

# Electrochemically Precipitated Struvite Effects on Extractable Soil Nutrients in Multiple Soil Textures

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## Abstract

Struvite recovery from wastewater streams may provide an alternative to traditional fertilizer-phosphorus (P) sources, while also providing the benefit of reducing nitrogen and P loads in wastewater effluent and reducing effects of eutrophication in surface waters. The objective of this study was to assess the effects of fertilizer-P source (*i.e.*, synthetically produced electrochemically precipitated struvite [ECST], chemically precipitated struvite [CPST], monoammonium phosphate [MAP], diammonium phosphate [DAP], triple superphosphate [TSP], rock phosphate [RP], and an unamended control [UC]) on water-soluble (WS) and Mehlich-3 (M3)-extractable soil nutrients (*i.e.*, K, S, Na, Mn, Zn, Cu, and B) periodically over a 9-month period (*i.e.*, 0.5, 1, 2, 4, 6, and 9 months) in multiple soil textures. All WS and M3 nutrients were affected by a combination of soil, fertilizer treatment and/or sampling time. Both WS- and M3-S concentrations increased from the initial for all fertilizer treatments, but the increase was the greatest with DAP (26.3 mg·kg<sup>-1</sup> and 23.4 mg·kg<sup>-1</sup>, respectively), which differed from MAP and TSP that had increases that were intermediate and were greater than for ECST, CPST, and the UC. Mehlich-3-Na concentrations increased from the initial after 0.5 months before decreasing from the initial after 1 month and steadily increasing until 9 months, at which point M3-Na concentrations had increased the most from the initial (12.26 mg·kg<sup>-1</sup>). Changes in WS and M3 nutrients were often similar among ECST, CPST, MAP, DAP, TSP, and RP, supporting the potential for struvite to be an effective alternative fertilizer-P source.

## Keywords

Phosphorus Fertilizers, Struvite, Electrochemical Precipitation, Extractable Soil Nutrients

## 1. Introduction

Phosphorus (P) fertilizers have become vital for sustaining food production to meet the demands of a growing global population. The world's food demands cannot be met without the use of such fertilizers [1]. At the same time, worldwide minable P reserves are dwindling, and rock phosphate (RP) resources, from which most of the world's P is mined, may be exhausted in as little as 100 years [1]. In the United States alone, consumption of P fertilizers increased from an average of 5.8 million metric tons per years from 1960 to 2007, to over 8.5 million metric tons in 2007 [2]. The combined effects of the growing global population and increasing food demands and dwindling mineable P reserves led Cordell *et al.* [1] to emphasize the need to identify and make use of alternative fertilizer-P sources. One such alternative fertilizer-P source is the utilization of P-recovery technologies with nutrient-rich wastewaters, which has the added benefit of reducing eutrophication of surface waters.

Nutrients recovered from wastewater via precipitation reactions can be used as fertilizers to be applied to soils to enhance plant growth. One such P-recovery technique is struvite precipitation. Struvite is a mineral that contains magnesium (Mg), ammonium, and phosphate and can be created from wastewater [3]. Struvite precipitation from wastewaters has shown promising P-capturing potential [3]. It has been shown that struvite precipitation can be induced by the addition of Mg-containing salts to both synthetic and municipal and agricultural wastewaters in a process known as chemical precipitation [4]. The resulting struvite material is referred to as chemically precipitated struvite (CPST). A more recent struvite recovery process that has been developed and tested is electrochemical precipitation. Electrochemically precipitated struvite (ECST) is produced using an electrical current and a sacrificial Mg anode that provides the Mg for struvite crystallization in nutrient-rich water sources [5]. Both CPST and ECST can be used as a fertilizer-P material. Chemically precipitated struvite is already in use on a large scale, but requires the addition of Mg salts to drive the reaction, whereas ECST does not require the input of Mg salts to drive the reaction, but is currently still in experimental phases of small-scale implementation.

The elemental makeup of wastewater-sourced struvite has been shown to vary depending on the source. Struvite sourced from palm (*Areca* spp.)-oil-mill effluent resulted in struvite containing 24.8% P, 21.3% Mg, 3.5% H, 2.0% N, 46.2% O by weight [6]. Struvite precipitates at pH between 7.0 and 10.5, but struvite has been shown to precipitate in the largest quantities at pH ~10 [7]. Struvite has low solubility in water, with a solubility of 169.2 mg·L<sup>-1</sup> in deionized water at 25°C [8]. Precipitating struvite from real wastewater has been shown to introduce other elements to the mineral. Struvite produced from palm-oil-mill effluent was shown to contain 1.0% Ca, 1.2% K, 2.3% C, and 0.1% S [6].

Struvite can be applied as an environmentally friendly, alternative, effective, fertilizer-P source in pot trials, but extensive field trials need to be conducted [3].

Previous studies have been conducted comparing the effects of ECST on soil properties such as water-soluble (WS-) and Mehlich-3-extractable (M3-) nutrient concentrations and soil pH and have all suggested that ECST is a viable alternative fertilizer-P source [9]-[13]. However, little research has investigated the effects of ECST on soil micronutrients.

Certain nutrients are present in relatively small concentrations in soils. For example, boron (B), copper (Cu), manganese (Mn), and zinc (Zn) are important plant-essential micronutrients [14]. However, while some micronutrients are essential for plant growth, others can become toxic in excess [15] [16]. Micronutrients often exist as impurities in mineral-P fertilizers and thus may be unknowingly added to soils when fertilizers are applied [16]. Micronutrient concentrations in fertilizers may vary greatly depending on the nutrient being considered, fertilizer type, and fertilizer origin [16].

Struvite has been shown to be an effective fertilizer, comparable to traditional RP-sourced fertilizers in nutrient concentrations in soils over time [10]-[14] [17]-[23]. However, struvite's potential effects on micronutrients once soil applied need to be studied. Therefore, the objective of this study was to assess the effects of fertilizer-P source (*i.e.*, synthetically produced ECST, CPST, monoammonium phosphate [MAP], diammonium phosphate [DAP], triple superphosphate [TSP], RP, and an unamended control [UC]) on WS and M3-extractable soil nutrients (*i.e.*, K, S, Na, Mn, Zn, Cu, and B) periodically over a 9-month period (*i.e.*, 0.5, 1, 2, 4, 6, and 9 months) in multiple soil textures (*i.e.*, loam, silty clay loam, and silt loam). It was hypothesized that ECST would have comparable WS and M3-extractable nutrient concentrations (*i.e.*, K, S, Na, Mn, Zn, Cu, and B) to CPST, while having smaller nutrient concentrations than MAP, DAP, and RP. It was also hypothesized that the SiL 1 and SiL 2 soils would exhibit similar changes in nutrient concentrations, differing from those of the SiCL and the loam soils. In addition, it was hypothesized that, in general, nutrient concentrations would increase over time among all treatment combinations.

## 2. Materials and Methods

The current study was an extension of Anderson *et al.* [12] who evaluated the effects of ECST and CPST compared to various other commonly used, commercially available fertilizer-P sources (*i.e.*, MAP, DAP, TSP, and RP) in multiple soils (*i.e.*, a loam, silty clay loam, and two silt loams) on soil pH and WS and M3-extractable, P, Ca, Mg, and Fe over time (*i.e.*, 0.5, 1, 2, 4, 6, and 9 months). All materials and procedures used in the study were previously described in Anderson *et al.* [12]. The current study reports on the effects of ECST and CPST compared to MAP, DAP, TSP, RP, and a UC in a loam, silty-clay-loam, and two silt-loam soils on WS and M3-extractable soil K, S, Na, Mn, Zn, Cu, and B, which have not previously been reported, after 0.5, 1, 2, 4, 6, and 9 months of soil application in a plant-less, soil incubation experiment. Materials and procedures are summarized below, as originally described in Anderson *et al.* [12].

**Table 1.** Summary of initial chemical properties of fertilizer-phosphorus (P) sources [*i.e.*, chemically precipitated struvite (CPST), electrochemically precipitated struvite (ECST), rock phosphate (RP), monoammonium phosphate (MAP), diammonium phosphate (DAP), and triple superphosphate (TSP)] used in the incubation experiment (adapted from Anderson *et al.* [12]).

Fertilizer property	Fertilizer-P source					
	CPST	ECST	RP	MAP	DAP	TSP
pH	8.78	–	6.67	4.37	7.32	2.42
Electrical conductivity (dS·m <sup>-1</sup> )	226	–	514	84.6	105	32.8
Water-soluble (mg·kg <sup>-1</sup> )						
P	216	124,050	70.6	196,000	163,300	178,840
Ca	11.6	12.7	148	2252	153	121,296
Mg	157	24,144	25.5	7784	79.9	5791
Fe	1.22	5.80	4.20	68.8	63.6	473
K	1.6	22.0	28.0	1048	1173	808
S	24.5	3.4	113	13,280	13,540	4391
Na	13.0	52.4	25.0	1169	2659	4543
Mn	0.2	4.8	0.2	42.0	1.5	28.8
Zn	0.3	0.9	0.3	4.9	30.8	360
Cu	0.3	0.2	<0.1	0.1	6.5	26.2
B	<0.1	2.5	0.1	15.4	21.6	26.8
Mehlich-3 extractable (mg·kg <sup>-1</sup> )						
P	24,479	158,798	638	181,919	164,349	171,493
Ca	83	2.20	3602	1931	228	105,735
Mg	21,444	27,197	338	6767	507	4715
Fe	127	19.9	226	254	146	362
K	230	16.2	139	1081	1244	786
S	14.6	2.4	112	16,067	17,780	8818
Na	20.4	38.0	19	725	1661	3108
Mn	110	9.4	16.1	72.7	26.4	17.3
Zn	0.35	1.1	4.8	40.3	132	300
Cu	0.6	0.4	1.6	0.3	11.4	25.0
B	1.4	1.6	0.2	15.2	17.8	21.1
Total N (g·g <sup>-1</sup> )	0.057	0.093	0.0004	0.107	0.181	0.0002

**Table 2.** Summary of initial physical and chemical properties of the soils used in the incubation experiment (adapted from Anderson *et al.* [12]).

Soil property	Soil			
	Loam	Silty clay loam	Silt loam 1	Silt loam 2
pH	6.17	6.50	6.53	6.70
Electrical conductivity (dS·m <sup>-1</sup> )	0.107	0.273	0.169	0.164
Water-soluble (mg·kg <sup>-1</sup> )				
P	11.9	9.6	5.5	3.7
Ca	34.0	74.4	62.8	62.0
Mg	21.5	28.1	23.4	17.5
Fe	49.5	40.8	41.4	59.9
K	44.7	44.7	25.4	28.4
S	4.9	12.0	11.1	9.0
Na	4.1	8.7	10.8	24.3
Mn	0.6	0.4	0.8	0.5
Zn	0.3	0.4	0.4	0.5
Cu	0.1	0.1	0.1	0.1
B	0.2	0.3	0.2	0.2
Mehlich-3-extractable (mg·kg <sup>-1</sup> )				
P	93.3	143	33.7	19.5
Ca	933	4,328	1,842	2,156
Mg	194	774	444	365
Fe	201	175	186	460
K	145	485	143	158
S	5.7	16.4	11.2	8.3
Na	10.0	21.9	24.1	42.3
Mn	32.9	59.0	89.9	84.3
Zn	2.4	16.2	3.0	2.5
Cu	1.1	1.1	1.6	1.4
B	2.3	3.1	2.5	2.9
Sand (g·g <sup>-1</sup> )	0.44	0.07	0.12	0.10
Clay (g·g <sup>-1</sup> )	0.09	0.37	0.14	0.11
Silt (g·g <sup>-1</sup> )	0.46	0.56	0.75	0.79
Soil organic matter (g·g <sup>-1</sup> )	0.007	0.025	0.024	0.019
Total C (g·g <sup>-1</sup> )	0.003	0.012	0.011	0.009
Total N (g·g <sup>-1</sup> )	0.0003	0.0011	0.0011	0.0008
C:N ratio	10.5	11.4	9.68	11.0
NO <sub>3</sub> -N (mg·kg <sup>-1</sup> )	9.50	6.30	15.8	12.2
NH <sub>4</sub> -N (mg·kg <sup>-1</sup> )	3.90	6.30	8.20	6.40

## 2.1. Fertilizer-P Sources

As described by Anderson *et al.* [12], the current study used two struvite sources. One was a commercially available, CPST material (*i.e.*, trade name Crystal Green, Ostara Nutrient Recovery Technologies, Inc., Vancouver, Canada), which was produced from a raw, municipal wastewater source from near Atlanta, GA. The second was an ECST material produced by researchers in the Department of Chemical Engineering at the University of Arkansas from synthetic wastewater [24]. The ECST material was electrochemically precipitated from a synthetic solution of known N and P concentrations with Mg supplied from a Mg anode as an electrical current was applied to the solution [24]. Along with the two struvite sources, four commercially available P-fertilizers were used in this study, MAP, DAP, TSP, and RP, along with a UC treatment. The CPST, MAP, DAP, and TSP materials were in pellet form, the RP was in powder form, and the ECST was in crystalline-flake form. **Table 1** summarizes properties of the various fertilizer-P sources.

## 2.2. Soil Collection and Processing

As described by Anderson *et al.* [12], four soils were collected from the top 10 - 15 cm from historic agricultural management throughout Arkansas for use in the current study. A Dardanelle silty clay loam (SiCL; fine-silty, mixed, superactive, thermic Typic Argiudolls) and a Roxana loam (L; coarse-silty, mixed, superactive, nonacid, thermic Typic Udifluvents) were collected at the University of Arkansas Division of Agriculture's Vegetable Research Station in Kibler, AR [25]. A Callo-way silt loam (SiL 1; fine-silty, mixed, active, thermic Aquic Fraglossudalfs) was collected at the Cotton Branch Experiment Station near Marianna, AR. A Henry silt loam (SiL 2; coarse-silty, mixed, active, thermic Typic Fragiaqualfs) was collected at the Pine Tree Branch Experiment Station near Colt, AR [25]. All soils were sieved field-moist to pass through a 7-mm mesh screen and air-dried for two weeks in a greenhouse at ~21 °C with periodic manual mixing to achieve uniform drying. Additional details of the soils were described in Anderson *et al.* [12]. **Table 2** summarizes initial properties among the four soils.

## 2.3. Soil Incubation Experiment

As described by Anderson *et al.* [12], moist-soil incubation was conducted for 9 months between 6 December, 2018 and 15 August, 2019. Approximately 150 g of air-dry soil were added to small plastic soil cups, 10.5-cm diameter by 4.5-cm tall. Two replicate soil cups were used for each soil-fertilizer combination for each of six sampling intervals: 0.5, 1, 2, 4, 6, and 9 months. Each soil cup replicate in each soil-fertilizer combination had one of seven fertilizer treatments applied to the cup, including a UC treatment that received no fertilizer addition. A total of 336 soil cups were prepared.

Based on the initial M3-extractable soil-test P concentrations, fertilizer-P sources were applied at a single rate recommended for row-crop production in

Arkansas, which was determined to be 56 kg P<sub>2</sub>O<sub>5</sub> ha<sup>-1</sup> (24.5 kg·P·ha<sup>-1</sup>). After fertilizer was added to soil cups containing air-dry soil, each soil cup was manually shaken for 10 seconds to mix the fertilizer material into the soil. After mixing, soil cups were randomly placed on wooden shelving units located in a laboratory with lids with multiple holes in them placed loosely on top of each soil cup. Soils cups were rotated among shelves every two weeks during the incubation period. The average room air temperature and relative humidity throughout the 9-mo incubation were 21.6°C and 56.5%.

Soil cups were watered periodically using identical methods as described in Anderson *et al.* [9], in which a target gravimetric water content of 0.20 g·g<sup>-1</sup> was used to determine the target mass for each soil after watering. Soil cups were initially watered on the day of fertilizer application and were gravimetrically rewatered to the same target mass every two weeks using tap water.

Over the 9-month period, soil cups were destructively sampled at the six sampling periods (*i.e.*, 0.5, 1, 2, 4, 6, and 9 mo). When sampled, soil was removed from the cups, oven dried at 70°C for at least 48 hours, mechanically crushed, and sieved through a 2-mm mesh screen. Soil subsamples were analyzed for WS and M3-extractable K, S, Na, Mn, Zn, Cu, and B. To determine WS concentrations, a 1:10 soil mass/water volume ratio was used. Mixtures were stirred for 1 hr before being filtered through a 0.45-µm filter and analyzed using inductively coupled, argon-plasma spectrometry (ICAPS; Spectro Arcos ICP, Spectro Analytical Instruments) [26]. To determine M3-extractable concentrations, a M3-extraction solution, 0.2 M acetic acid, 0.25 M ammonium nitrate, 0.015 M ammonium fluoride, 0.013 M nitric acid, and 0.001 M ethylenediaminetetraacetic acid [26], with a 1:10 soil mass/extractant volume ratio was used before being analyzed by ICAPS [27].

To determine changes in nutrient concentrations from their initial levels, each nutrient's initial mean concentration in the soil was subtracted on a cup-by-cup basis from that measured at each sampling period. More details of the incubation experiment are described in Anderson *et al.* [12].

#### 2.4. Data Analyses

Similar to Anderson *et al.* [12], based on a split-split-plot, complete factorial, completely randomized design, a three-factor analysis of variance (ANOVA) was conducted to evaluate the effects of soil (*i.e.*, L, SiCL, SiL 1, and SiL 2), fertilizer treatment (*i.e.*, MAP, DAP, TSP, RP, CPST, ECST, and UC), sampling time (*i.e.*, 0.5, 1, 2, 4, 6, and 9 mo), and their interactions on the change in WS and M3-extractable soil K, S, Na, Mn, Zn, Cu, and B from their initial values. The whole-plot factor was soil, the split-plot factor was fertilizer treatment, and split-split-plot factor was sampling time. Soil, fertilizer treatment, and sampling time were fixed effects. Replication was considered as a random effect. All data was analyzed according to a normal distribution. Significance was judged at  $P \leq 0.05$ . When appropriate, means were separated by least significant difference at the 0.05 level.

### 3. Results and Discussion

#### 3.1. Water-Soluble Nutrients

The changes in all WS nutrient concentrations compared to the initial value were affected by soil, fertilizer treatment, and/or sampling time (Table 3). The change in WS-S concentration from the initial differed ( $P < 0.01$ ) among fertilizer treatments (Table 3). The change in WS-K, -Mn, and -B concentrations from the initial differed ( $P < 0.01$ ) among fertilizer treatments within soils (Table 3). The change in WS-K, -S, -Na, -Mn, -Cu, and -B concentrations from the initial differed ( $P < 0.01$ ) among soils over time (Table 3). The change in WS-Mn and -Cu concentrations from the initial differed ( $P \leq 0.04$ ) among fertilizer treatments over time (Table 3). The change in WS-Zn concentration from the initial differed ( $P = 0.02$ ) among soil-fertilizer treatment combinations over time (Table 3).

**Table 3.** Analysis of variance summary of the effects of soil (S), fertilizer treatment (F), sampling time (T), and their interactions on the change in water-soluble (WS) and Mehlich-3 (M3)-extractable nutrient (*i.e.*, K, S, Na, Mn, Zn, Cu, and B) concentrations from initial values.

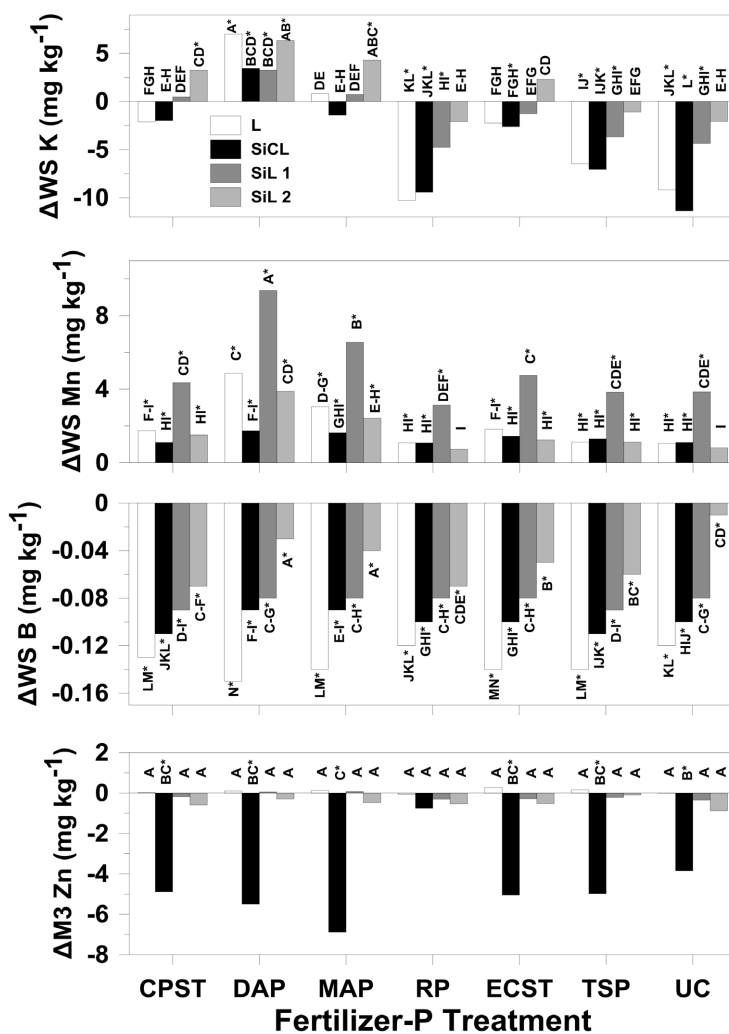
Source of variation	Water-soluble nutrients							Mehlich-3-extractable nutrients						
	$\Delta K$	$\Delta S$	$\Delta Na$	$\Delta Mn$	$\Delta Zn$	$\Delta Cu$	$\Delta B$	$\Delta K$	$\Delta S$	$\Delta Na$	$\Delta Mn$	$\Delta Zn$	$\Delta Cu$	$\Delta B$
	<i>P</i>													
S	<0.01	0.78	<0.01	<0.01	<0.01	0.38	<0.01	<0.01	0.01	<b>0.03</b>	<0.01	<0.01	<0.01	0.10
F	<0.01	<b>&lt;0.01</b>	0.20	<0.01	<0.01	0.70	<0.01	0.79	<b>&lt;0.01</b>	0.33	<b>0.01</b>	0.26	<b>&lt;0.01</b>	0.52
T	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<b>&lt;0.01</b>	<0.01	<b>0.04</b>	<0.01	<0.01
S x F	<b>&lt;0.01</b>	0.92	0.88	<b>&lt;0.01</b>	<0.01	0.81	<b>&lt;0.01</b>	0.92	0.24	1.00	0.83	<b>0.03</b>	0.07	0.72
S x T	<b>&lt;0.01</b>	<b>&lt;0.01</b>	<b>&lt;0.01</b>	<b>&lt;0.01</b>	<0.01	<b>&lt;0.01</b>	<b>&lt;0.01</b>	<b>&lt;0.01</b>	<b>&lt;0.01</b>	0.49	<b>&lt;0.01</b>	0.42	<b>&lt;0.01</b>	<b>0.03</b>
F x T	0.34	0.89	0.88	<b>&lt;0.01</b>	<0.01	<b>0.04</b>	0.26	0.61	0.99	1.00	0.46	0.86	0.68	0.84
S x F x T	1.00	1.00	0.90	0.22	<b>0.02</b>	0.06	0.44	0.85	1.00	1.00	1.00	0.99	0.49	0.99

**Table 4.** Fertilizer-phosphorus (P)-source effects, averaged over soils and time, on the change ( $\Delta$ ) in water-soluble (WS) soil sulfur (S) and Mehlich-3 (M3)-extractable soil S, manganese (Mn), and copper (Cu) concentrations from the initial concentrations for a 9-month laboratory incubation experiment in multiple soil textures.

Soil property	Fertilizer-P source						
	MAP <sup>†</sup>	DAP	TSP	CPST	ECST	RP	UC
$\Delta WS S$ (mg.kg <sup>-1</sup> )	23.7 B* <sup>‡</sup>	26.3 A*	22.1 B*	12.8 C*	13.8 C*	12.6 C*	12.8 C*
$\Delta M3 S$ (mg.kg <sup>-1</sup> )	21.2 B*	23.4 A*	20.9 B*	11.6 C*	13.0 C*	11.5 C*	11.6 C*
$\Delta M3 Mn$ (mg.kg <sup>-1</sup> )	66.6 BC*	70.6 A*	64.4 C*	65.9 BC*	64.4 C*	67.7 A-C*	68.0 AB*
$\Delta M3 Cu$ (mg.kg <sup>-1</sup> )	0.4 D*	0.4 CD*	0.5 B-D*	0.5 BC*	0.5 B-D*	0.5 A*	0.5 B*

<sup>†</sup>Monoammonium phosphate (MAP), diammonium phosphate (DAP), triple superphosphate (TSP), chemically precipitated struvite (CPST), electrochemically precipitated struvite (ECST), and rock phosphate (RP); <sup>‡</sup>Means in a row followed by different letters are different at  $P < 0.05$ ; \*An asterisk (\*) indicates mean change is different than zero ( $P < 0.05$ ).

Averaged across soils and sampling times, WS-S concentrations increased ( $P < 0.05$ ) from the initial in all fertilizer treatments (Table 4). The change in WS-S concentration was most positive from DAP and was least positive from RP, which did not differ from CPST, ECST, and the UC (Table 4). The change in WS-S concentration did not differ between MAP and TSP, but both were intermediate and differed from DAP and CPST, ECST, RP, and the UC (Table 4).



**Figure 1.** Combined soil-fertilizer effects, averaged over sampling times, on the change ( $\Delta$ ) in water-soluble (WS) and Mehlich-3 (M3)-extractable soil potassium (K), manganese (Mn), boron (B), and zinc (Zn) concentrations from the initial concentrations (Table 2) for a 9-month laboratory incubation experiment.

Averaged across sampling time, the change in WS-K concentrations from the initial was most positive for the loam soil with DAP, while the change in WS-K concentration was most negative for the SiCL soil in the UC (Figure 1). The change in WS-K concentration did not differ from a change of zero for the loam soil with CPST, MAP, and ECST, the SiCL soil with CPST and MAP, the SiL 1 soil with CPST, MAP, and ECST, and the SiL 2 soil with RP, ECST, TSP, and the UC

(**Figure 1**). In contrast, the change in WS-Mn concentrations from the initial was most positive for the SiL 1 with DAP, while the change in WS-Mn concentration was numerically least positive for the SiL 2 soil with RP (**Figure 1**). The change in WS-Mn concentration did not differ from a change of zero for the SiCL 2 soil with RP and the UC (**Figure 1**). The change in WS-B concentrations from the initial was least negative for the SiL 2 soil in the UC, while the change in WS-B concentration was most negative for the loam soil with DAP (**Figure 1**).

Averaged across fertilizer treatments, the change in WS-K concentrations from the initial was most positive for the SiCL soil after 9 months, while the change in WS-K concentration was most negative for the SiCL after 2 months of incubation (**Table 5**). The change in WS-K concentration did not differ from a change of zero for the loam soil after 4, 6, and 9 months, the SiL 1 soil after 4 months, and the SiL 2 soil after 0.5 and 1 month of incubation (**Table 5**). The change in WS-S concentrations from the initial was most positive for the SiL 1 after 9 months, while the change in WS-S concentration was least positive for the SiL 2 soil after 1 month of incubation (**Table 5**). The change in WS-Na concentrations from the initial was most positive for the SiL 2 soil after 9 months, while the change in WS-Na concentration was least positive for the SiL 1 soil after 0.5 months of incubation (**Table 5**). The change in WS-Mn concentrations from the initial was most positive for the SiL 1 soil after 9 months, while the change in WS-Mn concentration was least positive for the SiCL soil after 4 months of incubation (**Table 5**). The change in WS-Mn concentration did not differ from a change of zero for the loam soil after 0.5, 1, and 2 months, the SiCL soil after 0.5, 1, 2, and 6 months, and the SiL 2 soil after 0.5, 1, 2, and 6 months of incubation (**Table 5**). The change in WS-Cu concentrations from the initial was most positive for the loam, SiCL, SiL 1, and SiL 2 soils after 4 and 6 months, while the change in WS-Cu concentration was most negative for the loam, SiL 1, and SiL 2 soils after 2 months of incubation (**Table 5**). The change in WS-Cu concentration did not differ from a change of zero for the loam soil after 9 months and the SiCL soil after 9 months of incubation (**Table 5**). The change in WS-B concentrations from the initial was numerically most positive for the SiL 1 soil after 1 month and the SiL 2 soil after 0.5, 1, 2, 4, and 6 months, while the change in WS-B concentration was numerically most negative for the loam soil after 9 months of incubation (**Table 5**). The change in WS-B concentration did not differ from a change of zero for the loam soil after 4, 6, and 9 months, the SiL 1 soil after 4 months, and the SiL 2 soil after 0.5 and 1 month of incubation (**Table 5**).

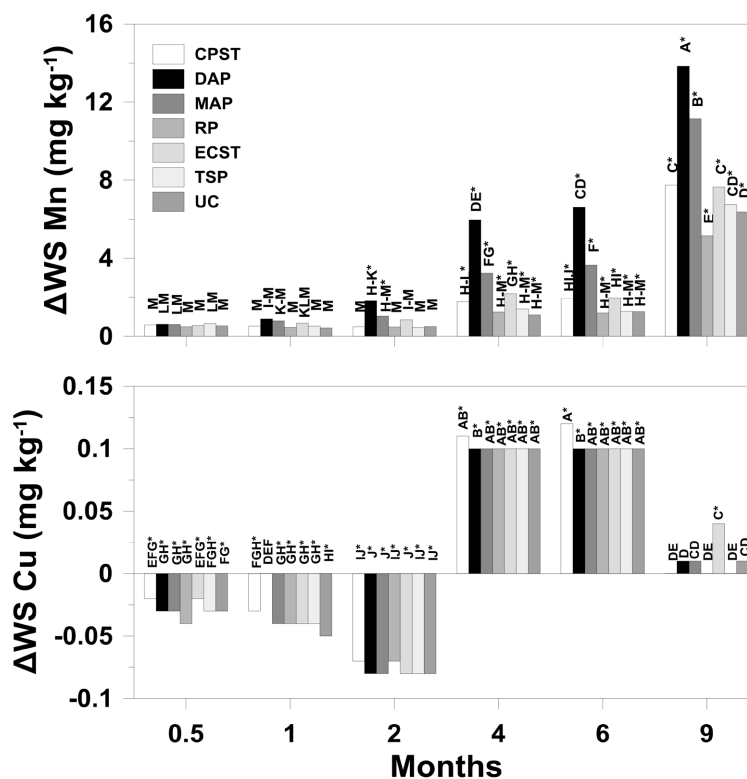
Averaged across soils, the change in WS-Mn concentrations from the initial was most positive for DAP after 9 months, while the change in WS-Mn concentration was numerically least positive for the UC after 1 month of incubation (**Figure 2**). The change in WS-Mn concentration did not differ from a change of zero for CPST, RP, ECST, TSP, and the UC after 0.5, 1, and 2 months or for DAP and MAP after 0.5 and 1 months of incubation (**Figure 2**). The change in WS-Cu concentrations from the initial was the most positive for CPST after 6 months, while the

change in WS-Cu concentration was most negative for MAP after 2 months of incubation (**Figure 2**). The change in WS-Cu concentration did not differ from a change of zero for CPST, MAP, RP, TSP, and the UC after 9 months or for DAP after 1 and 9 months of incubation (**Figure 2**).

**Table 5.** Soil effects, averaged over fertilizer treatments, on the change ( $\Delta$ ) in water-soluble (WS) soil potassium (K), sulfur (S), sodium (Na), manganese (Mn), copper (Cu), and boron (B) concentrations from the initial concentrations (**Table 2**) for a 9-month laboratory incubation experiment.

Soil property	Soil <sup>†</sup>	Incubation time (months)					
		0.5	1	2	4	6	9
$\Delta$ WS K (mg·kg <sup>-1</sup> )	L	-7.1 H-J**	-5.4 H*	-9.2 I-K*	0.5 FG	1.4 E-G	0.5 FG
	SiCL	-10.0 JK*	-10.4 JK*	-13.6 L*	-4.1 H*	3.1 C-F*	8.8 A*
	SiL 1	-4.3 H*	-4.0 H*	-10.8 KL*	1.1 FG	5.0 B-D*	4.7 B-E*
	SiL 2	-0.6 G	0.5 FG	-5.9 HI*	2.6 D-F*	7.3 AB*	5.5 BC*
$\Delta$ WS S (mg·kg <sup>-1</sup> )	L	9.3 G-I*	8.2 G-I*	10.3 F-H*	17.6 DE*	22.7 C*	35.7 B*
	SiCL	11.7 FG*	8.5 G-I*	10.5 F-H*	14.4 EF*	21.3 CD*	41.0 A*
	SiL 1	9.4 G-I*	6.4 HI*	10.3 F-H*	16.6 E*	24.4 C*	45.7 A*
	SiL 2	6.4 HI*	5.4 I*	9.0 G-I*	14.9 EF*	22.5 C*	42.9 A*
$\Delta$ WS Na (mg kg <sup>-1</sup> )	L	4.1 IJ*	5.2 H*	5.5 H*	11.3 E*	15.3 D*	20.6 B*
	SiCL	3.2 J-L*	4.5 HI*	3.6 I-K*	7.7 G*	10.1 F*	15.3 D*
	SiL 1	2.0 L*	2.6 KL*	3.7 I-K*	9.2 F*	14.8 D*	19.8 B*
	SiL 2	2.5 KL*	3.5 I-K*	5.6 H*	10.4 EF*	16.9 C*	22.0 A*
$\Delta$ WS Mn (mg·kg <sup>-1</sup> )	L	0.4 GH	0.6 GH	0.8 GH	2.4 EF*	2.7 E*	5.7 C*
	SiCL	0.6 GH	0.6 GH	0.6 GH	1.0 F-H*	0.8 GH	4.3 D*
	SiL 1	1.1 F-H*	1.1 F-H*	1.5 E-G*	5.1 CD*	5.8 C*	16.0 A*
	SiL 2	0.1 GH	0.1 H	0.2 GH	1.2 FG*	0.9 GH	7.4 B*
$\Delta$ WS Cu (mg·kg <sup>-1</sup> )	L	0.0 EF*	0.0 GH*	-0.1 I*	0.1 A*	0.1 A*	0.0 CD
	SiCL	0.0 EF*	0.0 E*	0.0 GH*	0.1 A*	0.1 A*	0.0 CD
	SiL 1	0.0 DE*	0.0 EF*	-0.1 H*	0.1 A*	0.1 A*	0.0 B*
	SiL 2	0.0 GH*	0.0 FG*	-0.1 I*	0.1 A*	0.1 A*	0.0 BC*
$\Delta$ WS B (mg·kg <sup>-1</sup> )	L	-0.1 HI*	-0.1 HI*	-0.1 K*	-0.1 E-G*	-0.1 IJ*	-0.2 L*
	SiCL	-0.1 G-I*	-0.1 B-D*	-0.1 F-H*	-0.1 C-E*	-0.1 EF*	-0.1 JK*
	SiL 1	-0.1 DE*	0.0 A-C*	-0.1 DE*	-0.1 D-F*	-0.1 BC*	-0.1 G-I*
	SiL 2	0.0 AB*	0.0 A*	0.0 AB*	0.0 AB*	0.0 AB*	-0.1 E-G*

<sup>†</sup>Loam (L), silty clay loam (SiCL), silt loam (SiL); \*Means in a row followed by different letters are different at  $P < 0.05$ ; \*An asterisk (\*) indicates mean change is different than zero ( $P < 0.05$ ).



**Figure 2.** Fertilizer treatment effects, averaged over soils, on the change ( $\Delta$ ) in water-soluble (WS) soil manganese (Mn) and copper (Cu) concentrations from the initial concentrations (**Table 2**) for a 9-month laboratory incubation experiment.

The combined effects of soil and fertilizer treatments over time on the change in WS-Zn concentrations from the initial were complex (**Table 6**). The change in WS-Zn concentrations ranged from a low of -0.46 in the SiL 2-TSP after 9 months to a 1.27 in the SiCL-RP soil-fertilizer combination after 4 months of incubation. Changes in WS-Zn concentration with a magnitude of  $\pm 0.19$  mg·kg<sup>-1</sup> were significantly different from a change of zero. After 0.5 months of incubation, all fertilizer treatments in the loam and all but TSP in the SiCL soil had WS-Zn concentrations that increased from the initial, while all fertilizers in the SiL 1 soil and all but ECST in the SiL 2 soil, which significantly decreased, had WS-Zn concentrations that did not change from the initial (**Table 6**). After 9 months of incubation, all fertilizer treatments in the loam had WS-Zn concentrations that did not change from the initial, except for TSP and the UC which decreased from the initial, while all fertilizer treatments in the SiCL, SiL 1, and SiL 2 soils had WS-Zn concentrations that had decreased from the initial soil-Zn concentration (**Table 6**).

Treatment effects on WS nutrients likely occurred for a variety of reasons. Averaged across soils and sampling times, WS-S concentrations increased for all fertilizer treatments relative to the initial concentration (**Table 4**). The greater increases in WS-S concentration from the initial from MAP, DAP, and TSP may have been due to the large WS-S concentrations present in MAP, DAP, and TSP (**Table 1**) [28].

**Table 6.** Combined effects of soil [loam (L), silty clay loam (SiCL), and silt loam (SiL)], fertilizer treatment, and sampling time (0.5, 1, 2, 4, 6, and 9 months) on the change ( $\Delta$ ) in the water-soluble (WS) soil zinc (Zn) concentration compared to the initial soil Zn concentration (Table 2) for a 9-month laboratory incubation experiment.

Soil	Treat <sup>†</sup>	$\Delta$ WS Zn (mg·kg <sup>-1</sup> )					
		0.5 Months	1 Month	2 Months	4 Months	6 Months	9 Months
L	MAP <sup>†</sup>	0.32 D-M* <sup>‡</sup>	0.13 I-Y	0.00 R-i	0.40 B-H*	0.00 R-i	0.02 P-h
	DAP	0.33 D-M*	0.08 L-c	-0.09 X-l	0.35 D-K*	0.05 N-d	-0.09 W-l
	TSP	0.39 B-I*	0.17 H-V	0.00 R-i	0.45 B-G*	0.10 K-a	-0.21 e-n*
	RP	0.44 B-G*	0.35 D-K*	0.35 D-K*	0.50 B-E*	0.15 I-W	-0.05 T-k
	CPST	0.30 E-N*	0.27 E-Q*	0.00 R-i	0.35 D-K*	0.00 R-i	-0.17 c-m
	ECST	0.19 H-U*	0.16 H-W	-0.04 S-k	0.40 B-H*	0.05 N-d	-0.17 c-m
	UC	0.23 F-R*	0.13 I-Y	0.05 N-d	0.60 BC*	0.10 K-a	-0.22 e-n*
SiCL	MAP	0.21 G-T*	0.39 C-I*	0.07 M-d	0.37 C-J*	-0.03 R-k	-0.22 e-n*
	DAP	0.37 C-J*	0.65 B*	0.02 P-g	0.42 B-H*	0.07 M-d	-0.24 h-n*
	TSP	0.13 I-Z	0.22 G-R*	0.22 G-R*	0.42 B-H*	0.07 M-d	-0.23 e-n*
	RP	0.31 E-N*	0.58 BCD*	0.22 G-R*	1.27 A*	0.17 H-W	-0.24 f-n*
	CPST	0.21 G-S*	0.29 E-O*	-0.03 R-k	0.42 B-H*	-0.08 V-l	-0.26 j-n*
	ECST	0.34 D-L*	0.22 G-R*	-0.08 V-l	0.47 B-F*	0.02 P-g	-0.25 i-n*
	UC	0.51 B-E*	0.27 E-Q*	0.27 E-Q*	1.22 A*	0.02 P-g	-0.23 e-n*
SiL 1	MAP	0.02 P-h	-0.04 S-k	-0.07 U-k	0.18 H-V	-0.07 U-k	-0.24 i-n*
	DAP	0.00 R-j	-0.01 R-j	0.33 D-M*	0.18 H-V	-0.02 R-j	-0.22 e-n*
	TSP	0.11 J-a	0.03 O-e	-0.07 U-k	0.18 H-V	-0.02 R-j	-0.28 k-n*
	RP	0.03 P-f	0.05 N-d	-0.12 Y-l	0.23 F-R*	-0.02 R-j	-0.28 k-n*
	CPST	0.14 I-X	0.10 K-b	-0.12 Y-l	0.23 F-R*	-0.02 R-j	-0.22 e-n*
	ECST	0.02 P-h	0.06 N-d	-0.07 U-k	0.28 E-P*	-0.02 R-j	-0.24 i-n*
	UC	0.05 N-d	0.09 L-c	-0.07 U-k*	0.38 C-I	-0.07 U-k	-0.25 i-n*
SiL 2	MAP	-0.14 a-l	-0.15 a-l	-0.29 k-n*	0.06 N-d	-0.19 d-m*	-0.45 n*
	DAP	-0.11 X-l	-0.18 d-m	-0.24 g-n*	0.06 N-d	-0.19 d-m*	-0.42 m-n*
	TSP	-0.06 U-k	-0.19 d-m*	-0.29 k-n*	0.01 Q-i	-0.19 d-m*	-0.46 n*
	RP	-0.15 a-l	-0.13 Z-l	-0.29 k-n*	0.11 J-a	-0.14 a-l	-0.45 n*
	CPST	-0.16 b-l	-0.18 d-m	-0.29 k-n*	0.01 Q-i	-0.19 d-m*	-0.45 n*
	ECST	-0.23 e-n*	-0.19 d-m*	-0.24 g-n*	0.11 J-a	-0.19 d-m*	-0.42 m-n*
	UC	0.09 K-b	-0.07 U-k	-0.34 l-n*	0.11 J-a	-0.19 d-m*	-0.43 m-n*

<sup>†</sup>Fertilizer 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); <sup>‡</sup>All means for soil-fertilizer treatment combinations followed by different letters are different at  $P < 0.05$ . Due to the large number of treatment combinations, letter notations started with a set of capital letters and continued to a set of lower-case letters if necessary; \*An asterisk (\*) indicates mean change is different than zero ( $P < 0.05$ ).

Averaged across sampling times, WS-K concentrations varied among soil-fertilizer combinations. The increase from the initial in all soils with DAP (**Figure 1**), may have been due to DAP having the largest initial total WS-K concentration among fertilizers (**Table 1**). The greater decreases of WS-K concentrations from the initial in loam and SiCL soils with RP and TSP fertilizer treatments may have been due to loam and SiCL soils having the greatest initial K concentrations (**Table 2**). The greater increase in WS-Mn concentrations from the initial for all fertilizer treatments in the SiL 1 soil was likely due to the SiL 1 soil having the largest initial WS-Mn concentration among soils (**Table 2**), which could be due to the aquatic properties of SiL 1. Water-soluble-B concentrations decreased from the initial for all soil-fertilizer combinations (**Figure 1**), which may have been attributed to the increased WS-Ca concentrations for all soil-fertilizer combinations observed by Anderson *et al.* [12], causing the formation of calcium borate complexes, which may have lowered the availability of B in the soils [29].

Averaged across fertilizer treatments, WS-K concentrations had generally decreased from the initial after 4 months of incubation, at which point, for all soils, except the SiCL soil, WS-K concentration had increased from the initial, and, for all soils, WS-K concentrations continued to increase and had increased from the initial after 9 months of incubation (**Table 5**). The behavior of WS-K may have been due to soil microbes taking up excess K at the beginning of the incubation period before dying and slowly releasing K back into the soil as microbes decomposed later during the incubation period. WS-S concentrations generally increased in all soils over time (**Table 5**). This could be due to phosphate anions added from fertilizers replacing sulfate anions at anion adsorption sites, thus releasing sulfate ions into the soil solution [30]. The loam having a significantly lower WS-S concentration than the other three soils (**Table 5**) may have been due to the loam having the lowest initial WS-S concentration (**Table 2**). WS-Na and -Mn concentrations generally increased in all soils over time (**Table 5**). The general increase in WS-Na and -Mn concentrations over time was likely due to the influx of cations from the dissolving fertilizers, which replaced Na and Mn ions on soil exchange sites [31]. WS-Cu concentrations had generally decreased slightly from the initial after 4 months of incubation, at which point WS-Cu concentrations had increased from the initial in all soils before decreasing slightly by after 9 months of incubation (**Table 5**). At 9 months of incubation, WS-Cu concentrations did not differ from a change of zero in the loam or SiCL soils (**Table 5**). There was no clear explanation for the behavior of WS-Cu over the 9-month period. The general decrease in WS-B concentrations (**Table 5**) may have been due to the formation of calcium borate complexes associated with the increase in WS-Ca concentrations observed in all soil-fertilizer combinations [12], which may have reduced soil-B availability [29].

Averaged across soils, WS-Mn concentrations generally increased for all fertilizer treatments over time (**Figure 2**). The larger increase in WS-Mn concentrations from the initial in the DAP treatment at most sampling times was unex-

pected since DAP had one of the lowest initial WS-Mn concentrations (**Table 1**). Water-soluble-Cu concentrations had generally decreased from the initial after 4 months of incubation, at which point WS-Cu concentrations increased from the initial for all fertilizer treatments before generally decreasing after 9 months of incubation (**Figure 2**). After 9 months of incubation, WS-Cu concentrations did not differ from a change of zero for all fertilizer treatments, except for ECST, which had increased from the initial (**Figure 2**). There was no clear explanation for the behavior of WS-Cu over the 9-month period.

### 3.2. Mehlich-3-Extractable Nutrients

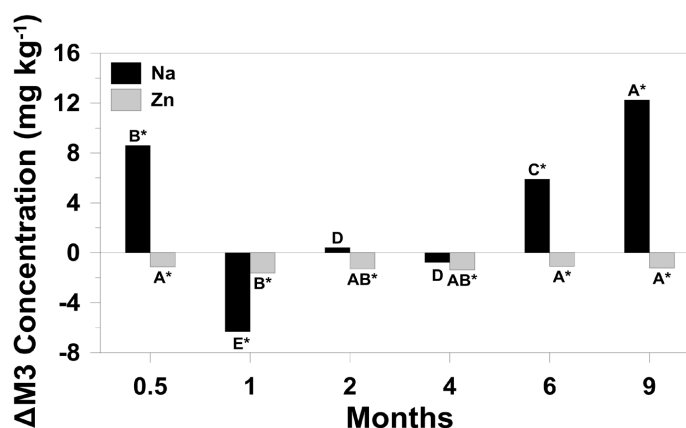
In general, though not formally compared, M3-extractable soil nutrient concentrations were greater than their respective WS nutrient concentrations, but the magnitude of M3 concentration changes from the initial was similar to those for WS nutrients. Similar to WS nutrients, the change in all M3-extractable nutrients evaluated in this study was affected by soil, fertilizer treatment, and/or sampling time (**Table 3**). The change in M3-Na concentration from the initial differed ( $P = 0.03$ ) among soils (**Table 3**). The change in M3-S, -Mn, and -Cu concentrations from the initial differed ( $P \leq 0.01$ ) among fertilizer treatments (**Table 3**). The change in M3-Na and -Zn concentrations from the initial differed ( $P \leq 0.04$ ) over time (**Table 3**). The change in M3-Zn concentrations from the initial differed ( $P = 0.03$ ) among fertilizer treatments within soils (**Table 3**). The change in M3-K, -S, -Mn, -Cu, and -B concentration from the initial differed ( $P \leq 0.03$ ) among soils over time (**Table 3**).

Averaged across fertilizer treatments and sampling times, M3-Na concentrations increased ( $P < 0.05$ ) from the initial only in the loam soil ( $8.15 \text{ mg}\cdot\text{kg}^{-1}$ ). The change in M3-Na concentration was most positive in the loam, which did not differ from the SiL 2 soil ( $4.08 \text{ mg}\cdot\text{kg}^{-1}$ ), and was numerically least positive in the SiL 1 soil ( $0.16 \text{ mg}\cdot\text{kg}^{-1}$ ), which did not differ from the SiCL ( $1.01 \text{ mg}\cdot\text{kg}^{-1}$ ) and SiL 2 soils.

Averaged across soils and sampling times, M3-S, -Mn, and -Cu concentrations increased ( $P < 0.05$ ) from the initial in all fertilizer treatments (**Table 4**). The change in M3-S concentration was most positive from DAP (23.4) and was least positive from RP (11.5), which did not differ from CPST (11.6), ECST (13.0) and the UC (11.6; **Table 4**). The change in M3-S concentration did not differ between MAP (21.2) and TSP (20.9), but both were intermediate and differed from DAP and CPST, ECST, RP, and the UC (**Table 4**). The change in M3-Mn concentration was most positive from DAP (70.6), which did not differ from RP (67.7) and the UC (68.0), and was least positive from ECST (64.4) and TSP (64.4), which did not differ from MAP (66.6), CPST (65.9), and RP (**Table 4**). The change in M3-Cu concentration was most positive from RP (0.5) and was least positive from MAP (0.4), which did not differ from DAP (0.4), TSP (0.5), and ECST (0.5; **Table 4**). The change in M3-Cu concentration did not differ between CPST (0.5) and the UC (0.5), but both were intermediate and CPST differed from MAP and RP, while

the UC differed from MAP, DAP, and RP (Table 4).

Averaged across soils and fertilizer treatments, M3-Na concentration from the initial increased ( $P < 0.05$ ) from the initial after 0.5, 6, and 9 months and decreased after 1 month of incubation (Figure 3). The change in M3-Na concentration from the initial was most positive after 9 months and was most negative after 1 month of incubation (Figure 3). The change in M3-Na concentrations differed from each other at each sampling time (Figure 3). The change in M3-Zn concentration decreased from the initial ( $P < 0.05$ ) at all sampling times (Figure 3). The change in M3-Zn concentration was least negative after 6 months, which did not differ from 0.5, 2, 4, and 9 months, and was most negative after 1 month, which did not differ from 2 and 4 months of incubation (Figure 3).



**Figure 3.** Change ( $\Delta$ ) in Mehlich-3 (M3)-extractable soil sodium (Na) and zinc (Zn) concentrations, averaged over soils and fertilizer treatments, from the initial concentrations (Table 2) for a 9-month laboratory incubation experiment.

Averaged across sampling time, M3-Zn concentrations decreased ( $P > 0.05$ ) from the initial only in the SiCL soil for all fertilizer treatments except RP (Figure 1). The change in M3-Zn concentration was numerically most positive in the loam soil with ECST and was most negative in the SiCL soil with MAP (Figure 1).

Averaged across fertilizer treatments, M3-K concentrations decreased ( $P > 0.05$ ) from the initial for all soil-time combinations except for the loam soil after 0.5, 2, and 6 months of incubation (Table 7). The change in M3-K concentration was numerically least negative for the loam soil after 6 months and was most negative for the SiCL soil after 4 months of incubation (Table 7). The change in M3-S concentrations increased ( $P > 0.05$ ) for all soil-time combinations (Table 7). The change in M3-S concentration was most positive for the SiL 1 soil after 9 months and was least positive for the SiCL soil after 1 month of incubation (Table 7). Similar to Mehlich-3-S, the change in M3-Mn concentrations increased ( $P > 0.05$ ) for all soil-time combinations (Table 7). The change in M3-Mn concentration was most positive for the SiL 2 soil after 9 months and was least positive for the loam soil after 1 month of incubation (Table 7). The change in M3-Cu concentration was most positive for the SiCL soil after 6 months and was most negative for the

SiL 2 soil after 1 and 9 months of incubation (Table 7). The change in M3-Cu concentration did not differ from a change of zero for the loam soil after 0.5, 4, and 6 months, the SiL 1 soil after 0.5 and 6 months, and the SiL 2 soil after 0.5 months of incubation (Table 7). In contrast to M3-S and -Mn, the change in M3-B concentrations decreased ( $P > 0.05$ ) for all soil-time combinations (Table 7). The change in M3-B concentration was least negative for the loam soil after 9 months and was most negative for the SiCL soil after 0.5, 1, and 2 months of incubation (Table 7).

**Table 7.** Soil effects, averaged over fertilizer treatment, on the change ( $\Delta$ ) in Mehlich-3 (M3)-extractable soil potassium (K), sulfur (S), manganese (Mn), copper (Cu), and boron (B) concentrations from the initial concentrations (Table 2) for a 9-month laboratory incubation experiment.

Soil property	Soil <sup>†</sup>	Incubation time (months)					
		0.5	1	2	4	6	9
$\Delta$ M3 K (mg·kg <sup>-1</sup> )	L	-5.0 A*	-18.3 CD*	-7.6 AB	-23.9 C-E*	-1.9 A	-16.9 BC*
	SiCL	-133.0 L*	-118.5 K*	-119.8 K*	-157.5 M*	-135.0 L*	-130.0 L*
	SiL 1	-39.4 F-I*	-44.4 G-I*	-34.7 E-H*	-50.8 IJ*	-40.4 F-I*	-30.1 C-F*
	SiL 2	-38.5 E-I*	-44.1 F-I*	-32.0 D-G*	-59.7 J*	-43.9 F-I*	-49.2 HI*
$\Delta$ M3 S (mg·kg <sup>-1</sup> )	L	7.2 F-H*	6.1 GH*	9.3 E-G*	14.6 D*	21.3 C*	37.3 B*
	SiCL	7.7 F-H*	4.3 H*	6.8 F-H*	7.7 F-H*	12.8 DE*	35.4 B*
	SiL 1	8.5 E-H*	6.5 F-H*	11.0 D-F*	15.3 D*	23.3 C*	47.7 A*
	SiL 2	7.6 F-H*	6.4 F-H*	10.7 D-G*	13.2 DE*	22.1 C*	45.4 A*
$\Delta$ M3 Mn (mg·kg <sup>-1</sup> )	L	26.4 LM*	21.2 M*	21.4 M*	35.6 K*	40.1 I-K*	40.7 I-K*
	SiCL	65.2 H*	49.1 IJ*	50.4 I*	97.4 D*	97.0 D*	117.9 BC*
	SiL 1	49.1 IJ*	34.6 KL*	38.5 JK*	75.9 FG*	84.6 E*	85.1 E*
	SiL 2	78.4 EF*	45.1 I-K*	67.2 GH*	112.2 C*	125.5 B*	144.1 A*
$\Delta$ M3 Cu (mg·kg <sup>-1</sup> )	L	0.0 F	-0.1 I-L*	-0.1 H-K*	-0.1 G-J	0.0 FG	-0.1 J-M*
	SiCL	2.5 B*	2.2 E*	2.3 D*	2.4 C*	2.6 A*	2.2 E*
	SiL 1	0.0 F-I	-0.2 L-N*	-0.2 J-N*	-0.2 K-N*	0.0 F-H	-0.2 M-O*
	SiL 2	0.0 F	-0.4 P*	-0.3 NO*	-0.3 OP*	-0.2 MN*	-0.4 P*
$\Delta$ M3 B (mg·kg <sup>-1</sup> )	L	-2.2 F-K*	-2.2 G-K*	-1.9 C-E*	-2.1 D-I*	-2.0 C-H*	-1.4 A*
	SiCL	-2.4 JK*	-2.4 K*	-2.4 I-K*	-2.3 I-K*	-2.2 G-K*	-1.7 BC*
	SiL 1	-2.1 D-J*	-2.1 D-J*	-2.1 D-H*	-2.0 C-G*	-1.9 C-F*	-1.5 AB*
	SiL 2	-2.1 E-K*	-2.1 E-J*	-2.2 E-K*	-2.1 D-J*	-1.9 C-G*	-1.8 B-D*

<sup>†</sup>Loam (L), silty clay loam (SiCL), silt loam (SiL); \*Means in a soil property followed by different letters are different at  $P < 0.05$ ; \*An asterisk (\*) indicates mean change is different than zero ( $P < 0.05$ ).

Similar to WS nutrients, treatment effects on M3 nutrients occurred for a variety of reasons. Averaged across fertilizer treatments and sampling times, the greater increase in M3-Na concentration from the initial in the loam was unexpected since the initial M3-Na concentration was lowest in the loam (**Table 2**).

Averaged across soils and sampling times, M3-S concentrations behaved similarly to WS-S concentrations (**Table 4**). The large changes in M3-S concentration from the initial from MAP, DAP, and TSP may have been due to the large initial M3-S concentrations present in MAP, DAP, and TSP (**Table 1**; Camberato *et al.*, 2023). Similar to S, M3-Mn concentrations increased from the initial for all fertilizer treatments (**Table 4**). The increase was greatest for DAP, which was unexpected since DAP had a relatively low initial M3-Mn concentration (**Table 1**). This behavior of M3-Mn was similar to that of WS-Mn among fertilizer treatments over time (**Figure 2**). Similar to S and Mn, M3-Cu concentrations increased from the initial for all fertilizer treatments (**Table 4**). The increase was greatest for RP, which was also unexpected since RP had a relatively low initial M3-Cu concentration (**Table 1**).

Averaged across soils and fertilizer treatments, M3-Zn concentrations decreased from the initial at all sampling times (**Figure 3**). Mehlich-3-Zn concentrations remained relatively constant and did not differ at any sampling time, except for after 1 month, at which point the change in M3-Zn concentrations was more negative than at any other sampling time (**Figure 3**). This decrease could be caused by the formation of zinc phosphate, which is relatively insoluble and could reduce the availability of Zn. Averaged across sampling times, the significant decrease in M3-Zn concentration in the SiCL soil (**Figure 1**) may have been due to the SiCL soil having the greatest initial M3-Zn concentration (**Table 2**).

Averaged across fertilizer treatments, M3-K concentrations decreased from the initial in all soils over time, where M3-K concentrations decreased from the initial after 0.5 months of incubation, and then remained somewhat constant, with no obvious temporal trend thereafter (**Table 7**). The decrease in M3-K concentration was greatest in the SiCL, which may have been due to SiCL having the greatest initial M3-K concentration (**Table 2**). Mehlich-3-S concentrations generally increased from the initial in all soils over time (**Table 7**), which may have been due to phosphate ions from fertilizers replacing sulfate ions at soil adsorption sites. By 9 months of incubation, the change in M3-S concentrations was 4.5 to 6 times greater than at 0.5 months of incubation (**Table 7**). Mehlich-3-Mn concentrations generally increased in all soils over time (**Table 7**). By 9 months of incubation, the change in M3-Mn concentrations was approximately two times greater than at 0.5 months of incubation (**Table 7**). The change in M3-Cu concentrations from the initial differed between soils over time (**Table 7**). In general, M3-Cu concentrations decreased from the initial in the loam, SiL 1, and SiL 2 soils, but increased from the initial in the SiCL (**Table 7**). The general decrease in M3-B concentrations (**Table 7**) may be due to the formation of calcium borate complexes associated with the increase in Ca concentrations observed in all soil-fertilizer combi-

nations [12], which likely reduced the soil-B availability [29].

### 3.3. Implications

Regarding the use of ECST as an alternative P-fertilizer and despite the experiment being conducted in the absence of plants, it is unlikely that there would be any negative effects on soil quality or crop growth from unintended additions and accumulation of micronutrients and potassium. The results of this study indicate that ECST had similar effects on soil micronutrients and potassium as commercially available CPST and traditional fertilizer-P sources (*i.e.*, DAP, MAP, and TSP) in multiple soil textures with a history of agricultural management. The results of this study indicated that struvite fertilization would not create any additional concern for runoff water quality or nutrient leaching due to the similar effects of struvite fertilization on soil nutrient concentrations compared to other fertilizer-P sources. Results supported conclusions of Anderson *et al.* [12] and others [13] [17]-[23] that struvite materials could be a viable alternative fertilizer-P source. In addition, results of this study and those of Anderson *et al.* [12] further support the idea that struvite recovery could be an effective option to reduce P and N concentrations and recycle nutrients from wastewater effluent that would otherwise be released into surface waters, thus decreasing eutrophication. The benefits of struvite recovery seem to be extensive, but further research will be needed to understand how plants respond to struvite fertilization, and research needs to be done at a larger scale to understand the behavior of struvite fertilizers in a less-controlled environment.

Though a detailed assessment of the potential environmental fate of soil micronutrients associated with CPST and ECST has not been conducted, Simms *et al.* [31] evaluate the leaching potential of P, N, Ca, Mg, and Fe across various soils. Simms *et al.* [31] concluded that macronutrient leachate concentrations from the top 10 cm varied among soil textures, fertilizer-P sources, and initial soil pH, but that temporal effects were complex. It stands to reason that, in lower concentrations than macronutrients, micronutrients added or rendered more mobile by the addition of struvite-P fertilizer materials likely pose little environmental threat.

## 4. Conclusions

The goal of this study was to evaluate the behavior of struvite in various soil textures over time compared to other conventional fertilizer-P sources, with the objective of assessing the effects of fertilizer-P source on water-soluble and Mehlich-3 extractable soil nutrients periodically over a 9-month period in multiple soils.

Results of this study did not support the hypothesis that ECST would have comparable WS and M3-extractable nutrient concentrations to CPST, while having smaller nutrient concentrations than MAP, DAP, and RP by the end of the incubation period. However, the micronutrient and potassium effects in soils fertilized with ECST did not differ from those of any other fertilizer treatment. Results of this study did not support the hypothesis that the SiL 1 and SiL 2 soils would ex-

hibit similar changes in nutrient concentrations, differing from those of the SiCL and the loam soils. Rather, there were no clear, general similarities or differences in the behavior of micronutrients and potassium among the four soils evaluated. The hypothesis that, in general, nutrient concentrations would increase over time among all treatment combinations was also not supported. While in some interactions there was a general increase in nutrient concentrations, in some cases there was a decrease and in other cases, there was no clear temporal trend. Results indicated that CPST and ECST fertilization have similar effects on soil nutrients. For all fertilizer, soil-fertilizer, fertilizer-time, and soil-fertilizer-time interactions, CPST and ECST exhibited similar effects on the change in soil nutrient concentrations from the initial.

Results indicated that struvite behaves similarly to other fertilizers and that soil texture and incubation time alone did not have a significant effect on soil nutrient concentrations. Combining results with other studies suggests that CPST and ECST behave similarly and that struvite, in general, can be an effective alternative fertilizer-P to more traditional fertilizers, such as DAP, MAP, and TSP. Although these results are promising, additional research needs to be conducted in other soil conditions and textures, in environments with plants, and on larger scales.

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## Conflicts of Interest

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

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