0.5, 1, 2, 4, and 6 months. All measured soil properties differed ($P < 0.05$) among fertilizer-P sources within soils over time. Soil-fertilizer combinations generally had an acidifying effect over time, with pH change from the initial lower at 6 than at 0.5 months and lower than the initial soil pH. Soil NO_3-N generally increased among fertilizer-P sources, ranging from an increase of 10.1 to 221 mg·kg^{-1} for AR-L-TSP after 1 month and NE-SiL-MAP after 6 months, respectively. Soil M3-P ranged from -29.6 mg·kg^{-1} in the AR-L-UC after 1 month to 429 mg·kg^{-1} AR-SiL-TSP after 0.5 months. Results showed that, over time, ECST had comparable pH and soil NO_3-N, NH_4-N, and M3-P, -Ca, -Mg, and -Fe behavior compared to CPST, MAP, and TSP across various soil textures.

Keywords

Electrochemically Precipitated Struvite, Chemically Precipitated Struvite, Soil Incubation, Soil Texture

1. Introduction

As human populations continue to increase globally, there is a considerably greater need for food, potable water, fertile soils, and a circular economy for environmental sustainability [1] [2] [3] [4]. To adequately support the needs of a growing human population, increased agricultural productivity is imperative, which will invariably cause a greater demand for fertilizers [5], thus depleting many natural nutrient deposits, such as phosphorites and apatites [*i.e.*, phosphorus (P) ores]. To sustain increased agricultural productivity, greater synthetic fertilizer production may be required, potentially leading to ore depletion and possibly upsetting global nutrient economies, potentially leading to increased food expenses [5].

Population swells not only drive crop productivity, but also produces a greater need for animal products and consequently more feed products. Since arable land area remains the same, intensive animal production systems become a necessity, which, in turn, will likely generate large amounts of nutrient-dense wastewater that, when discharged into the environment, can have major, negative implications to aquatic life and the environment (*i.e.*, increasing surface water eutrophication, algal blooms, and development of hypoxic zones) [6] [7].

Nutrient-dense wastewaters from intensified animal agriculture and/or municipal wastewaters do not have to lead to negative environmental implications [7]. Wastewaters are alternative sources of plant nutrients that can be re-claimed before being discharged into surface waters, potentially initiating harmful environmental issues. In the United States alone, over 131 billion L (34.5 billion gallons) of municipal wastewater are processed daily through publicly owned wastewater treatment plants (WWTPs) [8], while an additional 99.4 million L (26,150 million gallons) flow daily through WWTPs that use anaerobic digestion systems [9].

Wastewaters contain concentrated amounts of valuable plant nutrients, including P and nitrogen (N), which have the potential to be extracted and recovered before being released into natural water bodies [3]. The nutrient recapture process has the potential to enhance the quality of surface waters. Consequently, based on sheer volume, nutrient-rich wastewater {3810 and 60 mg·L^{-1} for ammonium-N (NH_4-N) and phosphate (PO_4^{3-}-P), respectively [10] and 300 and 63 mg·L^{-1} for NH_4-N and PO_4-P in swine lagoon wastewater, respectively [11]} could potentially add value to a WWTP's operation and serve as a renewable source of P and other essential plant nutrients [12].

To be effective, agricultural production systems must be practiced in a more sustainable manner, inspired by innovative, research-based solutions. Thus, new environmentally sound practices, coupled with new and developing technologies, such as electrochemical precipitation, are being developed and tested to recover nutrient compounds from wastewaters, such as the mineral struvite ($MgNH_4PO_4·6H_2O$), which can be converted into potential fertilizer-nutrient

products, thus converting the natural P cycle into a circular-P economy [1] [2] [3] [4]. In current systems, P passes through several local sinks and is obtained from various sources; however, globally, P is often inadequately cycled. Hence, more attention is recently being placed on efforts to narrow the gap in the P cycle by recapturing P from the current one-way, anthropogenic flow, where mined-P is lost in wastewater systems (*i.e.*, municipal, industrial, and agricultural). Phosphorus contained in sludge or manure is often over applied to land or landfills, and can be recovered for use as plant fertilizer [2]. In order to guarantee that there are sufficient food supplies and other plant-based items, clean water and a sustainable environment for future generations, it is necessary to capture P from the linear flow and introduce captured-P into the agricultural fertilizer-P supply. Recovering waste-P for reuse will help to establish a circular-P nutrient economy, mitigating P imbalances and reducing reliance on ore-mined P [2] [3] [11] [13].

Mined-P fertilizers are highly energy-intensive processes to convert phosphate rock into a form that is more readily available to plants, providing them with essential P for growth and development [14] [15]. Inorganic-P fertilizers are manufactured from the extraction of phosphate rock (PR) from apatite ores [14] [16]. Extraction is followed by the removal of impurities to increase the P concentration. Phosphoric acid is then produced by reactions with sulfuric acid followed by fertilizer production of monoammonium phosphate (MAP) or diammonium phosphate (DAP) by reacting with one or two molecules of ammonia, or triple superphosphate (TSP) by reacting crushed PR with the phosphoric acid, then granulation and the fertilizer is distributed for agricultural use [16].

Electrochemical struvite precipitation is a relatively new, eco-friendly, and less energy-intensive method of P capture and recycling, particularly from wastewater [15] [17] [18] compared to chemical precipitation. Electrochemical precipitation is a technique that is vital to mitigating the environmental impact of excess P deposition from agricultural, municipal, or industrial wastewater sources, while simultaneously providing an invaluable source of renewable, slow-release fertilizer-P for sustained crop production [18] [19]. The production of electrochemically precipitated struvite (ECST) involves the use of a pure, sacrificial Mg anode in an electrical cell, where the Mg is utilized to facilitate the precipitation of an equimolar struvite compound with 1 molecule of magnesium, 1 molecule of phosphate, and 1 molecule of ammonium [17] [18]. The precipitate is then harvested, washed to remove impurities, analyzed, and excess moisture removed. The ECST shares similar characteristics as the chemically precipitated struvite (CPST), like its partial solubility in water, but increasing solubility with a decrease in pH, and also the ability to increase soil pH over time [3] [19]. However, struvite precipitation and crystal formation are impacted by several factors, such as Mg^{2+}, NH_4^+, and PO_4^{3-} ion concentration, pH, ionic strength, mixing energy, temperature, and the presence of foreign ions [18] [20].

Since the various forms of struvite can be a comparable fertilizer-P source for

crop production [2] [3] [7] [13] [19] [21]-[30], commercially available CPST's dissolution dynamics have been studied in a series of plant-less, moist-soil incubation experiments in Arkansas loam, silt loam, and silty clay loam soils. The fertilizer-P materials used and compared included finely ground and pelletized CPST, MAP, DAP, TSP, and rock phosphate to determine their fertilizer-P behavior over a 6-month period in several Arkansas soils [31] [32] [33]. Results indicated that soil textures, fertilizer formulations, soil pH, fertilizer-P sources, and land managements are important factors to be considered when choosing struvite fertilizers [31]. However, a wider geographic assessment of struvite behavior in various agricultural soils is warranted.

The objective of this study was to evaluate the dissolution behavior over time of ECST and CPST compared to that of other commonly used, conventional fertilizer-P sources (*i.e.*, MAP and TSP) in a plant-less, soil incubation in multiple agricultural soils with varying soil textures. It was hypothesized that soils amended with CPST and ECST will have greater pH and larger nitrate-N (NO_3-N) and NH_4-N concentrations changes over time compared to soils amended with TSP, but will be similar to that for MAP. It was also hypothesized that the particle size (*i.e.*, pellets vs. crystals) of the struvite-P materials will allow for greater and quicker dissolution of ECST in various soil textures, where ECST would initially have a larger response, but would end up similar to that of CPST over time. It was further hypothesized that ECST will have comparable Mehlich-3 (M3)-P, -Ca, and -Fe, concentration changes to that of CPST, MAP, and TSP over time.

2. Materials and Methods

2.1. Soil Collection

Soils from agriculturally relevant regions of eastern Nebraska, southwest Missouri, and western and eastern Arkansas were collected for use in the plant-less moist-soil incubation study (Table 1). At each location, soils were collected in 18.9-L (5 gallons) buckets. Ten to 12 buckets of soil were manually collected from the top 15 to 20 cm (*i.e.*, above the local plow layer and above any prominent argillic horizon) at each location. All the soils were collected from the top 10 cm of soil and transported to the Arkansas Agricultural Research and Extension Center in Fayetteville, AR for later processing. The Arkansas soils included Roxana loam (AR-L; coarse-silty, mixed, superactive, nonacid, thermic Typic Udifluvents) [34], which was collected on 16 December, 2017 from a plowed field, near Kibler, AR in a floodplain with 0% slope at the Vegetable Research Station, and Calloway silt loam (AR-SiL; fine silty, mixed, active, thermic, Aquic Fraglossudalfs) [35], which was collected on 14 December, 2017 from a field at the Cotton Branch Experiment Station in Mariana, AR that was plowed and under a 16-year, wheat-soybean, double-crop production system, on a loess-covered stream terrace with < 0.5% slope. The Arkansas soils differed in origin, where

Table 1. Summary of the soil surface texture, mapped soil series, and partial taxonomic description of selected soils from Arkansas, Missouri, and Nebraska used in the plant-less, moist-soil incubation study.

State/Soil	Surface texture	Soil series	Taxonomic description
Arkansas (AR)			
AR-L	Loam (L)	Roxana	Udifluvents
AR-SiL	Silt loam (SiL)	Calloway	Fraglossudalfs
Missouri (MO)			
MO-SiL 1	Silt loam (SiL)	Creldon	Fragiudalfs
MO-SiL 2	Silt loam (SiL)	Dapue	Hapludolls
Nebraska (NE)			
NE-SiL	Silt loam (SiL)	Yutan	Hapludalfs
NE-SL	Sandy loam (SL)	Olmitz	Hapludolls

the Calloway silt loam was derived from loess deposited over alluvium on stream terraces and has a fragipan [35], while the Roxana loam originated from the deposition of loamy alluvium from the Arkansas River [34].

In Missouri, the Creldon (fine, mixed, active, mesic Oxyaquic Fragiudalfs) [36] and Dapue (fine-silty, mixed, active, mesic Fluventic Hapludolls) [37] soil series were collected on 19 March, 2019 from managed pasturelands located at the University of Missouri's Southwest Research Center, near Mount Vernon, MO, established in tall fescue (*Festuca arundinacea*). The Creldon soil (MO-SiL 1) was collected from a backslope position with approximately 1% slope, and consists of a thin layer of loess covering a sub-soil that is largely clay-rich and developed from a weathered limestone residuum [36]. The Dapue soil (MO-SiL 2) was collected from a <1% slope on a floodplain developed from silty alluvium [37].

In Nebraska, soils were collected from sections of the University of Nebraska's Eastern Nebraska Research and Extension Center near Mead. Olmitz sandy loam (fine-loamy, mixed, superactive, mesic Cumulic Hapludolls) [38] and Yutan silt loam (fine-silty, mixed, superactive, mesic, Mollic Hapludalfs) [39] were collected on 11 May, 2019. The Olmitz sandy loam (NE-SL) was collected from a cultivated field cropped with rye grass (*Lolium perenne*) in a footslope position with ~2% slope. The Olmitz soil had a dark brown to black, moist surface color that was friable, abundant with earthworms, and originated from loamy alluvium of mixed mineralogy deposition in drainage ways. The Yutan silt loam (NE-SiL) was collected from an annual corn-soybean rotation system on a backslope position with ~3% slope established with corn (*Zea mays*) at the time of soil collection. The Yutan soil had a very dark, grayish brown, moist surface color, with a thick layer of crop residue and abundant earthworms. The soils collected, two soils from each of the three states: Arkansas, Missouri, and Nebraska,

were examples of typical soils under agricultural management in the respective regions.

2.2. Soil Processing and Initial Analyses

All field-collected soil was moist-sieved through a 6-mm mesh screen. Debris, coarse fragments, and crop residues were also manually removed. The soil was then air-dried on greenhouse benches covered with a tarp. Soils were air-dried for a week at approximately 35°C in layers ~30 cm thick that were mixed daily to enable uniform drying. Subsamples of each soil were collected, oven-dried at 70°C for 48 hours, and ground to pass through a 2-mm mesh screen for physical and chemical property evaluations.

Soil pH was determined potentiometrically from a 1-part soil mass to 2-parts water volume suspension using an electrode. Soil NO_3- and NH_4-N concentrations were determined colorimetrically on a Skalar autoanalyzer (SAN$^+$ System Segmented Flow Analyzer, Skalar Analytical B.V., Breda, The Netherlands) after potassium chloride extraction and filtration using the Nesslerization and cadmium-reduction methods, respectively [40]. Following extraction with Mehlich-3 (M3) extractant solution in a 1:10 (wt/vol) soil-to-extractant solution suspension M3-extractable soil concentrations (*i.e.*, P, Ca, Mg, and Fe) were analyzed by inductively coupled, argon-plasma spectrometry (ICAPS; Spectro Analytical Instruments, Inc., Mahwah, NJ) [41]. Soil organic matter (SOM) concentrations were gravimetrically determined via weight loss-on-ignition after heating in a muffle furnace for 2 hours at 360°C [42]. Total carbon (TC) and total N (TN) concentrations were determined by high-temperature combustion using an Elementar VarioMax CN analyzer (Langenselbold, Germany). Sand, silt, and clay were determined via a modified 12-hr hydrometer method [43] to confirm the textural class of each soil (Table 2). Additional information about the six soils used in this study was reported in Simms [44].

2.3. Fertilizer-P Sources and Characterization

Selected fertilizer-P sources were used in the greenhouse potted-plant experiments, including 1) ECST (approximate fertilizer grade: 5-37-0) [44], precipitated from synthetic wastewater by means of electrolysis utilizing a sacrificial Mg anode and created by researchers in the Chemical Engineering Department at the University of Arkansas, 2) a commercially produced CPST material, chemically precipitated from wastewater (approximate fertilizer grade: 5-28-0 with 10% elemental Mg) [44] manufactured by Ostara Nutrient Recovery Technologies, Inc. from a wastewater treatment plant near Atlanta, GA, 3) MAP (fertilizer grade: 11-52-0), formed by the 1:1 molar blend of ammonia (NH_3) to phosphoric acid (H_3PO_4) and subsequent granulation, 4) TSP (fertilizer grade: 0-46-0), formulated from a reaction between rock phosphate (RP) and H_3PO_4 [45], and 5) an unamended control (UC) that received no P additions.

Fertilizer pH was determined potentiometrically from a 1-part ground fertilizer

Table 2. Summary of the initial soil physical and chemical properties among the six soils used in the plant-less, moist-soil incubation study.

Soil property[†]	AR-SiL[§]	AR-L	MO-SiL 1	MO-SiL 2	NE-SiL	NE-SL
Clay (g·g^{-1})	0.14 b[‡]	0.09 c	0.10 c	0.07 d	0.16 a	0.05 e
Sand (g·g^{-1})	0.12 e	0.44 b	0.24 c	0.19 d	0.24 c	0.64 a
Silt (g·g^{-1})	0.75 a	0.46 d	0.67 b	0.74 8 a	0.60 c	0.30 e
SOM (g·kg^{-1})	23.5 d	7.3 f	28.5 c	33.3 b	58.4 a	21.5 e
NO$_3$-N (mg·kg^{-1})	15.8 c	9.5 e	11.1 d	21.3 b	26.9 a	0.4 f
NH$_4$-N (mg·kg^{-1})	8.2 c	3.9 d	7.6 c	9.2 c	23.0 a	13.4 b
pH	6.5 a	6.2 b	6.0 c	5.9 d	6.2 b	6.0 c
Mehlich-3 extractable (mg kg^{-1})						
P	33.7 d	93.3 a	14.6 f	27.6 e	62.0 b	44.0 c
Ca	1842 b	933 e	1031 d	1474 c	3294 a	1056 d
Mg	444 b	194 e	291 c	212 d	495 a	155 f
Fe	186 b	201 a	100 f	158 c	121 e	137 d

[†]Soil organic matter (SOM); nitrate-nitrogen (NO$_3$-N); ammonium-nitrogen (NH$_4$-N). [§]AR-SiL = Arkansas silt loam, AR-L = Arkansas loam, MO-SiL 1= Missouri silt, MO-SiL 2 = Missouri silt loam, NE-SL = Nebraska sandy loam, and NE-SiL = Nebraska silt loam. [‡]Means in a row with different letters are different at $P < 0.05$.

mass to 2-parts water volume suspension using an electrode. Total C and TN concentrations of each fertilizer material were ascertained via high-temperature combustion using an Elementar VarioMax CN analyzer (Langenselbold, Germany). Mehlich-3 extractable concentrations (*i.e.*, P, Ca, Mg, and Fe) were determined by extraction using M3 extractant solution in a 1:10 (wt/vol) fertilizer-to-extractant solution suspension and analyzed by ICAPS (Spectro Arcos ICP) [41].

2.4. Soil Incubation Procedures

Similar to procedures used recently by Anderson *et al.* [31], a plant-less soil incubation study was conducted over a 180-day period beginning August 13, 2020 to February 10, 2021. Five, small holes (~1.5 cm in diameter) were made in the lids of small plastic soil cups, 10.5 cm in diameter at the surface by 4.5 cm tall, in which the soil-fertilizer mixture was incubated. The holes will serve as an inlet for air into the soil cups to facilitate gaseous exchange while the soil incubation proceeds.

Six agronomically important soils from Arkansas (AR-L and AR-SiL), Missouri (MO-SiL 1 and MO-SiL 2), and Nebraska (NE-SiL and NE-SL) were used in the soil incubation experiment. Soil cups containing each soil-fertilizer treatment combination was prepared in duplicate for each of the five sampling periods for a total of 300 soil cups prepared. Approximately 150 g of air-dried soil were placed into each soil cup. Fertilizer amendments (*i.e.*, ECST, CPST, MAP,

TSP, and a UC) were added to each soil cup at a single fertilizer-P rate equivalent to a 56 kg P_2O_5 ha^{-1} (24.46 kg P ha^{-1}) based on the average fertilizer-P recommendations for two common crops (corn and wheat) in all three states. The single fertilizer-P rate was used to calculate the quantity of each fertilizer to apply to each soil cup based on the surface area of the cup. The required fertilizer to be added to each cup equated to 121.8 ± 5 mg ECST, 170.7 ± 5 mg CPST, 93.7 ± 5 mg MAP, and 107.6 ± 5 mg TSP were applied to relevant soil cup based on the different soils. The required quantities of each fertilizer were added to the air-dried soils in the cups, after which each soil cup was shaken consistently vertically and in a circular manner for ~10 seconds to simulate fertilizer-soil incorporation via tillage.

Soil cups were periodically watered to a soil-dependent, pre-determined gravimetric water content to mimic natural field wetting and drying cycles. The target masses to which the soil cups were watered were obtained by using the calculated gravimetric water contents of the air-dried soils and the estimated bulk densities. The Soil, Plant, Atmosphere, Water (SPAW) Model [46] [47] was used to determine the gravimetric water contents using the estimated field moisture capacity for each soil. To begin with, the soil cups were watered with tap water using a wash bottle on the same day that the fertilizer was added and incorporated to wet each soil to the calculated target weight and then the cups were allowed to incubate at room temperature for 0.5, 1, 2, 4 and 6 months.

Soil cups were placed on three-shelf, wooden stands, 125.5 cm long by 123 cm wide, and 73 cm tall. Soil cups were randomly, but evenly, spaced on each of the three levels on the wooden stand. Soil cups were rotated every two weeks along the three shelves so as to ensure that all soil cups are exposed to the same environmental conditions (*i.e.*, light and air-flow) throughout the duration of the incubation period.

After the initial watering, the soils were rewetted bi-weekly to each soil's target mass, imitating a full natural wetting and drying cycle that would have occurred in the field. Following a series of wetting and drying cycles, as the soil settles, an estimated soil bulk density was computed for each soil using the known mass of soil that was added to the cup and the total volume of soil in the soil cup based on the height of soil in the cup. The final soil bulk densities were determined for each soil.

The entire soil incubation experiment was conducted in a laboratory, bench-top, climate-controlled environment. An Acurite thermometer (model 00554SBDI, Chaney Instrument Co., Lake Geneva, WI) was placed on the wooden soil incubation stand and used to measure the climatic conditions, such as variations in the ambient air temperature and relative humidity, periodically throughout the entirety of the soil incubation experiment.

2.5. Post-Incubation Soil Processing and Analyses

After each incubation period (*i.e.*, 0.5, 1, 2, 4, and 6 months), soil cups were de-

structively sampled by removing the soil from the plastic cups. At each sampling, the soil was moist sieved through a 2-mm mesh screen and oven-dried at 70°C for 48 hours and then manually re-sieved to pass through a 2-mm mesh screen. Soil pH, NO_3-N, NH_4-N, and M3-extractable soil nutrients (*i.e.*, P, Ca, Mg, and Fe) were analyzed similar to procedures used for initial soil properties. During the incubation period, soil cups will receive natural sunlight that passes through the laboratory glass window along with fluorescent lighting available when the laboratory is in use. After analysis, soil property changes over time were calculated by subtracting the initial mean for a given soil property from the measured soil property value of each soil, for each replication, at each sampling time.

2.6. Statistical Analyses

Based on a completely random design, a one-factor analysis of variance (ANOVA) was conducted using the PROC GLIMMIX procedure in SAS 9.4 (SAS Institute, Inc., Cary, NC) to evaluate soil property (*i.e.*, pH, percent sand, silt, and clay, and SOM, TC, TN, M3-extractable P, Ca, Mg, and Fe, NO_3-N, and NH_4-N concentrations) differences among the six soils used in the plant-less, moist-soil incubation experiment. Similar to Anderson *et al.* [31], a three-factor ANOVA was conducted using PROC GLIMMIX in SAS, based on a split-split-plot, randomized experimental design, to evaluate the effects of soil (*i.e.*, AR-L, AR-SiL, MO-SiL 1, MO-SiL 2, NE-SL, and NE-SiL), fertilizer treatment (*i.e.*, ECST, CPST, MAP, TSP, and UC), time (*i.e.*, 0.5, 1, 2, 4, and 6 months), and their interactions on the change in soil pH and NO_3-N, NH_4-N, and M3-extractable soil P, Ca, Mg, and Fe concentrations from their initial magnitudes. Soil was the whole-plot factor, fertilizer treatment was the split-plot factor, and time was the split-split-plot factor. When appropriate, least significant difference was used to separate means at the 0.05 level.

3. Results and Discussion

3.1. Initial Soil Property Differences

It was expected that initial soil properties would differ among the six soils due to differences in soil parent material, mineralogy, geography, topography, climatic region, and past agricultural management from which they were collected. The wide range of initial soil properties was specifically targeted and desired such that results of the current plant-less, moist-soil incubation study would have wide ranging application.

As expected, many initial property differences ($P < 0.05$) existed among the six agricultural soils used in the current plant-less, moist-soil incubation study (Table 2). Sand concentrations ranged from 0.12 g·g⁻¹ in the AR-SiL to 0.64 g·g⁻¹ in the NE-SL soil (Table 2). Clay concentrations among soils ranged from 0.05 g·g⁻¹ in the NE-SL to 0.16 g·g⁻¹ in the NE-SiL soil, while silt concentrations

ranged from 0.30 g·g^{-1} in the NE-SL to 0.75 g·g^{-1} in the AR-SiL soil (Table 2). Soil pH among all soils was slightly acidic, ranging from 5.9 in the MO-SiL 2 to 6.5 in the AR-SiL (Table 2). Soil organic matter ranged from 7.3 g·kg^{-1} in the AR-L to 58.4 g·kg^{-1} in the NE-SiL soil (Table 2). Soil NO$_3$-N ranged from 0.4 mg·kg^{-1} in the NE-SL to 26.9 mg·kg^{-1} in the NE-SiL soil, while NH$_4$-N ranged from 3.9 mg·kg^{-1} in the AR-L to 23.0 mg·kg^{-1} in the NE-SiL soil (Table 2).

Similar to other soil properties, initial M3 nutrient concentrations differed ($P <$ 0.05) among the six soils (Table 2). Initial M3-P concentrations ranged from 14.6 mg·kg^{-1} in the MO-SiL 1 to 93.3 mg·kg^{-1} in the AR-L soil, while initial M3-Ca ranged from 933 mg·kg^{-1} in the AR-L to 3294 mg·kg^{-1} in the NE-SiL soil (Table 2). Initial M3-Mg ranged from 155 mg·kg^{-1} in the NE-SL to 495 mg·kg^{-1} in the NE-SiL soil, while initial M3-Fe concentrations ranged from 100 mg·kg^{-1} in the MO-SiL 1 to 201 mg·kg^{-1} in the AR-L soil (Table 2). Table 2 summarizes all initial soil properties and their differences among the six soils.

3.2. Fertilizer Property Comparison

Similar to initial soil properties, it was expected that fertilizer properties would differ among the five fertilizer-P sources based on their origins and chemical compositions (Table 3). Fertilizer pH ranged from 2.4 for TSP to 8.8 for CPST, while the pH for ECST was 6.6 (Table 3). Total N ranged from 0.02% for TSP to 10.7% for MAP (Table 3). Mehlich-3-P concentrations ranged from 24.5 g·kg^{-1}

Table 3. Summary of fertilizer pH, total carbon (C) and nitrogen (N), Mehlich-3 extractable elemental concentrations, and fertilizer grade for electrochemically precipitated struvite (ECST), chemically precipitated struvite (CPST), monoammonium phosphate (MAP), and triple superphosphate (TSP) used in a six-month, plant-less, moist-soil incubation. Means (n = 5) are reported with standard errors in parentheses for all fertilizer properties, except pH for ECST where n = 3.

	Fertilizer-phosphorus source			
Fertilizer property[§]	CPST	ECST	MAP	TSP
pH	8.8 (0.1)	6.6 (0.01)	4.4 (0.02)	2.4 (0.02)
Total C (%)	0.2 (0.01)	0.1 (0.04)	0.3 (0.01)	0.3 (<0.01)
Total N (%)	5.7 (0.2)	5.1 (0.2)	10.7 (0.1)	0.02 (<0.01)
Mehlich-3 elements				
P (g·kg^{-1})	24.5 (0.3)	26.9 (0.20)	182 (1.3)	171 (3.8)
Ca (g·kg^{-1})	0.1 (<0.01)	<0.01 (<0.01)	1.9 (0.04)	106 (2.3)
Mg (g·kg^{-1})	21.4 (0.2)	23.8 (0.1)	6.8 (0.1)	4.7 (0.1)
Fe (g·kg^{-1})	0.1 (<0.01)	<0.01 (<0.01)	0.3 (<0.1)	0.4 (0.02)
Measured fertilizer grade[†]	6 - 26 - 0	5 - 37 - 0	11 - 47 - 0	0 - 41 - 0

[§]Fertilizer properties were reproduced from Simms (2023). [†]Fertilizer grade reported as percent N-P$_2$O$_5$-K$_2$O.

for CPST to 182 g·kg⁻¹ for MAP, while M3-Ca ranged from <0.01 g·kg⁻¹ for ECST to 106 g·kg⁻¹ for TSP (Table 3). Mehlich-3-Mg concentration ranged from 4.7 g·kg⁻¹ for TSP to 23.8 g·kg⁻¹ for ECST, while M3-Fe ranged from <0.01 g·kg⁻¹ for ECST to 0.4 g·kg⁻¹ for TSP (Table 3). Between the two struvite-P sources, ECST had larger M3-P and -Mg than CPST (Table 3).

While both ECST and CPST are struvites, they have different origins, hence, were expected to have many similarities, but may also have variations in their chemical composition. The ECST was made from a synthetic wastewater containing NH_4^+ and PO_4^{3-} ions forming the struvite precipitate with Mg ions from a sacrificial Mg electrode. Hence, ECST had greater purity than CPST, which was manufactured from municipal wastewater that naturally contained other ions, organic matter, and sediments. Similar fertilizer differences were reported in a recent plant-less, moist-soil incubation study by Anderson *et al.* [31], where ECST had larger M3-P and -Mg than CPST, but CPST had larger TN and M3-Ca and -Fe than ECST.

3.3. Soil Chemical Property Changes Over Time

At any given time, many chemical reactions are potentially occurring at the soil's interfaces as a result of pH dynamics and the interactions and exchanges of various ions, hence soil property changes were also expected to differ among soil and/or fertilizer-P source combinations over time. Changes among all measured soil properties differed ($P < 0.05$) among soil-fertilizer-P-source-time combinations (Table 4). All soil property changes over time were determined relative to the initial soil property mean for a given soil (*i.e.*, the initial soil property mean value was subtracted from the measured soil property at a given sampling time on a replication-by-replication basis).

Table 4. Analysis of variance summary of the effects of soil (S), fertilizer-phosphorus source (F), time (T), and their interactions on the changes in soil pH, nitrate (NO₃-N), ammonium (NH₄-N), and Mehlich-3 (M3)-extractable soil nutrients (*i.e.*, P, Ca, Mg, and Fe).

Soil property	Source of variation						
	S	F	T	S*F	S*T	F*T	S*F*T
pH	<0.01	<0.01	<0.01	0.14	<0.01	<0.01	**<0.01**
NO₃-N	0.03	<0.01	<0.01	<0.01	<0.01	<0.01	**0.1**
NH₄-N	0.07	0.12	<0.01	0.49	<0.01	<0.01	**<0.01**
M3-P	0.84	<0.01	<0.01	0.02	<0.01	<0.01	**<0.01**
M3-Ca	0.01	<0.01	<0.01	<0.01	<0.01	0.48	**<0.01**
M3-Mg	0.02	0.01	<0.01	0.93	0.03	<0.01	**0.05**
M3-Fe	<0.01	0.08	<0.01	0.36	<0.01	<0.01	**<0.01**

†Bolded values indicate significant effects at $P \leq 0.05$.

3.3.1. Soil pH

Soil pH changes from the initial over the 6-month period ranged from a decrease of −1.0 units in the AR-SiL, NE-SL, MO-SiL 1-MAP soil-fertilizer-P source combination after 4 months to an increase of 0.5 units in the AR-L-ECST and -CPST combinations after 0.5 months (Table 5). However, there was a general steady numerical decline in soil pH, relative to the initial, among all soil-fertilizer-P source combinations in the 0.5- and 4-month sampling times, but, after 6 months, all soil-fertilizer-P source combinations experienced a pH decrease, relative to the initial, except for the AR-L-ECST, -TSP, and -UC combinations for which the soil pH was similar again to the initial (Table 5). Only 25 of the 150 soil-fertilizer-P-source-time combinations did not differ from a change of zero relative to the initial (Table 5).

After 0.5 months, the soil pH changes relative to the initial between ECST and CPST did not differ and resulted in either no change or a small pH decrease among all fertilizer-P sources, except for the AR-L, where both struvites resulted in the largest pH increase (Table 5). In contrast to after 0.5 months, after 1 month, CPST did not cause a pH change relative to the initial in any of the six soils, except for the NE-SL, where there was a −0.4-unit decrease (Table 5). In contrast to CPST, after 1 month, ECST resulted in a −0.1 unit decrease for the AR-L and NE-SiL soils to a −0.5 unit decrease in the NE-SL soil (Table 5), which overall had the smallest buffering capacity (Table 2). After 2 and 4 months, all soils experienced pH decreases from the initial regardless of fertilizer-P source, except for AR-L-UC, which had not changed from the initial after 2 months and had increased by after 4 months (Table 5). After 6 months, all treatment combinations had smaller decreases from the initial from that measured after 4 months, except for the AR-L-UC and NE-SiL-MAP combinations, which had not changed from the initial, and the AR-L-MAP and AR-SiL-CPST combinations, which continued to decrease relative to the initial (Table 5). The largest pH decreases from the initial after 6 months occurred from MAP for all soils and there was generally a 0.1 to 0.2 unit increase from the initial soil pH among treatment combinations over that measured after 4 months (Table 5).

The general pH declines over time relative to the initial, particularly in the NE-SL and MO-SiL 1 and 2 soils, may have been due to their initially lowest pH (5.9) among the soils, and may also have been due to the coarsest texture of the NE-SL, thus reducing its buffering capacity (Table 2). Anderson *et al.* [31] also reported an acidifying effect among soil-fertilizer-P source combinations over time, similar to what occurred in the current study, and attributed the cause to microbial nitrification of ammonium and H^+ displacement from the soils' cation exchange sites by Mg^{2+} and possibly NH_4^+ as the fertilizer-P sources dissolved.

The greater pH declines after 2 and 4 months among soils amended with ECST than for CPST may have been due to the particle sizes of the initial fertilizer materials used. The CPST material was applied in pelletized form, while ECST was applied as a crystalline powder, hence would possess a larger surface

Table 5. Summary of soil pH changes relative to the initial pH in soils amended with electrochemically precipitated struvite (ECST), chemically precipitated struvite (CPST), monoammonium phosphate (MAP), and triple superphosphate (TSP) fertilizer-phosphorus (P) materials in a six-month, plant-less, moist-soil incubation study.

Soil	Fertilizer-P source	Incubation time (months)				
		0.5	1	2	4	6
AR-L[§]	CPST	0.5 A*[†]	0.1 B-D	−0.4 W-i*	−0.4 W-i*	−0.3 O-a*
	ECST	0.5 A*	−0.1 E-P*	−0.2 M-X*	−0.3 O-a*	0.0 C-G
	MAP	−0.1 D-M	−0.5 b-o*	−0.8 y-bb*	−0.8 y-bb*	−0.9 z-bb*
	TSP	0.0 C-G	0.0 C-K	−0.1 E-P*	−0.2 J-T*	0.0 C-G
	UC	0.2 B*	0.2 B*	0. 0 C-G	0.1 C-E	0.1 BC
AR-SiL	CPST	−0.2 I-T*	0.0 C-G	−0.3 R-e*	−0.5 c-p*	−0.6 m-w*
	ECST	−0.1 D-M	−0.2 N-X*	−0.4 a-n*	−0.5 i-u*	−0.4 W-i*
	MAP	−0.5 g-s*	−0.4 U-h*	−0.7 q-y*	−1.0 bb*	−0.8 x-z*
	TSP	−0.3 O-Z*	−0.3 O-Z*	−0.4 U-h*	−0.6 K-V*	−0.3 Q-b*
	UC	−0.1 E-O*	−0.1 D-M	−0.3 O-Z*	−0.4 Y-l*	−0.2 I-R*
MO-SiL 1	CPST	0.1 BC	−0.1 E-O	−0.4 a-n*	−0.8 x-aa*	−0.6 n-w*
	ECST	0.1 B-D	−0.3 O-a*	−0.6 m-w*	−0.7 w-z*	−0.5 h-t*
	MAP	-0.1 G-P*	−0.3 S-f*	−0.7 u-y*	−1.0 bb*	−0.9 aabb*
	TSP	-0.1 E-P*	−0.4 W-i*	−0.5 b-o*	−0.7 w-z*	−0.5 h-t*
	UC	0.0 C-E	−0.2 L-W*	−0.4 Y-l*	−0.7 v-z*	−0.5 b-o*
MO-SiL 2	CPST	0.0 C-F	0.0 C-J	−0.3 Q-c*	−0.7 s-y*	−0.5 h-t*
	ECST	0.1 BC	0.0 C-H	−0.5 a-n*	−0.6 n-w*	−0.4 X-j*
	MAP	−0.1 E-P*	−0.1 E-P*	−0.5 c-p*	−0.8 x-aa*	−0.6 m-w*
	TSP	−0.2 I-Q*	−0.2 H-Q*	−0.4 V-h*	−0.7 r-y*	−0.4 V-h*
	UC	0.0 C-K	0.1 B-D	−0.4 W-j*	−0.5 i-u*	−0.3 R-e*
NE-SiL	CPST	0.0 C-G	0.0 C-F	−0.2 I-S*	−0.4 Z-m*	−0.3 Q-d*
	ECST	0.0 C-I	−0.1 D-L	−0.4 X-k*	−0.5 f-r*	−0.4 T-g*
	MAP	−0.1 E-O*	−0.1 D-M	−0.3 Q-d*	−0.6 o-x*	−0.6 L-V*
	TSP	−0.2 H-P*	−0.2 G-P*	−0.4 X-j*	−0.5 a-n*	−0.3 P-a*
	UC	0.0 C-G	0.1 B-D	−0.3 R-e*	−0.4 W-j*	−0.3 P-a*
NE-SL	CPST	−0.1 F-P*	−0.2 N-X*	−0.4 W-j*	−0.7 t-y*	−0.5 i-u*
	ECST	−0.2 K-U*	−0.5 a-n*	−0.5 e-q*	−0.7 p-x*	−0.5 c-p*
	MAP	−0.4 Z-m*	−0.7 s-y*	−0.8 y-bb*	−1.0 bb*	−0.8 y-bb*
	TSP	−0.4 Z-m*	−0.5 b-o*	−0.4 W-i*	−0.6 m-w*	−0.5 b-o*
	UC	−0.3 N-Y*	−0.2 K-V*	−0.2 L-V*	−0.6 j-v*	−0.4 Y-l*

[§]AR-L = Arkansas loam, AR-SiL = Arkansas silt loam, MO-SiL 1 = Missouri silt loam, MO-SiL 2 = Missouri silt loam, NE-SL = Nebraska sandy loam and NE-SiL = Nebraska silt loam. [†]Means for soil-fertilizer-P source combinations followed by different letters are different at $P < 0.05$. Owing to the large number of similarities among soil-fertilizer-P source combinations, the corresponding letter notations began with a capital letter and continued to a second set of lowercase letters or doubled lower case letters. *An asterisk (*) specifies mean change is different than zero ($P < 0.05$).

area for soil-fertilizer contact, resulting in more rapid reactions leading to a quicker and larger change in pH among soils treated with ECST compared to CPST [31] [48]. Since the same trend was observed in the UC for each soil, though generally as a smaller pH change from the initial than for ECST, interactions among the soils' initial properties/components and microbial activity also likely contributed to the measured changes in soil pH in the UC treatments (Table 5).

After 6 months of incubation, the soil pH change was generally similar between ECST and CPST, but was less than the soil pH change from MAP among the various soils. Since all soils' initial pH was ≤6.5 (Table 1). The smaller the change in soil pH under the acidic conditions would likely be more conducive to crop growth, as nutrient availability would be less negatively impacted than if the soil pH decreased more. However, the actual soil pH, below which would be non-conductive for crop growth and productivity, would likely vary among soils due to potential differences in soil texture and SOM and other plant-nutrient concentrations.

3.3.2. Soil NO_3-N

Soil NO_3-N changes from the initial over the 6-month period ranged from an increase of 10.1 mg·kg^{-1} in the AR-L-TSP combination after 1 month to a much larger increase of 221 mg·kg^{-1} in the NE-SiL-MAP combination after 6 months (Table 6). However, there was a general steady numeric increase in soil NO_3-N, relative to the initial, among all soil-fertilizer-P source combinations across all sampling times (*i.e.*, 0.5 to 6 months), except for the AR-L-TSP and -UC and the AR-SiL-CPST, -ECST, and -TSP combinations, which had soil NO_3-N changes decrease after 1 month, but continued to increase steadily from 1 to 6 months (Table 6). After 6 months, all soil-fertilizer-P source combinations experienced a NO_3-N increase relative to the initial between the 0.5- and 6-month sampling times (Table 6). Only 5 of the 150 soil-fertilizer-P-source-time combinations did not differ from a change of zero relative to the initial (Table 6).

After 0.5 months, the soil NO_3-N change relative to the initial for ECST did not differ among fertilizer-P sources in any soil except for MAP and the UC in the AR-SiL and for CPST, TSP, and the UC in the NE-SL, where the ECST change was larger for each soil (97.7 and 58.8 mg·kg^{-1}, respectively; Table 6). In contrast, after 1 month, the soil NO_3-N change relative to the initial for ECST was larger than for CPST and the UC in the AR-L, AR-SiL, MO-SiL 1, and NE-SL soils; larger than for MAP in the MO-SiL 1 and NE-SiL soils; and also larger than for TSP in the AR-L, AR-SiL, MO-SiL 1, NE-SiL, and NE-SL soils, but similar in all other soil-fertilizer-P source combinations (Table 6). Similar to after 1 month, the soil NO_3-N change relative to the initial for ECST after 2 months was larger than for all other fertilizer-P sources in the MO-SiL 2 and NE-SiL soils (Table 6). However, the soil NO_3-N change relative to the initial for ECST after 2 months was larger than for CPST in the MO-SiL 1 and 2 and

Table 6. Summary of soil nitrate (NO₃-N; mg·kg⁻¹) concentration changes relative to the initial NO₃-N in soils amended with electrochemically precipitated struvite (ECST), chemically precipitated struvite (CPST), monoammonium phosphate (MAP), and triple superphosphate (TSP) fertilizer-phosphorus (P) materials in a six-month, plant-less, moist-soil incubation study.

Soil	Fertilizer-P source	Incubation time (months)				
		0.5	1	2	4	6
AR-L§	CPST	20.3 ee-hh*†	33.2 v-gg*	73.0 c-n*	77.3 Z-l*	80.9 Y-j*
	ECST	13.7 gghh	57.4 k-u*	67.7 g-p*	69.4 e-p*	70.1 d-p*
	MAP	16.9 ff-hh*	35.0 u-gg*	66.1 i-q*	78.3 Z-k*	83.6 X-i*
	TSP	13.5 gghh	10.1 hh	23.2 dd-hh*	27.3 aa-hh*	38.7 s-ee*
	UC	15.7 ff-hh	13.4 gghh	23.3 dd-hh*	31.9 w-hh*	38.4 s-ff*
AR-SiL	CPST	89.2 V-h*	51.0 n-z*	76.1 a-l*	103.8 P-Y*	153.5 CD*
	ECST	97.7 S-b*	76.7 a-l*	89.6 V-g*	108.0 N-W*	127.5 F-O*
	MAP	50.9 n-z*	59.2 j-s*	96.4 S-b*	115.2 J-T*	148 C-E*
	TSP	106.2 O-X*	37.5 t-ff*	48.4 p-bb*	66.9 h-p*	75.2 a-m*
	UC	28.5 z-hh*	38.6 s-ff*	51.9 n-y*	59.2 j-s*	83.2 X-i*
MO-SiL 1	CPST	25.9 aa-hh*	43.0 r-ee*	82.5 Y-i*	118.8 I-S*	135.0 D-K*
	ECST	31.5 w-hh*	69.8 d-p*	106.2 O-X*	135.2 D-K*	159.8 C*
	MAP	25.4 bb-hh*	43.6 q-dd*	96.4 S-b*	137.4 D-J*	155.6 CD*
	TSP	24.5 cc-hh*	41.3 s-ee*	66.5 h-p*	97.9 R-b*	109.1 N-V*
	UC	26.0 aa-hh*	42.0 s-ee*	68.8 e-p*	83.8 X-i*	110.1 M-V*
MO-SiL 2	CPST	32.4 v-hh*	54.4 l-w*	85.0 W-i*	121.6 H-Q*	144.7 C-G*
	ECST	34.6 u-gg*	71.6 c-n*	129.3 E-N*	139.1 C-I*	161.1 C*
	MAP	30.9 x-hh*	52.8 m-x*	93.6 T-c*	123.2 H-P*	143.7 C-H*
	TSP	31.8 w-hh*	50.4 n-z*	79.3 Z-k*	108.1 N-V*	116.7 I-S*
	UC	31.8 w-hh*	48.8 o-aa*	90.4 U-g*	98.1 R-a*	118.3 I-S*
NE-SiL	CPST	32.9 v-hh*	71.9 c-n*	100.1 Q-Z*	134.5 D-K*	190.9 B*
	ECST	34.8 u-gg*	82.6 Y-i*	136.1 D-J*	147.6 C-F*	185.1 B*
	MAP	33.3 v-gg*	69.0 e-p*	111.2 L-V*	155.8 CD*	220.6 A*
	TSP	33.3 v-gg*	68.8 e-p*	91.5 U-e*	114.8 J-T*	138.9 C-I*
	UC	28.4 z-hh*	68.4 f-p*	92.5 T-d*	120.9 H-R*	139.7 C-I*
NE-SL	CPST	29.4 y-hh*	58.9 j-t*	75.7 a-m*	91.1 U-f*	123.4 G-P*
	ECST	58.8 j-t*	91.6 U-e*	96.7 S-b*	112.9 K-U*	132.8 D-M*
	MAP	40.2 s-ee*	75.1 b-m*	96.2 S-b*	129.0 E-O*	133.2 D-L*
	TSP	35.1 u-gg*	48.3 p-bb*	50.7 n-z*	71.6 c-o*	78.0 Z-k*
	UC	30.2 x-hh*	47.0 p-cc*	55.1 l-v*	66.0 i-r*	76.0 a-l*

§AR-L = Arkansas loam, AR-SiL = Arkansas silt loam, MO-SiL 1 = Missouri silt loam, MO-SiL 2 = Missouri silt loam, NE-SL = Nebraska sandy loam and NE-SiL = Nebraska silt loam. †Means for soil-fertilizer-P source combinations followed by different letters are different at $P < 0.05$. Owing to the large number of similarities among soil-fertilizer-P source combinations, the corresponding letter notations began with a capital letter and continued to a second set of lowercase letters or doubled lower case letters. *An asterisk (*) specifies mean change is different than zero ($P < 0.05$).

NE-SiL soils and larger than for TSP and the UC in all six soils (Table 6). The soil NO_3-N change for ECST after 2 months was similar to that for MAP (156 mg·kg^{-1}) in the MO-SiL 1, and 35.5 mg·kg^{-1} smaller than MAP in the NE-SiL soil (Table 6). After 4 months, the soil NO_3-N change relative to the initial for ECST was larger than for TSP and the UC in all six soils, but did not differ from that for MAP in any soil (Table 6). After 6 months, while all soil-fertilizer-P source combinations were at their largest NO_3-N concentration change, the NO_3-N change for ECST did not differ from that for CPST and MAP in the AR-L, MO-SiL 2, and NE-SL soils, but was less than for CPST and MAP in the AR-SiL soil (Table 6). However, after 6 months, the soil NO_3-N change relative to the initial for ECST was larger than for TSP and UC in all soils (Table 6).

After 6 months, the soil NO_3-N change relative to the initial for ECST was statistically and/or numerically larger, up to 2 times in the NE-SL after 0.5 months, than for CPST in all soils, except for AR-L after 2 and 4 months, where CPST was 1.1 times numerically larger than for ECST (Table 6). After 6 months, soil NO_3-N change relative to the initial for ECST did not differ from CPST in four of the six soils, but was 1.2 times smaller than CPST in the AR-SiL, but was 1.2 times larger than for CPST in the MO-SiL 1 soil (Table 6).

3.3.3. Soil NH_4-N

Soil NH_4-N changes from the initial over the 6-month period ranged from a decrease of −15.1 mg·kg^{-1} in the NE-SiL-TSP combination after 6 months to an increase of 191 mg·kg^{-1} in the AR-SiL-MAP soil-fertilizer-P source combination after 0.5 months (Table 7). However, there was a general steady numerical decrease in soil NH_4-N, relative to the initial, among all soil-fertilizer-P source combinations over the 6 months, with a few fluctuating increases and decreases in NH_4-N between sampling times, particularly for the AR-L and AR-SiL soils (Table 7). Furthermore, after 6 months, all soil-fertilizer-P source combinations experienced an NH_4-N concentration decrease or at least a smaller increase, relative to the initial, than after 0.5 months (Table 7). Only 41 of the 150 soil-fertilizer-P-source-time combinations differed from a change of zero relative to the initial, mostly between the 0.5- and 1-month sampling times (Table 7).

After 0.5 months, the soil NH_4-N change relative to the initial for ECST did not differ from that for MAP in four of the six soils (*i.e.*, AR-L, MO-SiL 1 and 2, and NE-SiL), but was 122 and 23.2 mg·kg^{-1} smaller than for MAP in the AR-SiL and NE-SL soils, respectively (Table 7). The soil NH_4-N change relative to the initial for TSP and the UC was generally smaller than for ECST among all soils after 0.5 months, except in the AR-L, where soil NH_4-N change relative to the initial for ECST (17.5 mg·kg^{-1}) did not differ among any fertilizer-P source, and in the AR-SiL, where TSP had larger soil NH_4-N change relative to the initial than for ECST (69.3 mg·kg^{-1}; Table 7). In contrast, after 1 month, the soil NH_4-N change relative to the initial for ECST was less than for CPST and MAP

Table 7. Summary of soil ammonium (NH₄-N; mg·kg⁻¹) concentration changes relative to the initial NH₄-N in soils amended with electrochemically precipitated struvite (ECST), chemically precipitated struvite (CPST), monoammonium phosphate (MAP), and triple superphosphate (TSP) fertilizer-phosphorus (P) materials in a six-month, plant-less, moist-soil incubation study.

Soil	Fertilizer-P source	Incubation time (months)				
		0.5	1	2	4	6
AR-L§	CPST	39.6 I-Q*†	20.7 O-a*	10.0 U-n	0.3 Z-q	−0.1 Z-q
	ECST	17.5 Q-c*	−1.9 b-q	2.7 X-q	−0.4 Z-q	−0.4 Z-q
	MAP	36.8 K-S*	15.9 R-e*	7.6 U-p	8.2 U-p	0.7 Y-q
	TSP	3.9 X-q	−1.8 b-q	2.7 X-q	1.2 X-q	−1.8 b-q
	UC	9.7 U-o	−1.8 b-q	2.4 X-q	0.4 Z-q	−1.3 a-q
AR-SiL	CPST	89.3 C*	29.2 M-U*	6.8 V-q	3.1 X-q	−7.3 j-q
	ECST	69.3 C-F*	−6.7 g-q	−4.6 c-q	−6.4 f-q	−8.6 l-q
	MAP	190.8 A*	15.9 R-e*	0.1 Z-q	−3.9 c-q	−7.4 j-q
	TSP	161.1 B*	−5.3 d-q	−7.2 i-q	−6.1 e-q	−9.5 m-q
	UC	0.0 Z-q	−8.2 l-q	−4.9 d-q	−6.1 e-q	−8.5 l-q
MO-SiL 1	CPST	56.2 E-L*	72.4 C-E*	15.3 S-g	11.2 U-m	5.9 V-q
	ECST	45.8 G-N*	22.9 O-Y*	2.8 X-q	−0.5 Z-q	−5.5 d-q
	MAP	60.6 D-I*	60.2 D-I*	27.2 N-V*	4.9 W-q	−1.2 a-q
	TSP	19.8 P-b*	13.2 T-l	−0.5 Z-q	0.1 Z-q	−5.5 d-q
	UC	20.6 O-a*	8.5 U-p	−0.3 Z-q	−1.7 b-q	−5.4 d-q
MO-SiL 2	CPST	57.7 E-K*	51.3 E-M*	15.1 S-h	1.1 X-q	4.1 X-q
	ECST	46.8 G-N*	37.9 J-R*	3.0 X-q	−2.2 b-q	−6.5 f-q
	MAP	62.0 D-H*	61.4 D-I*	6.3 V-q	14.7 S-k	5.7 V-q
	TSP	11.0 U-n	15.0 S-i	−1.0 a-q	−2.9 c-q	−6.8 g-q
	UC	14.8 S-j	10.7 U-n	9.4 U-o	−3.7 c-q	−7.1 h-q
NE-SiL	CPST	80.8 CD*	39.4 I-Q*	34.1 L-T*	16.6 R-d*	−1.7 b-q
	ECST	50.0 F-M*	39.9 H-P*	0.8 X-q	−11.0 n-q	−13.2 pq
	MAP	63.7 D-G*	59.5 D-J*	26.7 N-W*	−0.4 Z-q	−9.6 m-q
	TSP	9.1 U-o	6.2 V-q	−4.7 d-q	−12.3 o-q	−15.1 q
	UC	21.2 O-Z*	2.4 X-q	−8.8 l-q	−13.6 pq	−13.6 pq
NE-SL	CPST	21.5 O-Z*	36.2 K-S*	15.6 S-f	−3.2 c-q	−3.6 c-q
	ECST	19.4 P-b*	−4.2 c-q	−6.0 e-q	−10.1 m-q	−10.0 m-q
	MAP	42.6 G-O*	22.9 O-X*	1.1 X-q	−7.0 h-q	−8.4 l-q
	TSP	-6.3 f-q	−7.5 k-q	−8.4 l-q	−8.9 l-q	−10.8 m-q
	UC	-8.8 l-q	−2.9 c-q	−5.8 e-q	−9.6 m-q	−10.9 m-q

§AR-L = Arkansas loam, AR-SiL = Arkansas silt loam, MO-SiL 1 = Missouri silt loam, MO-SiL 2 = Missouri silt loam, NE-SL = Nebraska sandy loam and NE-SiL = Nebraska silt loam. †Means for soil-fertilizer-P source combinations followed by different letters are different at $P < 0.05$. Owing to the large number of similarities among soil-fertilizer-P source combinations, the corresponding letter notations began with a capital letter and continued to a second set of lowercase letters. *An asterisk (*) specifies mean change is different than zero ($P < 0.05$).

in the AR-SiL, MO-SiL 1, and NE-SL soils; less than for CPST in the AR-L and less than for MAP in the MO-SiL 2 soil (Table 7). Soil NH_4-N change relative to the initial for ECST was similar to TSP and the UC in all soils, except for the MO-SiL 2 and NE-SiL, where ECST (37.9 and 39.9 mg·kg^{-1}, respectively) was larger (Table 7). Contrary to after 1 month, the soil NH_4-N change relative to the initial for ECST after 2 months was similar to all soil-fertilizer-P source combinations, except for the MO-SiL-1-MAP (27.2 mg·kg^{-1}) and NE-SiL-CPST and -MAP combinations (34.1 and 26.7 mg·kg^{-1}, respectively), which were all larger than for ECST (0.8 mg·kg^{-1}; Table 7). Similar to after 2 months, after 4 months, the soil NH_4-N change for ECST was similar to that for all soil-fertilizer-P source combination, except for MO-SiL 1 and NE-SiL-CPST combinations (11.2 and 16.6 mg·kg^{-1}, respectively; Table 7). After 6 months, while all soil-fertilizer-P source combinations were at their smallest NH_4-N concentration change, the soil NH_4-N change for ECST did not differ among any soil-fertilizer-P source combination (Table 7).

After 0.5 months, the soil NH_4-N change relative to the initial between ECST and CPST did not differ among soils, except for the NE-SiL, where the change for CPST was 30.8 mg·kg^{-1} larger than ECST (Table 7). In contrast to after 0.5 months, after 1 month, CPST had a larger NH_4-N change than ECST, relative to the initial, in four of the six soils (*i.e.* AR-L, AR-SiL, MO-SiL 1, and NE-SiL), but did not differ in the MO-SiL 2 and NE-SL soils (Table 7). After 2 to 6 months, ECST and CPST did not differ in any soil, except for in the NE-SiL after 2 and 4 months, where CPST was larger, but did not differ in any soil after 6 months (Table 7).

Changes in soil NO_3-N and NH_4-N concentrations relative to the initial behaved oppositely among soil-fertilizer-P source combinations over the 6-month period (Table 6 and Table 7). Changes in soil NO_3-N concentrations generally increased among all soil-fertilizer-P-source-time combinations, while soil NH_4-N concentrations changes generally decreased over time. Since the soil was not sterilized, the increase in NO_3-N was possibly due to soil microbial activity, leading to nitrification of NH_4^+ ions and SOM into NO_3^- by microbes, as evidenced by NH_4-N concentrations decreasing over time, achieving its peak decline at the same time that NO_3-N achieved its peak increase. Thus, it is likely that NH_4-N was converted NO_3-N in the soil via microbial activity, such as ammonia (*Nitrosomonas*)- and nitrite (*Nitrobacter*)-, or complete ammonia-oxidizing (*Nitrospira*) microbes that are known to carry out NH_4^+ to NO_3^- conversions in the soil [49] [50] [51]. Moreover, the soils amended with TSP, a non-NH_4-N-containing fertilizer, and the UC generally had smaller NH_4-N changes over time than for the soils amended with NH_4-N-containing fertilizer-P sources.

In a 6-week soil incubation study on soil carbon and nitrogen dynamics in a manured fine-sandy-loam-textured Ultisol (Christiana series), Calderón *et al.* [52] reported a similarly opposite NO_3-N and NH_4-N concentration behavior,

which was described to be microbially driven, resulting in mineralized ammonium and conversion to nitrate. Moreover, in a 9-month soil incubation study comparing extractable nutrients from ECST to that of other fertilizer-P sources, including those used in the current study, Anderson *et al.* [33] also reported similar soil NO_3-N and NH_4-N responses in a loam, silt-loam, and silty-clay-loam soil from Arkansas. Both aforementioned studies agreed that soil NH_4-N mineralization via nitrification to nitrate was the reason for the decreased NH_4-N and increased NO_3-N over the soil incubation periods. Other potentially lesser contributory factors to the degree of NH_4-N nitrification may have been due to slight initial differences in soil pH, soil texture and cation exchange capacity, SOM, and initial NO_3-N and NH_4-N concentrations in the incubated soils [33] [53] [54].

3.3.4. M3-P

Mehlich-3-P concentration changes from the initial over the 6-month period fluctuated consistently from one sampling time to another (Table 8). The M3-P change relative to the initial ranged from a decrease of −29.6 mg·kg⁻¹ in the AR-L-UC combination after 1 month to an increase of 429 mg·kg⁻¹ in the AR-SiL-TSP combinations after 0.5 months (Table 8). Only 48 of the 150 soil-fertilizer-P-source-time combinations did not differ from a change of zero relative to the initial (Table 8).

After 0.5 months, the M3-P change relative to the initial for ECST (73.5 mg·kg⁻¹) did not differ among any fertilizer-P sources in the NE-SiL soil, or among CPST, MAP, and TSP fertilizer-P source in the MO-SiL 1 (105 mg·kg⁻¹), but was larger than for the MO-SiL-1-UC (Table 8). In contrast, after 0.5 months, the M3-P change relative to the initial for ECST was numerically largest among fertilizer-P sources, but was similar to that for MAP and TSP, and larger than for CPST and the UC in the AR-L, AR-SiL, MO-SiL 2, and NE-SL soils (Table 8). However, for the AR-SiL soil, the M3-P change relative to the initial for MAP and TSP (413 and 429 mg·kg⁻¹, respectively) was larger than for ECST (225 mg·kg⁻¹; Table 8). Similar to the 0.5 months, after 1 month, the M3-P change relative to the initial for ECST did not differ among any fertilizer-P source in the MO-SiL 1 soil, or among CPST, MAP, or TSP in the AR-L, NE-SiL, and NE-SL soils, but was larger than for the UC in all other soils (Table 8). However, soil M3-P change relative to the initial for ECST (62.9 mg·kg⁻¹) after 1 month in the AR-SiL soil was 154 mg·kg⁻¹ smaller than for CPST, while ECST (137 mg·kg⁻¹) was 112 mg·kg⁻¹ larger than for CPST in the MO-SiL 2, but did not differ from MAP and TSP fertilizer-P source (Table 8). Similar to after 1 month, the M3-P change relative to the initial for ECST after 2 months did not differ from that for CPST, MAP, or TSP in the AR-SiL and NE-SL soils, but was consistently larger than for the UC in all soils (Table 8). However, for the AR-L, MO-SiL 2, and NE-SiL soils, the M3-P change relative to the initial for ECST (134, 153, and 170 mg·kg⁻¹, respectively) was at least numerically larger than for

Table 8. Summary of Mehlich-3 phosphorus (M3-P; mg·kg^{-1}) concentration changes relative to the initial M3-P in soils amended with electrochemically precipitated struvite (ECST), chemically precipitated struvite (CPST), monoammonium phosphate (MAP), and triple superphosphate (TSP) fertilizer-phosphorus (P) materials in a six-month, plant-less, moist-soil incubation study.

Soil	Fertilizer-P source	Incubation time (months)				
		0.5	1	2	4	6
AR-L[§]	CPST	46.2 R-k	57.9 N-i*[†]	50.9 Q-j	61.0 M-g*	56.7 N-i*
	ECST	142.5 C-K*	108.1 D-W*	134.4 D-O*	165.6 B-F*	102.8 D-Y*
	MAP	80.3 H-e*	90.3 F-Z*	92.7 D-Y*	121.6 D-R*	118.5 D-S*
	TSP	104.6 D-X*	94.0 D-Y*	100.6 D-Y*	115.1 D-U*	133.6 D-P*
	UC	−27.6 jk	−29.6 k	−26.2 jk	−21.5 jk	−26.6 jk
AR-SiL	CPST	40.9 S-k	216.7 BC*	11.0 b-k	28.7 X-k	53.8 Q-i
	ECST	225.0 B*	62.9 L-g*	68.3 J-g*	134.3 D-O*	67.2 J-g*
	MAP	413.0 A*	49.3 Q-j	51.0 Q-j	47.7 Q-k	53.9 Q-i
	TSP	428.6 A*	48.8 Q-k	73.3 I-g*	90.2 F-a*	70.7 J-g*
	UC	−20.5 h-k	−25.0 jk	−24.8 jk	−20.3 h-k	−26.0 jk
MO-SiL 1	CPST	85.9 G-b*	36.2 V-k	31.0 W-k	122.9 D-R*	59.4 M-g*
	ECST	105.0 D-X*	65.6 K-g*	91.0 D-Y*	163.9 B-G*	90.6 E-Y*
	MAP	61.9 M-g*	69.5 J-g*	71.8 I-g*	68.9 J-g*	79.5 H-e*
	TSP	81.1 H-d*	72.5 I-g*	93.7 D-Y*	100.4 D-Y*	76.0 H-f*
	UC	3.3 d-k	2.8 d-k	0.2 g-k	1.9 e-k	−0.9 g-k
MO-SiL 2	CPST	69.1 J-g*	25.5 Y-k	46.9 R-k	61.2 M-g*	144.6 C-J*
	ECST	169.2 B-E*	137.3 D-M*	153.1 B-H*	117.7 D-S*	124.6 D-R*
	MAP	86.7 G-a*	72.4 I-g*	38.9 T-k	122.7 D-R*	92.3 D-Y*
	TSP	101.5 D-Y*	91.4 D-Y*	100.1 D-Y*	115.2 D-U*	140.7 C-L*
	UC	5.8 c-k	0.5 f-k	49.6 Q-j	11.7 Z-k	3.4 c-k
NE-SiL	CPST	37.3 U-k	118.7 D-S*	55.9 O-i*	86.6 G-a*	87.5 F-a*
	ECST	73.5 I-g*	114.2 D-V*	169.5 B-D*	143.6 C-K*	105.2 D-X*
	MAP	91.0 E-Y*	110.1 D-V*	101.5 D-Y*	110.6 D-V*	109.1 D-W*
	TSP	92.2 D-Y*	97.3 D-Y*	101.9 D-Y*	122.1 D-R*	107.6 D-W*
	UC	0.2 f-k	0.2 f-k	5.6 c-k	6.6 c-k	7.8 b-k
NE-SL	CPST	55.2 P-i	116.8 D-T*	82.0 H-c*	74.8 H-g*	85.6 G-b*
	ECST	152.5 B-H*	126.4 D-Q*	120.3 D-R*	121.3 D-R*	149.8 B-I*
	MAP	104.2 D-X*	114.9 D-U*	112.3 D-V*	122.3 D-R*	106.5 D-X*
	TSP	135.0 D-N*	116.0 D-U*	111.4 D-V*	112.3 D-V*	101.8 D-Y*
	UC	3.8 c-k	7.0 c-k	2.7 d-k	6.3 c-k	4.4 c-k

[§]AR-L = Arkansas loam, AR-SiL = Arkansas silt loam, MO-SiL 1 = Missouri silt loam, MO-SiL 2 = Missouri silt loam, NE-SL = Nebraska sandy loam and NE-SiL = Nebraska silt loam. [†]Means for soil-fertilizer-P source combinations followed by different letters are different at $P < 0.05$. Owing to the large number of similarities among soil-fertilizer-P source combinations, the corresponding letter notations began with a capital letter and continued to a second set of lowercase letters. *An asterisk (*) specifies mean change is different than zero ($P < 0.05$).

all fertilizer-P sources (Table 8). For the MO-SiL 1 soil, the M3-P change relative to the initial for ECST (91.0 mg·kg^{-1}) was larger than for CPST, but similar to that for MAP and TSP (Table 8). Similar to after 2 months, after 4 months, ECST did not differ among CPST, MAP, or TSP in the MO-SiL 2, NE-SiL, and NE-SL soils, but again was larger than for the UC in all soils (Table 8). Additionally, after 4 months, the M3-P change relative to the initial for ECST (166, 134, and 164 mg·kg^{-1}) was at least numerically largest among the fertilizer-P sources in the AR-L, AR-SiL, and MO-SiL 1 soils, respectively, and did not differ from that for TSP (Table 8). Furthermore, the M3-P change relative to the initial for ECST after 4 months was larger than for CPST and MAP in the AR-L, AR-SiL, and MO-SiL 1 soils, except the MO-SiL-1-CPST combination was similar to ECST (Table 8). After 6 months, the M3-P change relative to the initial for ECST did not differ among any fertilizer-P source in the AR-SiL soil and did not differ among CPST, MAP, or TSP in the AR-L, MO-SiL 1, MO-SiL 2, NE-SiL, and NE-SL soils (Table 8). However, the M3-P change relative to the initial for ECST was larger than for the UC in all soils after 6 months (Table 8).

After 0.5 months, the M3-P change relative to the initial between ECST and CPST did not differ in only two of the six soils (*i.e.*, MO-SiL 1 and NE-SiL), and the M3-P change relative to the initial for ECST was 96.3, 184, 100, and 97.3 mg·kg^{-1} larger than for CPST in the AR-L, AR-SiL, MO-SiL 2, and NE-SL soils, respectively (Table 8). In contrast, after 1 month, the M3-P change relative to the initial for ECST only differed from that for CPST in the AR-SiL and MO-SiL 2 soils, where ECST had a 154 and 112 mg·kg^{-1}, respectively, larger M3-P change from the initial than CPST (Table 8). After 2 months, the M3-P change relative to the initial for ECST was 83.5, 60.0, 106, and 114 mg·kg^{-1} larger than for CPST in the AR-L, MO-SiL 1 and 2, and NE-SiL soils, respectively (Table 8). Additionally, the M3-P change relative to the initial did not differ between ECST and CPST in any soil after 4 months, except for the AR-L and AR-SiL, where ECST was 105 and 106 mg·kg^{-1} larger than for CPST (Table 8). However, after 6 months, the M3-P change relative to the initial for ECST did not differ from that for CPST in any of the six soils (Table 8). Mehlich-3-extractable nutrients, which are the result of a weak-acid extraction, are critically important for crop productivity and constitute the available nutrients for plant uptake, hence, it is imperative to also determine the capability of potentially new fertilizer materials to supply M3-extractable nutrients in adequate amounts over time and whether the potentially new fertilizer materials are comparable to other similar, presently commercially available products.

Changes in M3-P concentrations from the initial among soil-fertilizer-P-source-time combinations were generally positive, except for the UC in the AR-L and AR-SiL soils (Table 8). Only the AR-SiL soil had a decreased positive M3-P change from the initial after 0.5 months, but, like all other soils, did not differ over the remainder of the soil incubation period, which was not surprising, as AR-SiL had the largest initial M3-P among all six soils. The increased M3-P

change relative to the initial for ECST was either the largest among soil-fertilizer-P-source-time combinations or among the largest, similar to that for MAP and TSP which were predominantly largest in each soil (Table 8). However, ECST's crystalline form provided a larger surface area compared to pellets to be exposed to the soil environment for greater and faster chemical reactivity may have resulted in the release of larger M3-P concentrations closer to that of MAP and TSP rather than that of CPST. The reduced M3-P increase after 0.5 and 6 months in the AR-SiL soil was likely due to the lowered soil pH facilitating the precipitation and formation of insoluble, secondary P-minerals, P adsorption to clay minerals, and/or microbial P immobilization [31] [32] [33] [55] [56] [57]. Anderson et al. [31] [32] [33] also reported similar results, where M3-P concentrations increased above the initial over time and were particularly larger for finely ground CPST, potentially resulting from the larger surface area, which would be similar to that for ECST in this study.

3.3.5. M3-Ca

Mehlich-3-Ca changes from the initial over the 6-month period ranged from a decrease of -463 mg·kg^{-1} in the AR-SiL-ECST combination after 1 month to an increase of 393 mg·kg^{-1} in the NE-SiL-TSP combination after 6 months (Table 9). However, there was a general steady numeric M3-Ca increase, relative to the initial, among all soil-fertilizer-P source combinations between the 0.5- and 6-month sampling times, except for the AR-L-CPST and -ECST combinations, which was largest at 0.5 months but stabilized over time (Table 9). Only 39 of the 150 soil-fertilizer-P-source-time combinations did not differ from a change of zero relative to the initial (Table 9).

After 0.5 months, the M3-Ca change relative to the initial for ECST did not differ among fertilizer-P sources in any of the six soils, except for TSP, which was larger than for ECST in all soils (Table 9). Similar to after 0.5 months, after 1 month, the M3-Ca change relative to the initial for ECST did not differ among any fertilizer-P source in the MO-SiL 1 soil, or among CPST, MAP, or the UC in any other soil (Table 8). Similar to after 1 month, the M3-Ca change relative to the initial for ECST after 2 months did not differ from that for CPST, MAP, or the UC in any soil, but was smaller than for TSP in all soils, except for the MO-SiL 1, where ECST was similar to that for TSP (Table 9). After 2 months, the M3-Ca change relative to the initial for ECST did not differ from that for CPST, MAP, or the UC in any soil, but was consistently smaller than for TSP (Table 9). However, the M3-Ca change relative to the initial for ECST after 6 months also did not differ among CPST, MAP, or the UC in all soils, except the AR-L (Table 9). The M3-Ca change relative to the initial for ECST after 6 months was consistently smaller than for TSP in all soils, and the M3-Ca change relative to the initial for ECST was also smaller than for MAP in the AR-L and NE-SiL soils and smaller than for the UC in the AR-L soil (Table 9). The M3-Ca change relative to the initial between ECST and CPST did not differ among any

Table 9. Summary of Mehlich-3 calcium (M3-Ca; mg·kg⁻¹) concentration changes relative to the initial M3-Ca in soils amended with electrochemically precipitated struvite (ECST), chemically precipitated struvite (CPST), monoammonium phosphate (MAP), and triple superphosphate (TSP) fertilizer-phosphorus (P) materials in a six-month, plant-less, moist-soil incubation study.

Soil	Fertilizer-P source	Incubation time (months)				
		0.5	1	2	4	6
AR-L§	CPST	−133.5 t-x*†	−154.6 t-x*	−162.2 t-x*	−102.3 o-s*	−114.7 r-u*
	ECST	−163.9 u-x*	−178.9 w-y*	−175.1 v-y*	−62.3 o-r*	−119.6 s-v*
	MAP	−132.7 t-x*	−173.8 v-y*	−107.3 q-u*	−17.8 g-o	−33.6 j-o
	TSP	−53.5 m-q*	−55.2 m-q*	−31.7 j-o	57.2 U-e*	39.0 Y-h
	UC	−122.6 s-w*	−186.0 xy*	−112.7 r-u*	−53.3 m-q*	−46.8 l-p*
AR-SiL	CPST	−432.6 ffgg*	−433.6 ffgg*	−446.8 gg*	−332.7 aa-dd*	−307.4 aabb*
	ECST	−430.5 ffgg*	−462.6 gg*	−437.2 ffgg*	−286.7 zaa*	−352.0 bb-dd*
	MAP	−437.7 ffgg*	−459.1 gg*	−386.1 dd-ff*	−341.2 aa-dd*	−318.8 aa-cc*
	TSP	−147.1 t-x*	−387.8 dd-ff*	−301.3 aabb*	−188.2 xy*	−233.6 yz*
	UC	−421.4 ee-gg*	−459.7 gg*	−372.7 cc-ee*	−318.7 aa-cc*	−345.1 bb-dd*
MO-SiL 1	CPST	27.1 Z-k	42.4 X-g	87.3 O-Z*	169.6 G-M*	149.2 G-O*
	ECST	51.6 V-f*	29.1 Z-j	122.4 J-T*	171.1 G-M*	172.6 G-L*
	MAP	46.1 W-g	49.2 V-f*	112.9 L-V*	200.6 E-H*	175.1 G-L*
	TSP	156.6 G-N*	137.1 I-Q*	162.6 G-N*	302.1 B-D*	272.1 B-D*
	UC	70.6 R-b*	69.4 R-b*	105.0 N-X*	168.6 G-M*	206.0 E-G*
MO-SiL 2	CPST	−17.9 g-o	−28.3 i-o	23.6 Z-l	127.3 I-S*	106.1 M-X*
	ECST	−8.5 f-o	−36.8 j-o	34.7 Z-i	142.8 G-P*	133.3 I-R*
	MAP	−10.8 f-o	−7.1 e-o	76.7 Q-a*	150.8 G-O*	149.8 G-O*
	TSP	56.8 U-e*	25.1 Z-k	146.8 G-P*	246.3 D-F*	274.8 B-D*
	UC	−2.1 c-o	−5.1 d-o	38.4 Y-h	159.3 G-N*	106.3 M-X*
NE-SiL	CPST	−64.0 o-s*	−65.6 o-s*	117.7 L-U*	39.0 Y-h	296.4 B -D*
	ECST	−33.7 j-o	−66.6 o-s*	59.0 T-d*	44.0 X-g	263.8 C-E*
	MAP	61.4 T-c*	−25.6 h-o	82.5 P-a*	123.0 J-S*	325.7 B*
	TSP	35.5 Z-i	11.4 b-l	180.4 G-K*	101.0 N-Y*	393.4 A*
	UC	−31.1 j-o	−39.6 l-p	70.4 R-b*	48.5 V-f*	314.3 BC*
NE-SL	CPST	22.1 a-l	−1.8 c-n	7.8 b-n	133.3 I-R*	128.1 I-R*
	ECST	−18.6 g-o	29.9 Z-i	21.3 a-l	129.3 I-R*	111.4 L-V*
	MAP	−2.7 c-o	37.5 Y-h	43.1 X-g	183.8 F-J*	133.5 I-R*
	TSP	111.0 L-W*	154.2 G-N*	118.4 K-U*	251.3 C-E*	190.5 F-I*
	UC	25.8 Z-k	36.1 Y-i	67.3 S-b*	131.8 I-R*	138.1 H-Q*

§AR-L = Arkansas loam, AR-SiL = Arkansas silt loam, MO-SiL 1 = Missouri silt loam, MO-SiL 2 = Missouri silt loam, NE-SL = Nebraska sandy loam and NE-SiL = Nebraska silt loam. †Means for soil-fertilizer-P source combinations followed by different letters are different at *P* < 0.05. Owing to the large number of similarities among soil-fertilizer-P source combinations, the corresponding letter notations began with a capital letter and continued to a second set of lowercase letters, or doubled lower case letters. *An asterisk (*) specifies mean change is different than zero (*P* < 0.05).

of the six soils at any of the sampling times (Table 9).

The change in M3-Ca started out decreasing from the initial among all soils, except for MO-SiL 1, for which the change from the initial was positive among all fertilizer-P sources, then decreased only in three random soil-fertilizer-P source combinations after 1 month (*i.e.*, AR-L-UC, AR-SiL-TSP, and NE-SiL-MAP). However, the M3-Ca change relative to the initial after 1 month remained negative among the five soils and gradually increased after 2 and 4 months, then mostly remaining constant after 6 months, but reached their largest numeric change between 0.5 and 6 months among all fertilizer-P sources (Table 9). As described previously, the reduction in soil pH occurred at approximately the same time and in the same soils, likely contributing to the increase in M3-Ca, as the lowered soil pH may have facilitated a release of Ca from other compounds in the soil or from cation exchange sites that with weakly bound Ca^{2+}. Anderson *et al.* [31] reported a similar occurrence, where the M3-Ca concentration change, which was also predominantly negative early, gradually increased and was largest after 6 months, with the likely cause attributed to the result of clay fixation and the release of cations from the dissolving fertilizers. Other potential contributory causes, particularly in the current study, could have been the decreased soil pH, which would make the formation of Fe-P complexes more favorable than Ca-P complexes, hence, the M3-Ca concentrations would begin to increase with the pH decrease [58].

3.3.6. M3-Mg

Mehlich-3-Mg changes from the initial over the 6-month period ranged from a decrease of -96.7 mg·kg^{-1} in the AR-SiL-UC combination after 1 month to an increase of 202 mg·kg^{-1} in the MO-SiL-1-ECST combination after 4 months (Table 10). However, there was a generally steady M3-Mg increase relative to the initial among all soil-fertilizer-P source combinations between the 0.5- and 6-month sampling times (Table 10). Eighty-six of the 150 M3-Mg soil-fertilizer-P-source-time combinations did not differ from a change of zero relative to the initial (Table 10).

Between 0.5 and 6 months, the M3-Mg change relative to the initial for ECST was predominantly numerically largest among soil-fertilizer-P sources after all sampling times and was generally larger than for MAP, TSP, and the UC (Table 10). After 0.5 to after 4 months, the M3-P change relative to the initial between ECST and CPST did not differ among several soil-fertilizer-P sources, except for the AR-SiL, MO-SiL 2, and NE-SL soils, where ECST was 177, 92.4, and 87.8 mg·kg^{-1}, respectively, larger than for CPST after 0.5 months (Table 10). Similarly, the M3-Mg change relative to the initial for ECST after 1 month was 111 mg·kg^{-1} larger for CPST in the AR-SiL, but M3-Mg for ECST was also 111 mg·kg^{-1} larger in the MO-SiL 2, but did not differ from that for CPST in any other soil (Table 10). In contrast, after 2 months, the M3-Mg change relative to the initial for ECST only differed from that for CPST in the MO-SiL 1, MO-SiL

Table 10. Summary of Mehlich-3 magnesium (M3-Mg; mg·kg^{-1}) concentration changes relative to the initial M3-Mg in soils amended with electrochemically precipitated struvite (ECST), chemically precipitated struvite (CPST), monoammonium phosphate (MAP), and triple superphosphate (TSP) fertilizer-phosphorus (P) materials in a six-month, plant-less, moist-soil incubation study.

Soil	Fertilizer-P source	Incubation time (months)				
		0.5	1	2	4	6
AR-L§	CPST	44.0 K-a	50.8 J-Y*†	77.8 D-S*	99.9 C-N*	96.5 C-P*
	ECST	111.3 B-K*	96.2 C-P*	125.9 B-H*	162.2 A-C*	97.7 C-N*
	MAP	−13.7 X-l	−17.5 Z-m	1.4 U-f	14.0 R-e	6.6 T-e
	TSP	−13.2 X-l	−13.7 X-l	−11.3 X-j	1.2 U-f	−8.0 W-i
	UC	−19.9 a-m	−28.6 c-o	−17.1 Y-m	−11.5 X-j	−12.8 X-k
AR-SiL	CPST	−41.0 d-o	103.6 B-M*	−47.3 e-o	−27.8 b-n	42.6 K-a
	ECST	135.6 A-F*	−7.0 V-h	13.2 R-e	74.0 E-T*	31.1 N-c
	MAP	−65.9 f-o*	−81.8 k-o*	−76.0 i-o*	−76.0 i-o*	−65.8 f-o*
	TSP	−85.0 m-o*	−90.6 no*	−84.4 m-o*	−69.3 g-o*	−71.0 h-o*
	UC	−93.9 no*	−96.7 o*	−82.1 l-o*	−81.5 k-o*	−78.7 j-o*
MO-SiL 1	CPST	68.7 F-U*	31.2 N-c	45.3 K-a	126.9 B-H*	105.4 B-L*
	ECST	117.6 B-J*	97.0 C-O*	135.7 A-F*	201.9 A*	172.2 AB*
	MAP	17.9 Q-e	18.1 Q-e	38.8 L-c	49.4 K-Z*	50.4 J-Z*
	TSP	27.4 P-d	24.1 Q-d	23.0 Q-d	44.5 K-a	49.2 K-a*
	UC	13.9 R-e	11.9 S-e	21.7 Q-e	31.9 N-c	46.6 K-a
MO-SiL 2	CPST	52.0 J-Y*	20.7 Q-e	37.7 L-c	60.0 H-W*	139.8 A-E*
	ECST	144.4 A-D*	130.2 B-F*	158.5 A-C*	122.0 B-I*	154.6 A-C*
	MAP	2.0 U-f	5.0 T-e	10.6 S-e	17.8 Q-e	14.2 R-e
	TSP	1.3 U-f	-0.9 V-g	11.5 S-e	23.5 Q-d	21.6 Q-e
	UC	−2.2 V-h	0.4 U-f	1.4 U-f	13.5 R-e	7.4 T-e
NE-SiL	CPST	18.5 Q-e	81.9 D-R*	54.3 I-X*	68.8 F-U*	96.0 C-P*
	ECST	77.1 D-S*	104.1 B-L*	160.9 A-C*	162.5 A-C*	134.7 A-F*
	MAP	23.1 Q-d	18.0 Q-e	22.6 Q-d	33.0 N-c	43.9 K-a
	TSP	12.4 S-e	16.6 Q-e	20.8 Q-e	18.4 Q-e	36.1 M-c
	UC	4.7 U-e	1.9 U-f	8.6 T-e	20.5 Q-e	28.3 O-c
NE-SL	CPST	41 L-b	84.8 D-Q*	61.4 G-V*	82.0 D-R*	96.7 C-O*
	ECST	128.8 B-H*	122.7 B-I*†	118.9 B-J*	145.8 A-D*	163.3 A-C*
	MAP	5.0 T-e	11.3 S-e	7.5 T-e	28.2 O-c	20.7 Q-e
	TSP	11.7 S-e	18.8 Q-e	11.1 S-e	22.4 Q-d	16.8 Q-e
	UC	5.3 T-e	5.5 T-e	8.7 S-e	14.6 R-e	11.8 S-e

§AR-L = Arkansas loam, AR-SiL = Arkansas silt loam, MO-SiL 1 = Missouri silt loam, MO-SiL 2 = Missouri silt loam, NE-SL = Nebraska sandy loam and NE-SiL = Nebraska silt loam. †Means for soil-fertilizer-P source combinations followed by different letters are different at $P < 0.05$. Owing to the large number of similarities among soil-fertilizer-P source combinations, the corresponding letter notations began with a capital letter and continued to a second set of lowercase letters. *An asterisk (*) specifies mean change is different than zero ($P < 0.05$).

2, and NE-SiL soils, where ECST was 90.4, 121, and 107 mg·kg⁻¹, respectively, larger than for CPST (Table 10). In addition, after 4 months, the M3-Mg change relative to the initial for ECST was similar to that for CPST in the AR-L and NE-SL soils, but ECST had 102, 75.0, 62.0, and 93.7 mg·kg⁻¹, respectively, larger M3-Mg change relative to the initial than for CPST in the AR-SiL, MO-SiL 1, MO-SiL 2, and NE-SiL soils (Table 10). After 6 months, the M3-Mg change relative to the initial for ECST did not differ from that for CPST in any soil (Table 10).

Unlike for M3-Ca, the change in M3-Mg concentrations relative to the initial among fertilizer-P sources predominantly increased in all soils, except for in the AR-L and AR-SiL soils, which decreased (Table 10). Although all fertilizer-P sources, except for ECST and CPST, started with negative M3-Mg changes in the AR-L and AR-SiL soils by after 0.5 months, CPST and AR-L-MAP increased above the initial after 2 months (Table 10). However, among fertilizer-P sources within soils or averaged across time or among fertilizers within time averaged across soils, ECST had the statistically and/or numerically largest M3-Mg change relative to the initial in all soil-fertilizer-P-source-time combinations, except for the AR-SiL after 1 month (Table 10). Furthermore, between the 0.5 and 6-month sampling times, the M3-Mg change relative to the initial predominantly remained constant (Table 10). In addition, between 0.5 and 4 months, ECST's M3-Mg change relative to the initial was statistically and/or numerically larger than for CPST in all soils, but, after 6 months, there were no differences between the two struvite materials (Table 10).

As previously described, since ECST was applied in a crystalline form, greater dissolution and opportunity for chemical reactions likely occurred from larger surface area compared to pellets, and since Mg is a component of ECST, 23.8 mg·kg⁻¹; which had the largest M3-Mg concentration, may have contributed to the consistently larger M3-Mg concentrations in the ECST-amended soils over time. Additionally, the larger Mg concentrations of ECST could be beneficial in soils with Mg deficiencies or for crops that require large concentrations of Mg [7]. Similarly, since CPST was in a pelleted form, its dissolution was likely slower than ECST, but M3-Mg dissolution for both caught up after 6 months. Anderson *et al.* [31] reported M3-Mg for both struvites did not differ after 6 months, likely due to the attainment of similar dissolution rates.

3.3.7. M3-Fe

Unlike for the other measured parameters, the M3-Fe changes from the initial had many differences among soil-fertilizer-P source combinations over time during the 6-month incubation period, hence, means separation for M3-Fe failed (Table 11). The M3-Fe change relative to the initial for ECST ranged from a decrease of −63.5 mg·kg⁻¹ the MO-SiL 2-UC combination after 0.5 months to an increase of 19.4 mg·kg⁻¹ in the AR-SiL-MAP combination after 0.5 months (Table 11). However, there was a general numeric increase in M3-Fe among

Table 11. Summary of Mehlich-3 iron (M3-Fe; mg·kg⁻¹) concentration changes relative to the initial M3-Fe in soils amended with electrochemically precipitated struvite (ECST), chemically precipitated struvite (CPST), monoammonium phosphate (MAP), and triple superphosphate (TSP) fertilizer-phosphorus (P) materials in a six-month, plant-less, moist-soil incubation study.

Soil	Fertilizer-P source	Incubation time (months)				
		0.5	1	2	4	6
AR-L§	CPST	−46.1*†	−37.2*	−40.4*	−18.5*	−22.4*
	ECST	−37.5*	−34.6*	−37.1*	−12.5*	−24.9*
	MAP	−46.1*	−37.2*	−40.4*	−18.5*	−22.4*
	TSP	−28.3*	−31.8*	−26.5*	−6.6	−9.8*
	UC	−54.0*	−48.9*	−43.1*	−27.1*	−32.3*
AR-SiL	CPST	−47.3*	−51.0*	−50.7*	−8.1*	−7.1*
	ECST	−42.1*	−51.8*	−42.7*	4.5	−11.6*
	MAP	19.4*	−46.4*	−28.5*	9.4*	3.8
	TSP	9.2*	−44.6*	−34.4*	13.2*	−1.7
	UC	−50.8*	−58.8*	−51.0*	−15.3*	−25.5*
MO-SiL 1	CPST	−33.5*	−26.9*	−29.7*	−6.1*	−12*
	ECST	−30.2*	−27.0*	−21.6*	0.0	−2.2
	MAP	−31.4*	−19.5*	−16.3*	3.2	6.1
	TSP	−23.0*	−20.6*	−19.0*	8.1*	−3.3
	UC	−33.2*	−26.5*	−26.8*	−10.4*	−15.0*
MO-SiL 2	CPST	−60.8*	−53.0*	−46.7*	−13.7*	−22.0*
	ECST	−54.2*	−46.9*	−44.3*	−4.4	−15.7*
	MAP	−53.1*	−48.7*	−45.8*	0.4	−6.2
	TSP	−49.9*	−48.3*	−40.2*	−8.1*	−5.9
	UC	−63.5*	−56.8*	−45.6*	−19.3*	−28.0*
NE-SiL	CPST	−47.5*	−40.1*	−35.4*	−9.5*	−9.9*
	ECST	−43.6*	−38.8*	−32.9*	−8.4*	−10.8*
	MAP	−41.6*	−34.9*	−32.0*	−3.3	−4.9
	TSP	−41.2*	−33.5*	−26.2*	−5	−4.7
	UC	−48.2*	−42.2*	−37.9*	−11.6*	−14.4*
NE-SL	CPST	−34.7*†	−21.9*	−32.0*	−8.3*	−5.4
	ECST	−30.7*	−22.9*	−26.1*	−0.5	2.2
	MAP	−29.5*	−22.1*	−23.3*	6.8	3.3
	TSP	−21.6*	−20.3*	−25.7*	0.2	−1.8
	UC	−33.4*	−24.8*	−28.1*	−11.1*	−11.9*

§AR-L = Arkansas loam, AR-SiL = Arkansas silt loam, MO-SiL 1 = Missouri silt loam, MO-SiL 2 = Missouri silt loam, NE-SL = Nebraska sandy loam and NE-SiL = Nebraska silt loam. *Means for soil-fertilizer-P source combinations followed by an asterisk (*) specifies a mean change that is significantly different than zero at $P < 0.05$. †Owing to the large number of similarities among soil-fertilizer-P source combinations, the corresponding letter notations could not be produced.

soil-fertilizer-P source combinations between 0.5 and 6 months (Table 11). Only 27 of the 150 soil-fertilizer-P-source-time combinations did not differ from a change of zero relative to the initial (Table 11).

The M3-Fe changes relative to the initial were predominantly negative and significantly lower than a mean change of zero across all sampling times between 0.5 and 2 months, except for AR-SiL-MAP and -TSP after 0.5 months (Table 11). However, the M3-Fe change relative to the initial among the soil-fertilizer-P source combinations also decreased after 4 months, where most changes were smaller than a mean change of zero, except for AR-SiL-MAP and -TSP and MO-SiL-1-TSP, which had a larger change than zero, and MO-SiL-1-ESCT and -MAP, MO-SiL-2- and NE-SL-MAP and -TSP, which did not differ from a change of zero (Table 11). The M3-Fe change relative to the initial among the soil-fertilizer-P source combinations also decreased after 6 months, with most differing from a mean change of zero, except for AR-SiL- and MO-SiL-1-MAP and NE-SL-ECST and -MAP, which increased, but did not differ from a mean change of zero (Table 11).

The general decreased change relative to the initial in M3-Fe along with the generally decreased change in M3-P over time makes it likely that both elements were co-precipitated, forming less-soluble compounds that led to the decrease in M3-P over time. Additionally, the presence of holes in the soil cup's lids allowed for aeration, which may have caused some of the Fe in the soil to exist as insoluble Fe^{3+} compounds or become fixed to clays or organic matter [59]. Negative changes from the initial M3-Fe levels in soil-fertilizer-time combinations may also be attributed to Fe-phosphate co-precipitation [31] [33].

3.4. Implications

Initially perceived as a troublesome byproduct of WWTPs due to its tendency to obstruct pipes and foul pumps [4] [6] [60], over the past two decades, struvite has gained attention as a potentially environmentally sustainable solution based on struvite's potential as a promising alternative fertilizer-P source [28] [29] [30] [61]. The systematic recovery of struvite presents an opportunity to recycle P from various waste sources across different sectors that produce large quantities of waste, particularly agriculture [19] [22] [62], municipal wastewater systems [63] [64], dairy wastewater [65] [66], lagoon wastewater [22], waste and sewage sludge and various other industries [6] [67] [68].

Although struvite has been reported to have slow-release properties because of struvite's low solubility in water and greater solubility with increasing acidity [19], results of this study indicated that ECST exhibits fertilizer-P behavior that is comparable to commercially available CPST and other commonly used fertilizer-P sources, such as MAP and TSP, in a range of soil textures. Consequently, struvite can be used directly as fertilizer-P source for agricultural production among numerous crops [28] [29] [30].

Recovering P through electrochemical precipitation can help reduce the cur-

rent national, and eventually global, reliance on conventional-P fertilizers produced from non-renewable, mined rock phosphate. Electrochemical struvite recovery can also preserve water quality by reducing the risk of eutrophication in freshwater sources, preserving aquatic life and livelihood, and improving ecosystem functioning [4] [7] [57] [69] [70]. Furthermore, struvite-P recovery can aid in averting potential geopolitical crises when mineable proportions of phosphate rocks become scarce in the few countries that have economically important quantities of rock phosphate [14] [71] [72]. The effectiveness of struvite as a renewable fertilizer-P source holds the potential to ensure a sustainable supply of P to support and drive global agricultural productivity, thereby narrowing or closing the inefficient P loop (*i.e.*, creating localized P cycles) and strengthening and safeguarding food security for future generations [2] [3] [11] [13] [19]. Reclaiming struvite from wastewater as a fertilizer-P source by electrochemical means also simultaneously reduces N and P loading in surface waters and reduces production and/or operation costs [4] [7] [73] [74]. Moreover, struvite precipitation by electrochemical technology produces hydrogen as a by-product, which can be valuable in industrial operations and further enhance cost savings and causing the technology to be more attractive as an incentive for industrial application [7].

Although there are many advantages to electrochemical struvite recovery, more in-depth feasibility studies and economic analyses are required to determine the potential successfulness of implementing the technology on large scales, particularly in animal agriculture. At the present, due to still being in the experimental phase of development, large-scale production of ECST is expensive and not yet economical [28] [29] [30]. Further studies are needed to determine the long-term impacts of struvite use on soil pH and nutrient behaviors, such as the effects of increased soil Mg concentrations and struvite's effects on the soil microbial community, runoff potential, and greenhouse gas emissions.

4. Conclusions

Various small-scale, plant-response studies have previously been conducted and have shown the potential for using recovered struvite as a fertilizer-P source for agronomic production. However, there is still limited research on the soil behavior of wastewater-reclaimed struvite-P materials and their interactions in different textured agricultural soils, particularly without the influence of plants. This study aimed to evaluate the dissolution behavior over time of an innovative ECST material compared to that of other commonly used, conventional fertilizer-P sources (*i.e.*, MAP and TSP) and a CPST material in a plant-less soil incubation in various agricultural soil textures.

The hypothesis that soils amended with CPST and ECST will have greater pH and larger NO_3-N and NH_4-N concentrations over time compared to soils amended with TSP, but similar to that for MAP, was partially supported. Generally, soil pH decreased over time among all fertilizer-P sources within soils, par-

ticularly after 2 to 6 months. However, in most instances, ECST, TSP, and UC had larger pH than MAP and CPST. For soil NO_3-N, concentrations increased from the initial among all soil-fertilizer-P sources over time. After 1, 2, and 6 months, ECST had either larger or similar NO_3-N change to one or more of the other fertilizer-P sources, but was predominantly larger than for TSP and the UC over time. The hypothesis that the particle size of the applied struvite material (*i.e.*, pellets for CPST vs. crystals for ECST) allowed for greater and quicker dissolution of ECST in the various soil textures, but larger or similar to that of CPST over time, was generally supported, as, in most instances and across multiple parameters, ECST had similar or at least numerically larger nutrient concentration changes than for CPST, which could be attributed to the differences in applied form as observed in other studies. The hypothesis that ECST would have comparable M3-P, -Ca, and -Fe concentrations to that of CPST, MAP, and TSP over time was supported. Since ECST was applied in a crystalline form, it was expected that soils amended with ECST would have a larger M3-P concentration changes than the other fertilizer-P sources. However, while ECST did not always have the statistically largest M3-P change, ECST had similar or numerically larger M3-P compared to all other fertilizer-P sources, except for the UC, where the change for ECST was almost always larger. The hypothesis that ECST would have comparable M3-P, -Ca, and -Fe concentration changes to that of CPST, MAP, and TSP over time, was generally supported. Mehlich-3-P decreased after 0.5 months in five of the six soils and remained constant then increased in four of the six soils after 2 months. In most instances, ECST had mostly similar M3-Ca to that of the other fertilizer-P sources, but numerically less than that for MAP and TSP. Mehlich-3-Fe declined among fertilizer-P sources within soils over time; however, M3-Fe for ECST was generally similar to that of the other soil-fertilizer-P-source-time combinations.

Overall, results of this study confirmed what had been previously reported that ECST is a viable, alternative fertilizer-P material, with similar behavior as other fertilizer-P sources, that can be applied across soils of various textures. Results also indicated that ECST could provide similar agronomic benefits for crops and is suitable for use in soils with slightly acidic pH. Electrochemically precipitated struvite can also help to alleviate mild Mg deficiencies and does not appear to further facilitate any greater nutrient loss to the environment than CPST, MAP, and TSP.

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Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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