

Real-Wastewater-Derived-Struvite Effects on Dissolved Nutrients in Simulated Rainfall-Runoff Water

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How to cite this paper: Pruitt, L., Brye, K.R. and Miller, D.M. (2026) Real-Wastewater-Derived-Struvite Effects on Dissolved Nutrients in Simulated Rainfall-Runoff Water. *Journal of Environmental Protection*, 17, 321-341.
<https://doi.org/10.4236/jep.2026.175015>

Received: April 23, 2026

Accepted: May 12, 2026

Published: May 15, 2026

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Abstract

Phosphorus (P) is an essential nutrient needed in agriculture for proper plant growth and is often introduced into agricultural systems in the form of synthetic-P fertilizers. Struvite ($\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$) can be precipitated from municipal wastewater, recovering P, and has potential use as an alternative fertilizer-P source. The objective of this study was to evaluate the effects of fertilizer-P source [*i.e.*, synthetic electrochemically precipitated struvite (ECST_{syn}), real-wastewater derived ECST ($\text{ECST}_{\text{real}}$), chemically precipitated struvite (CPST), and monoammonium phosphate (MAP)], soil, and water source (*i.e.*, rainwater, groundwater, and struvite-removed real wastewater) on dissolved nutrient concentrations in runoff water from laboratory rainfall-runoff simulations. Concentration changes in runoff K, Na, Mn, Zn, and Cu were not attributed to fertilizer-P source ($P > 0.05$), but rather significant ($P < 0.05$) water-soil interactions. Runoff concentration changes for K and Na differed from zero ($P < 0.05$) among water source-soil combinations, with runoff K concentration changes ranging from -21.9 to $0.30 \text{ mg}\cdot\text{L}^{-1}$ and Na concentration changes ranging from -7.46 to $0.25 \text{ mg}\cdot\text{L}^{-1}$. Runoff concentration changes in Mn, Zn, and Cu did not differ from zero across most water source-soil combinations or were relatively small ($< 0.12 \text{ mg}\cdot\text{L}^{-1}$). Monoammonium phosphate produced a significant change in runoff S concentration ($0.20 \text{ mg}\cdot\text{L}^{-1}$), while changes in S concentration from CPST ($0.04 \text{ mg}\cdot\text{L}^{-1}$), $\text{ECST}_{\text{real}}$ ($-0.02 \text{ mg}\cdot\text{L}^{-1}$), and ECST_{syn} ($-0.03 \text{ mg}\cdot\text{L}^{-1}$) did not differ from a change of zero. The similarities in micronutrient responses between MAP and struvite treatments suggested potential parallels in fertilizer-P source behavior within an agroecosystem.

Keywords

Dissolved Nutrients, Struvite, Rainfall Simulation, Runoff, Water Quality

1. Introduction

Phosphorus (P) is a key nutrient needed for plant growth and production in agricultural ecosystems, as P is required by plants to undergo processes such as root development, nucleic acid replication, and, most significantly, energy transfer utilizing adenosine triphosphate (ATP) molecules [1]. Maximum plant productivity is achieved at approximately 30 μmol of available soil P; however, about 1 μmol of P is truly available for plant uptake in the majority of soils [2]. As such, P is recognized as one of the primary growth-limiting factors in agricultural ecosystems [2].

Synthetic-P fertilizers provide supplemental P to cultivated crops across agricultural systems [3]. With modern intensive management practices, agricultural soils have been depleted of many nutrients, thus requiring the input of synthetic-P fertilizers in order to continue to produce sufficiently large yields [4]. Non-renewable rock-phosphate (RP) reserves are the primary P source for synthetic-P fertilizers. Currently, the demand for P fertilizers is increasing with rising agricultural demands, potentially exhausting the extraction of finite RP reserves in as little as 100 to 250 years [3]. One renewable, alternative-P source currently being explored is the recycling of excess nutrients contained in municipal wastewater.

Wastewater became a focal point of nutrient recovery due to the P availability in wastewater and the large wastewater-production volume [5]. Struvite, magnesium ammonium phosphate hexahydrate ($\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$) is a crystalline mineral that can be precipitated from P-containing wastewaters. Two methods of extracting nutrients from wastewater to create struvite are: 1) via chemical precipitation through the addition of magnesium (Mg)-containing salts, and 2) electrochemical precipitation using an electrical current imposed on a Mg electrode [6] [7].

Current research into the viability of struvite's use as an alternative fertilizer-P source is ongoing, but struvite has several notable characteristics. First, struvite often acts as a slow-release fertilizer due to struvite's low solubility in water, yet large citrate, a plant-root-exuded compound, solubility. As such, nutrients released from applied struvite are suspected to be less susceptible to loss via runoff water and are believed to be released in better timing with plant needs throughout a growing season [8]. Second, struvite has been shown to release adequate P concentrations for plant growth, even with the slow-release characteristics [5]. Lastly, struvite dissolution is significantly correlated with more acidic soil conditions [8].

One research focus in the characterization of struvite as a potential alternative fertilizer-P source involves exploring the interactions between struvite, irrigation water source, and soil characteristics. Morrison [9] reported results from an experiment evaluating the effects of soil, fertilizer-P source, and water source over time on runoff-water-quality parameters. The fertilizer-P sources evaluated included electrochemically precipitated struvite (ECST) from real (ECST_{real}) and synthetic (ECST_{syn}) wastewater, chemically precipitated struvite (CPST), and monoammonium phosphate (MAP), a commonly used, commercially available

fertilizer-P source [9]. Results indicated that runoff-nutrient concentrations from struvite treatments did not differ from those from MAP [9]. However, analyses of micronutrient concentrations in runoff water as affected by soil, fertilizer-P source, and water source have yet to be evaluated.

Dissolved nutrients, such as iron (Fe), manganese (Mn), copper (Cu), and zinc (Zn), serve multiple essential functions in soil ecosystems. There is significant correlation between adequate concentrations of micronutrients and the healthy structure and function of soil microbial communities [10]. Additionally, micronutrients impact plant growth, as micronutrients are necessary in small concentrations to avoid plant deficiencies, but can become toxic in the case of increased concentrations beyond tolerable limits [11]. Therefore, understanding the effects of struvite, soil characteristics, potential irrigation water sources, and their interactions on the movement and concentrations of micronutrients is vital for evaluating the viability of struvite's use as an alternative fertilizer-P source.

The ability of struvite to perform similarly to commercially available fertilizer-P sources, while providing a slow release of nutrients, has presented a great argument for the potential use of struvite as a viable, alternative, fertilizer-P source. However, research is still needed to fully understand the extent of struvite's potential behavior and effects on agroecosystems to ensure the informed, mindful use of the material as an alternative fertilizer-P source. The objective of this study was to evaluate the effects of soil, fertilizer-P source, and simulated-rainfall water source on micronutrient concentrations (*i.e.*, K, S, Na, Mn, Zn, and Cu) in runoff water. It was hypothesized that runoff water quality would differ among soils due to differences in initial soil pH and soil organic matter concentration among the soils [9]. It was also hypothesized that runoff water quality would differ among simulated-rainfall water sources due to differences in pH and electrical conductivity (EC) among simulated-rainfall water sources [9]. Furthermore, it was hypothesized that runoff water quality will differ among fertilizer-P sources due to differences in chemical composition resulting from the method of creation among fertilizer-P sources [9].

2. Materials and Methods

The current study is an extension of a 2023 study by Morrison [9], which examined the effects of different soils, fertilizer-P sources, and potential irrigation-water sources on runoff-water quality utilizing a rainfall-runoff simulator. Runoff-water pH, EC, and total P, calcium (Ca), magnesium (Mg), nitrate (NO_3^-), ammonium (NH_4^+), and soluble-reactive P (SRP) were measured in the runoff water after the rainfall simulation [9]. Results from Morrison [9] largely emphasized the impact of water-soil-fertilizer-P source treatment combinations on macronutrients. However, soluble dissolved nutrients (*i.e.*, K, S, Na, Mn, Zn, and Cu) in runoff water were not evaluated by Morrison [9], which will serve as the response variables evaluated in the current study. All runoff concentrations reported in the current study were measured in 2023 in association with the original study [9],

but were not previously statistically analyzed or reported.

2.1. Soil Collection and Analyses

Following procedures described in Morrison [9], four different soils were used in this study to represent an array of common agricultural soils from the mid-southern United States, specifically southwestern Missouri (MO) and western and eastern Arkansas (AR). The soils used in this study were collected from managed pastureland (MO) or cultivated, row-crop (AR) agricultural management [9].

Collection of bulk soil occurred for all four soils from a depth of 0 to 15 cm at the various sample locations. A Roxana fine sandy loam (Typic Udifluvents) was collected in December 2017 from a row-cropped field at the University of Arkansas Division of Agriculture's Vegetable Research Station located near Kibler in west-central AR. A Calloway silt loam (Aquic Fraglossudalfs) was collected early in spring 2021 from the edge of a row-cropped field at the University of Arkansas Division of Agriculture's Pine Tree Research Station located near Colt in eastern AR [9]. A Creldon silt loam (Oxyaquic Fragiudalfs) was collected in June 2021 from pastureland located at the University of Missouri Southwest Research Center located near Mount Vernon in southwest MO. A Dapue silt loam (Fluventic Hapludolls) was also collected in June 2021 from a different managed pastureland at the University of Missouri Southwest Research Center located near Mount Vernon, MO [9]. Once collection was completed, the moist soils were manually pressed through a 6-mm screen to remove large aggregates and any unwanted debris. Soils were then placed on tarps inside a greenhouse to air-dry for at least one week [9].

From each of the four sets of air-dried, bulk soil, three sub-samples were collected. Soil sub-samples were oven-dried at 70°C for 48 hours to determine the gravimetric water content before being grinding manually using a mortar and pestle. Dried, ground samples were sieved through a 2-mm mesh screen for chemical analyses [9].

Initial soil pH, electrical conductivity (EC), soil organic matter (SOM), total N (TN) and total C (TC) concentrations, particle-size distribution, and water-soluble nutrient concentrations were measured on oven-dried, ground, sieved soil [9]. Soil pH and electrical conductivity were measured potentiometrically in a 2-part-water: 1-part-soil slurry. The concentration of soil organic matter was measured gravimetrically using weight-loss-on-ignition, in which soil samples were combusted at 360°C in a muffle furnace [12] for two hours before comparison to initial masses. Total N and C concentrations were measured by high-temperature combustion using a VarioMAX CN analyzer (Elementar Americas Inc., Mt. Laurel, NJ). Sand, silt, and clay were measured using a modified hydrometer method over 12 hours [13]. Soil sub-samples were prepared in a 1:10 soil mass: water volume suspension, agitated for 1 hour, filtered through a 0.45- μ m filter, and analyzed for water-soluble soil K, S, Na, Mn, Zn, and Cu concentrations by inductively coupled, argon-plasma spectrometry (ICAPS; Spectro Arcos ICP, Spectro Analytical

Instruments, Inc., Kleve, Germany) [9].

Table 1 summarizes initial soil physical and chemical properties of each of the four soils [14] [15]. Initial SOM concentration ranged from 0.7% in the Roxanna to 4.2% in the Dapue soil, where all soils differed ($P \leq 0.05$) among one another [9]. Soil pH ranged from 5.77 in the Dapue to 7.46 in the Calloway soil, where all soils differed ($P \leq 0.05$) among one another [9]. Water-soluble nutrient concentrations (*i.e.*, K, Na, and Mn) differed ($P \leq 0.05$) among all soils [9]. Water-soluble K had the largest concentration of all water-soluble soil nutrients across all soils, except for the Calloway soil, which had the largest water-soluble Na concentration among all soils [9].

Table 1. Summary of the initial physical and chemical properties of the Dapue, Creldon, Calloway, and Roxana soil series used in the rainfall simulations (adapted from Morrison [9]).

Soil property	Dapue	Creldon	Calloway	Roxana	<i>P</i>
Sand (g·g ⁻¹)	0.20c [†]	0.24b	0.09d	0.44a	<0.01
Silt (g·g ⁻¹)	0.74b	0.67c	0.79a	0.47d	<0.01
Clay (g·g ⁻¹)	0.07c	0.09b	0.12a	0.10b	<0.01
Soil organic matter (%)	4.2a	3.4b	2.6c	0.7d	<0.01
pH	5.77d	6.03c	7.46a	6.17b	<0.01
Water-soluble nutrients (mg·kg ⁻¹)					
K	19.7b	20.3b	6.2c	44.8a	<0.01
S	12.2a	9.5b	12.0a	4.9c	<0.01
Na	4.8c	5.9b	19.5a	4.1d	<0.01
Mn	2.2a	1.7b	0.12d	0.57c	<0.01
Zn	0.90a	0.47bc	0.52b	0.33c	<0.01
Cu	0.06b	0.04c	0.03c	0.08a	<0.01

[†]Means within a soil property with different lowercase letters are different at $P \leq 0.05$.

2.2. Fertilizer Treatments

Following procedures in Morrison [9], four fertilizer treatments were used in this study along with an unamended control. The fertilizer treatments used were a commercially available CPST known as Crystal Green [16], commercially available MAP, and ECST_{real} and ECST_{syn} [9]. The real wastewater used for the ECST_{real} was gathered from the West Side Wastewater Treatment Facility in Fayetteville, AR and was produced in the Chemical Engineering Department at the University of Arkansas following procedures of Kékedy-Nagy [17]. The synthetic wastewater used for the ECST_{syn} was created in the Chemical Engineering Department at the University of Arkansas using similar average N and P concentrations to typical municipal wastewater [17] [18].

Anderson [14] characterized and reported the chemical composition of the EC-

ST_{syn}, CPST, and MAP fertilizer-P sources, while Morrison [9] characterized and reported the chemical composition of the ECST_{real} material using similar procedures to that of Anderson [14]. The ECST_{real} and ECST_{syn} materials both had a crystalline-flake-like consistency and the CPST and MAP materials were pelletized, thus all materials were finely ground to a similar powdery consistency across all four fertilizer treatments for chemical analyses [9].

Importantly from the chemical analyses, concentrations of water soluble elements (*i.e.*, P, K, Ca, Mg, Na, S, Fe, Mn, Zn, B, and Cu) were measured using a 1:10 fertilizer mass to water volume ratio. The fertilizer-water mixture was agitated for 1 hour and filtered through a 0.45- μ m filter before being analyzed by ICAPS [9] [12].

Table 2 adapts and summarizes initial chemical properties determined for each fertilizer P source from both Anderson [14] and Morrison [9]. Of the measured initial water-soluble, fertilizer nutrients, K, S, Na, Mn, and Zn concentrations were all greatest for MAP [9] [14]. Particularly, water-soluble MAP-K (1048 mg·kg⁻¹), -Na (1169 mg·kg⁻¹), and -S (13280 mg·kg⁻¹) concentrations had magnitudes larger than concentrations of the same nutrients in CPST, ECST_{real}, and ECST_{syn} [9] [14].

Table 2. Summary of the initial chemical properties of the fertilizer-phosphorus (P) sources for monoammonium phosphate (MAP), chemically precipitated struvite (CPST), and synthetically produced electrochemically precipitated struvite (ECST_{syn}; adapted from Anderson [14]) and real-wastewater-derived ECST (ECST_{real}; adapted from Morrison [15]).

Fertilizer property	MAP	CPST	ECST _{syn}	ECST _{real}
pH	4.37	8.78	-	-
Total N (mg·kg ⁻¹)	107,000	57,000	33,000	32,800
Total P (mg·kg ⁻¹)	209,215	116,556	184,510	154,720
Total K (mg·kg ⁻¹)	1312	842	0.01	40.6
Water-soluble nutrients (mg·kg ⁻¹)				
K	1048	1.5	3.0	12.0
S	13,280	24.5	2.0	24.0
Na	1169	13.0	2.3	10.0
Mn	42.0	0.2	0.1	0.2
Zn	4.9	0.3	0.3	0.5
Cu	0.1	0.3	0.1	< 0.1

2.3. Water Sources

Following procedures in Morrison [9], three water sources (*i.e.*, rainwater, groundwater, and struvite-removed wastewater), representing three potential irrigation sources, were collected for use in this study. Rainwater was collected from a singular rainfall event in July 2021 in Fayetteville, AR. Groundwater was gathered in July 2021 from an already existing well located west of Fayetteville, AR at

the Savoy Research Center. Struvite-removed wastewater, produced as a by-product of the electrochemical precipitation of the ECST_{real} fertilizer material, was also obtained for use in the rainfall simulation. Morrison [15] conducted and reported the chemical characterization of three water sources.

Table 3 summarizes initial chemical properties among water sources [9]. Initial water pH ranged from 7.22 for the rainwater to 9.77 for the wastewater, where all initial pH values differed ($P \leq 0.05$) among water sources [9]. Similar to initial pH, initial water EC ranged from 0.0148 dS·m⁻¹ for the rainwater to 0.578 dS·m⁻¹ for wastewater, with ECs differing ($P \leq 0.05$) among water sources [9]. Initial K, S, and Na concentration were all greatest in wastewater and least in rainwater [9]. Initial Mn and Cu concentrations were notably small (<0.01 mg·L⁻¹) in all water sources [9].

Table 3. Summary of the initial chemical properties of the wastewater, groundwater, and rainwater used in the rainfall simulations (adapted from Morrison [9]).

Water property	Wastewater	Groundwater	Rainwater	<i>P</i>
pH	9.77a [†]	7.89b	7.22c	<0.01
Electrical conductivity (dS·m ⁻¹)	0.578a	0.461b	0.015c	<0.01
Nutrients (mg·L ⁻¹)				
K	20.2a	1.17b	0.41c	<0.01
S	12.1a	0.92b	0.16c	<0.01
Na	35.8a	13.2b	0.41c	<0.01
Mn	<0.01a	<0.01b	<0.01b	<0.01
Zn	0.05c	0.11a	0.06b	<0.01
Cu	<0.01a	<0.01b	<0.01b	<0.01

[†]Means within a water property with different lowercase letters are different at $P \leq 0.05$.

2.4. Rainfall-Runoff Simulation

Following procedures in Morrison [9], the ECST_{real} and ECST_{syn} fertilizer materials were only produced in small quantities due to their experimental nature and, as such, the study could not be conducted on the field scale. Thus, in order to simulate rainfall, or irrigation, onto soil-fertilizer treatment combinations in a smaller, laboratory setting, a rainfall-runoff simulator was constructed [9]. The simulator consisted of a simple wooden frame measuring 91.1-cm tall by 87.0-cm wide by 73.7-cm deep with four, tapered, plastic trays (*i.e.*, rain gutters intended for housing) placed along the bottom of the frame at a slope of 22.1% [9] (Figure 1). Above each of the four plastic trays was a set of seven drip emitters, with drip rates of 31.5 mL·min⁻¹ connected to each other via plastic tubing, which connected to a 20-L carboy that contained the water used for each rainfall-runoff simulation [9] (Figure 1).

2.4.1. Soil-Fertilizer Treatment Preparation

Following procedures in Morrison [9], to evaluate the response of soil and fertilizer treatments to simulated rainfall/irrigation, teabags made of synthetic fibers were used to contain each soil-fertilizer treatment combination. The intent was that the teabags would allow soil-fertilizer treatment combinations to remain stationary and contained, while still interacting with the simulated rainfall/irrigation. Each teabag (TamBee, B07TCDT76Q), measuring 15-cm wide by 20-cm long and made from a synthetic fiber, contained 175 grams of air-dried soil from each soil sample plus the appropriate mass of a fertilizer treatment that would be necessary to deliver a fertilizer-P rate of 56 kg P₂O₅ ha⁻¹ based on the mass of air-dried soil used. The specific fertilizer-P application rate was based on the recommendation for soybeans (*Glycine max* L. [Merr.]) grown on a loamy soil in Arkansas [19]. Once filled with soil and the appropriate fertilizer, each teabag was manually mixed using a gentle, massaging pressure to thoroughly combine the soil with the intended fertilizer treatment [9]. One week before the rainfall-runoff simulation was conducted, each teabag was gently wet with 20 mL of the intended water source to rehydrate the air-dried soil, while attempting to minimize any loss of soil-fertilizer treatment through the teabags during handling. In total, 180 teabags were prepared. There were three replications of each soil-fertilizer treatment combination, resulting in 60 teabags for each of the three water sources used [9].

2.4.2. Runoff Water Collection

Following procedures in Morrison [9], rainfall simulations were grouped by intended water source, with one water source tested each day on 21, 22, and 23 July 2021. Prior to starting the simulations, the plastic tubing was primed with the intended water source for that day (Figure 1). Additionally, prior to raining on each set of soil-fertilizer treatments, two rainfall simulations were conducted with one on the four empty trays and another with an empty, dry tea bag in each tray, for a total of eight blanks [9]. The order in which each soil-fertilizer treatment combination was tested was randomized within each water source grouping. One teabag was placed at a time near the lower end of the slope on a single tray of the four plastic trays, which allowed not only rainfall directly on to each teabag, but also allowed up-slope rainwater to flow underneath and through the soil-fertilizer-filled teabags (Figure 1). Then, the 20-L carboy containing the intended water source for that day's treatments was hoisted above the rainfall simulator to rest on a ladder and connected via a spigot and plastic tubing to the drip emitters above each tray. The water gravity-flowed into and through the tubing and drip emitters at a rate of approximately 3.5 cm·hr⁻¹, similar to the rate of a high-intensity rainfall event [9]. Each soil-fertilizer treatment combination was subjected to simulated rainfall for 6 minutes, with runoff water from each soil-fertilizer treatment allowed to flow down the plastic tray and collect in a rectangular, plastic container at the base of the tray. Approximately 220 mL of runoff water were collected for each treatment combination and were transferred from the collection container to smaller sample cups for pre-processing for immediate water-quality-parameter

measurements and eventual chemical analyses [9].



Figure 1. Images of the rainfall-runoff simulator (left) with additional angle to view the drip-emitters and tubing (right). The white trays were set at a slope of 22.1%. Tea bags containing the soil-fertilizer treatment combinations were placed on the white trays directly below the four down-slope drip emitters. Images adapted from 2021 photographs by M. Morrison.

2.4.3. Runoff Water Analyses

Following procedures of Morrison [9], sub-samples of all collected runoff water were filtered through a 0.45- μm filter into two, 20-mL vials and acidified using concentrated hydrochloric acid to preserve them for chemical analyses. Filtered, acidified runoff-water sub-samples were analyzed for K, S, Na, Mn, Zn, and Cu by inductively coupled, argon-plasma spectrometry (ICAPS; Spectro Arcos ICP, Spectro Analytical Instruments, Inc., Kleve, Germany) [20].

Following similar procedures used by Morrison [9], the resulting runoff water quality data from each separate water source was grouped and, for each treatment, the average of all eight blanks for each water quality parameter were subtracted from the raw measured water quality parameters to create blank-corrected values for each treatment combination tested. Then, water quality parameters were averaged across the unamended control treatment combinations and subtracted from the blank-corrected values to reduce the potential of data bias that could have arisen from the different initial soil properties (Table 1). The resulting values were a final set of water quality parameters that specifically quantified positive or negative changes in dissolved nutrient concentrations among soil-fertilizer treatment combinations and water sources. A positive change represented a concentration increase relative to the average of the unamended control treatment's runoff concentration, while a negative change represented a concentration decrease rela-

tive to the average of the unamended control treatment's runoff concentration.

2.5. Statistical Analyses

Following procedures in Morrison [9], water sources were randomized for the day they were used. Soil-fertilizer treatment combinations were randomized within each water source on the day each water source was used. A three-factor ANOVA was performed in SAS (version 9.4, SAS Institute, Inc., Cary, NC) using the PROC GLIMMIX procedure to evaluate the effects of water source, soil, fertilizer-P source, and their interactions on the change in runoff K, S, Na, Mn, Zn, and Cu concentrations. A normal data distribution was used for statistical analyses since the corrected, final water quality parameters ranged from negative to positive values [9]. Significance was judged at $P < 0.05$ for all data analyses. Means were separated by least significant difference at the 0.05 level.

For logistical reasons, a single water source was used at a time to process all soil-fertilizer treatment combinations. Furthermore, only four individual soil-fertilizer treatment combinations could be processed at a time due to having only four runoff trays available to use. Despite these procedural limitations, water source was assumed to be a randomized, but fixed effect, while any potential effect of tray set was ignored, when analyzing the resulting data. Consequently, these methodological limitations should be considered when interpreting the resulting water-source effects.

3. Results and Discussion

3.1. Runoff Nutrient Parameters

All changes in runoff water concentrations were affected ($P \leq 0.05$) by either water source, soil, and/or fertilizer-P source (Table 4). The change in dissolved sulfur concentrations differed only among fertilizer-P sources and was unaffected

Table 4. Analysis of variance summary of the effects of water source (W), soil (S), fertilizer-phosphorus (P) source (F), and their interactions on the change (Δ) in dissolved potassium (K), sulfur (S), sodium (Na), manganese (Mn), zinc (Zn), and copper (Cu) from the rainfall runoff simulation.

Source of variation	Δ [K]	Δ [S]	Δ [Na]	Δ [Mn]	Δ [Zn]	Δ [Cu]
	<i>P</i>					
Water source	<0.01	0.49	<0.01	0.98	<0.01	<0.01
Soil	<0.01	0.51	<0.01	0.81	<0.01	<0.01
Fertilizer-P source	0.32	0.03	0.29	0.68	0.81	0.85
W \times S	<0.01	0.70	<0.01	0.04	<0.01	<0.01
W \times F	0.16	0.47	0.58	0.29	0.20	0.57
F \times S	0.31	0.70	0.41	0.90	0.63	0.25
W \times F \times S	0.26	0.26	0.14	0.64	0.34	0.19

by soil and water source (Table 4). In contrast, the changes in dissolved K, Na, Mn, Zn, and Cu concentrations differed among water source-soil combinations, but were unaffected by fertilizer-P source (Table 4).

3.1.1. Sulfur

Sulfur was unique among all measured nutrients in that the runoff-S concentration change differed ($P \leq 0.05$) only among fertilizer-P source. The change in runoff S concentration was greatest from MAP ($0.20 \text{ mg}\cdot\text{L}^{-1}$), which did not differ from CPST ($0.04 \text{ mg}\cdot\text{L}^{-1}$), and was smallest from ECST_{syn} ($-0.03 \text{ mg}\cdot\text{L}^{-1}$). The change in runoff S concentration was similar among CPST, ECST_{syn}, and ECST_{real} ($-0.02 \text{ mg}\cdot\text{L}^{-1}$), while the change in runoff S concentration for MAP was the only change that differed ($P \leq 0.05$) from a change of zero.

3.1.2. Potassium

The change in runoff K concentration ranged from $0.30 \text{ mg}\cdot\text{L}^{-1}$ for the groundwater-Roxana combination to $-21.9 \text{ mg}\cdot\text{L}^{-1}$ for the wastewater-Calloway combination (Table 5). The change in runoff K concentration did not differ among soils for the rainwater or groundwater sources (Table 5). In contrast, the change in runoff K concentration differed significantly ($P \leq 0.05$) among all soils for the wastewater source ranging from a decrease of -12.9 in the Calloway to a decrease of $-2.07 \text{ mg}\cdot\text{L}^{-1}$ in the Roxana (Table 5). Furthermore, the change in runoff K concentration in the four soil-wastewater combinations decreased ($P \leq 0.05$) relative to the unamended control, whereas the change in runoff K concentration in all four soils in the rainwater and groundwater sources did not differ from a change of zero (Table 5).

3.1.3. Sodium

The change in runoff Na concentration ranged from $0.25 \text{ mg}\cdot\text{L}^{-1}$ for the rainwater-Creldon combination to $-7.46 \text{ mg}\cdot\text{L}^{-1}$ for the wastewater-Dapue combination (Table 5). Similar to runoff K, the change in runoff Na concentration did not differ among soils for the rainwater source (Table 5). Among soil-groundwater combinations, the change in runoff Na concentration was similar for the Creldon and Roxana soils, which both differed ($P \leq 0.05$) from the change in runoff Na concentration for the Dapue and Calloway soils, which also differed from each other (Table 5). Furthermore, the change in runoff Na concentration differed significantly ($P \leq 0.05$) among all soils for the wastewater source ranging from a decrease of $-0.23 \text{ mg}\cdot\text{L}^{-1}$ in the Calloway to a decrease of $-7.46 \text{ mg}\cdot\text{L}^{-1}$ in the Dapue (Table 5). The change in runoff Na concentration for the Creldon, Dapue, and Roxana soils in both the groundwater and wastewater sources differed ($P \leq 0.05$) from a change of zero, where all decreased relative to the unamended control (Table 5).

3.1.4. Manganese

The change in runoff Mn concentration ranged from $0.04 \text{ mg}\cdot\text{L}^{-1}$ for the rainwater-Creldon combination to $-0.02 \text{ mg}\cdot\text{L}^{-1}$ for both the rainwater-Roxana and groundwater-Creldon combinations (Table 5). Similar to runoff K, the change in

runoff Mn concentration did not differ among soils for the wastewater source (Table 5). The change in runoff Mn concentration was also similar among all soils, except for the Calloway soil, with the groundwater source and the change in runoff Mn concentration was similar among all soils, except for the Creldon soil, for the rainwater source (Table 5). Uniquely, the runoff Mn concentration change differed ($P \leq 0.05$) from a change of zero in only the rainwater-Creldon combination, where the runoff Mn concentration increased relative to the unamended control (Table 5).

Table 5. Summary of the effects of water source and soil on the change in runoff nutrient concentrations from the rainfall runoff simulation.

Water property	Water source	Soil series			
		Creldon	Dapue	Roxana	Calloway
Potassium	Rainwater	0.09a [†]	0.13a	0.16a	0.08a
	Groundwater	0.19a	0.03a	0.30a	-0.21a
	Wastewater	-13.3c*	-13.8d*	-2.07b*	-21.9e*
Sodium	Rainwater	0.25a [†]	-0.14abc	0.17ab	0.06abc
	Groundwater	-0.68d*	-1.67e*	-0.41cd*	0.12ab
	Wastewater	-6.65g*	-7.46h*	-2.42f*	-0.23bcd
Manganese	Rainwater	0.04a ^{†*}	0.02abc	-0.02bc	-0.01bc
	Groundwater	-0.02c	>-0.01abc	0.02abc	0.03ab
	Wastewater	0.02abc	0.01abc	>-0.01abc	>-0.01abc
Zinc	Rainwater	>-0.01abc [†]	<0.01a	>-0.01ab	-0.01d*
	Groundwater	-0.10f*	-0.06e*	-0.07e*	-0.12g*
	Wastewater	>-0.01abcd	>-0.01bcd*	>-0.01cd*	>-0.01abc
Copper	Rainwater	<0.01ab [†]	<0.01ab	>-0.01b*	<0.01ab
	Groundwater	>-0.01ab	>-0.01ab	<0.01a	<0.01ab
	Wastewater	>-0.01e*	>-0.01c*	>-0.01ab	>-0.01d*

[†]Means within a water property with different lowercase letters are different at $P \leq 0.05$.

An asterisk () indicates a mean value differs ($P \leq 0.05$) from a change of zero.

3.1.5. Zinc

The change in runoff Zn concentration ranged from $<0.01 \text{ mg}\cdot\text{L}^{-1}$ for the rainwater-Dapue combination to $-0.12 \text{ mg}\cdot\text{L}^{-1}$ for the groundwater-Calloway combination (Table 5). Similar to runoff K and Mn, the change in runoff Zn concentration did not differ among soils for the wastewater source (Table 5). The change in Zn concentration was, additionally, similar among all soils, except the Calloway soil, in the rainwater source (Table 5). However, the change in runoff Zn concentration differed ($P \leq 0.05$) among soils in the groundwater source, with the change in runoff Zn from the Creldon and Calloway soils differing from both each other

and the Dapue and Roxana soils, which did not differ (**Table 5**). The runoff Zn concentration decreased ($P \leq 0.05$) relative to the unamended control in all soils for the groundwater source (**Table 5**).

3.1.6. Copper

The change in runoff Cu concentration remained within a range of $0.01 \text{ mg}\cdot\text{L}^{-1}$ to $-0.01 \text{ mg}\cdot\text{L}^{-1}$ for all soil-water combinations (**Table 5**). Similar to runoff K, the change in runoff Cu concentration was similar between all soils for the rainwater and groundwater sources (**Table 5**). In contrast, the change in runoff Cu concentration differed ($P \leq 0.05$) among all soils for the wastewater source where concentrations ranged from decreases of $>-0.01 \text{ mg}\cdot\text{L}^{-1}$ in the Creldon, Dapue, and Calloway to no change from zero in the Roxana (**Table 5**). As such, the change in runoff Cu concentration differed ($P \leq 0.05$) from a change of zero for all soil-wastewater combinations, except the Roxana-wastewater combination (**Table 5**). However, outside the soil-wastewater combinations, the only other combination whose change in runoff Cu concentration differed ($P \leq 0.05$) from a change of zero was the Roxana-rainwater combination (**Table 5**).

3.2. Explanation of Nutrient Responses

The significant interactions ($P \leq 0.05$) between soils and water sources revealed unique trends in runoff K, Na, Mn, Zn, and Cu concentration responses, including both increases and decreases in concentrations relative to the unamended control (**Table 4**). The dynamics of specific nutrient responses typically varied across water sources, with few notable responses being unique to any one soil series (**Table 5**).

3.2.1. Wastewater's Pronounced Negative Impacts

The most pronounced effects from wastewater were the large reductions in runoff K concentrations (-21.9 to $-2.1 \text{ mg}\cdot\text{L}^{-1}$) and Na (-7.5 to $-2.4 \text{ mg}\cdot\text{L}^{-1}$) across all soils, with the exception of Na for the wastewater-Calloway combination (**Table 5**). A potential reason for the consistent effect is competitive adsorption of cations on soil exchange sites between multiple species initially present in the wastewater or present on soil exchange sites [21]. Under normal conditions, soil exchange sites adsorb ions with the largest valence, such as Ca^{2+} and Mg^{2+} . However, an increase in total concentration of dissolved solids, such as salts, in the soil solution from wastewater application enhances adsorption of more monovalent species, such as Na^+ and K^+ [22]. The wastewater used in the rainfall runoff simulation had an initial EC of $0.578 \text{ dS}\cdot\text{m}^{-1}$, which could correlate to sufficiently concentrated salts in the wastewater to produce the effect of greater Na and K adsorption compared to Ca and/or Mg adsorption (**Table 3**) [9] [23]. Increased Na and K adsorption to the soil exchange sites may explain the decrease in concentration of water-soluble Na and K in the runoff following the rainfall simulations.

A study conducted by Jalali [24] offered comparable results to the current study. Although Jalali [24] focused on soil nutrient leaching rather than runoff, the study was similar in that it analyzed the interaction between soil, wastewater, and the

nutrient parameters of effluent leaving the soil system following soil-wastewater interaction. In Jalali [24] two soils, both silt loam, were collected from fields irrigated with wastewater. Laboratory-scale soil columns were constructed, and the soils were saturated with the corresponding irrigation source, a distilled control or wastewater. The irrigation source was allowed to flow through the soil columns and leachate was collected and analyzed for nutrient parameters. The amount of irrigation source added was compared to the total pore volume of either soil sample. Jalali [24] reported the minimum leachate Na concentration occurred at 2.1 and 2.7 pore volumes of wastewater, respectively, for soil 1 and 2. The decrease leachate Na concentration indicated soil adsorption of Na had reached its maximum, replacing Ca and Mg on cation exchange sites [24]. However, in contrast to the current study, Jalali [24] observed an increase in leachate K concentration.

3.2.2. Variability in Groundwater Impacts

Runoff nutrient responses exhibited wide ranges of concentration changes in the groundwater treatment group. Potassium concentration changes as a result of soil-groundwater interactions ranged from a net increase of $0.30 \text{ mg}\cdot\text{L}^{-1}$ in the Roxana soil to a net decrease of $-0.21 \text{ mg}\cdot\text{L}^{-1}$ in the Calloway soil (Table 5). Similarly, Na concentration changes in the groundwater group ranged from an increase of $0.12 \text{ mg}\cdot\text{L}^{-1}$ in the Calloway to a decrease of $-1.67 \text{ mg}\cdot\text{L}^{-1}$ in the Dapue soil (Table 5). All Zn concentrations significantly decreased within the groundwater group ($-0.06 \text{ mg}\cdot\text{L}^{-1}$ to $-0.12 \text{ mg}\cdot\text{L}^{-1}$) (Table 5).

A comprehensive review conducted by Shaheen [25] provided a comparable framework for understanding the variable groundwater responses in the current study. Shaheen [25] synthesized results from multiple sorption experiments and field studies examining, specifically, how soil properties controlled nutrient retention or release, including Zn, Cu, and Mn. Shaheen [25] emphasized that the distribution of trace elements is impacted by competition for sorption sites with foreign ions introduced by water entering the soil system. The groundwater used by Morrison [9] was analyzed and reported to have moderate electrical conductivity ($0.461 \text{ dS}\cdot\text{m}^{-1}$) and notable concentrations of cations (*i.e.*, $13.2 \text{ mg}\cdot\text{L}^{-1}$ Na and $1.2 \text{ mg}\cdot\text{L}^{-1}$ K) (Table 3). Thus, the explanation by Shaheen [25] suggests that the groundwater could have introduced additional cations into each soil system, creating conditions under which competition among ions for the same available sorption sites tended to suppress the strength and magnitude of element retention, which may enhance the environmental availability of such elements. The groundwater in Morrison [9] likely acted as a source of additional cations that differentially displaced or exchanged with present soil nutrients through electrostatic sorption and cation exchange reactions. Soil characteristics noted by Shaheen [25] as being impactful on sorption characteristics were pH, clay mineral content, and SOM, Fe/Mn oxide, and calcium carbonate concentrations. Generally, the sorption of nutrients in competitive systems is lower than in mono-metal sorption systems and more strongly adsorbed elements, like Zn, which is seldom impacted by competition, show consistent retention patterns across soils, while other nutri-

ents, such as K and Na, exhibited more variable responses to the interaction of groundwater and soil [25].

The Dapue soil had the largest initial SOM concentration (4.2%) and was most acidic (5.77 pH) and, when interacting with the groundwater source, exhibited decreased runoff Na concentrations ($-1.67 \text{ mg}\cdot\text{L}^{-1}$) and a moderate decrease in Zn ($-0.06 \text{ mg}\cdot\text{L}^{-1}$) (Table 1, Table 5). Using the framework suggested by Shaheen [25], it is likely the organic matter initially present in the Dapue soil contributed sorption sites, which retained nutrients like Na and Zn, decreasing runoff concentrations of both dissolved nutrients. Furthermore, Shaheen [25] noted that Zn appears to be much less affected by competition than other elements, suggesting that, in competitive groundwater-soil ion interactions, Zn retains much of its affinity for specific sorption sites independent of the initial abundance of those sites within each soil. This explanation of Zn sorption behavior reinforces the observation of decreased runoff Zn concentrations across soils in the groundwater group (-0.06 to $-0.12 \text{ mg}\cdot\text{L}^{-1}$), reflecting the soil differences in abundance of specific sorption sites for the Zn to interact with (Table 5).

3.2.3. Sulfur and MAP

Sulfur was unique among the other nutrients, as the runoff S concentration change differed ($P \leq 0.05$) only among fertilizer-P sources. Initial chemical analysis of fertilizer-P sources revealed considerable differences among fertilizer treatment S concentrations. Monoammonium phosphate exhibited a water-soluble S concentration of $13,280 \text{ mg}\cdot\text{kg}^{-1}$, which was several orders of magnitude greater than S concentrations in all other treatments (Table 2). The distinct characteristic of large initial S concentration in MAP directly corresponded with runoff results in which MAP was the only fertilizer treatment to result in a significant increase from a change of zero ($0.20 \text{ mg}\cdot\text{L}^{-1}$).

The large initial S concentration in MAP can be traced back to the inherent refinement processes of the MAP manufacturing process. As explained by Bertau [26], the conventional wet chemical process for creating phosphoric acid, used in fertilizer-P production, involves the digestion of phosphate sources, typically phosphate rock, using sulfuric acid. Depending on the specific phosphate rock material, the wet-process-produced phosphoric acid can contain a variety of impurities, such as sulfate (SO_4) and other trace metals [27]. The concentrated phosphoric acid must then undergo precipitation with calcium or barium salts to remove sulfates as unwanted impurities from the phosphoric acid [28]. However, sulfate impurities in the final phosphoric acid product may still be prevalent, with sulfate concentrations up to 20,300 ppm in phosphoric acids depending on their source and application (Table 3) [28]. After processing, phosphoric acid is lastly reacted with gaseous ammonia to produce MAP which could potentially retain trace amounts of the sulfate impurities [27] [29].

3.2.4. Sodium Response from the Calloway Soil

The runoff Na response from the Calloway soil defied anticipated results based on

the Calloway's initial soil chemistry (**Table 1**). The Calloway soil contained the largest initial water-soluble Na concentration among the four soils ($19.5 \text{ mg}\cdot\text{kg}^{-1}$; **Table 1**). Consequently, it was reasonably expected, from the initially large water-soluble Na concentration, that the Calloway soil would interact with water sources and produce a significant increase in runoff Na concentrations or, at minimum, a decrease in Na runoff concentration comparable to the other soils. However, across all three water sources, runoff Na concentration were unaffected in the Calloway soil (**Table 5**).

A plausible explanation for the paradoxical response of the Calloway soil is potentially grounded in the results of Saygin [30]. Saygins [30] conducted a laboratory experiment aimed at examining the impacts of irrigation water quality and flow velocity on soil solute transport dynamics and ion exchange processes using carbonate-rich clay soils. Saygins [30] utilized undisturbed soil columns to preserve natural soil structure and conducted vertical leaching simulations with tap water and Na-enriched sodic water through the soil columns. The soil used in Saygins [30] was described as a slightly alkaline, non-saline, clay loam. Results from Saygins [30] suggested that the relatively large clay content, combined with the effects of sodicity on aggregate disintegration, resulted in clogging of large- and medium-sized pores and reduced hydraulic conductivity in the soils. The mechanism of soil-pore clogging through aggregate disintegration within soils that have large clay contents could potentially provide an explanation as to why runoff Na concentrations neither significantly increased nor decreased in interactions with the Calloway soil (**Table 5**). The pore-clogging mechanism is further supported by the Calloway's largest initial clay content ($0.12 \text{ g}\cdot\text{g}^{-1}$) and largest initial pH (7.46) compared to the other three soils (**Table 1**), as the elevated soil pH could have also facilitated some soil dispersion [30]. Thus, though not directly measured nor observed in the current study, the initial alkaline pH and notable clay content of the Calloway soil could have created a micro-environment in which clay platelets more readily separated and dispersed that subsequently reduced soil-pore diameters and physically blocked the movement of the present Na ions through the soil, preventing transport and measurement in the resulting runoff.

3.3. Implications

Phosphorus capture and reuse from wastewater rather than the conventional mining of rock phosphate for fertilizer-P sources could potentially aid in stabilizing the fertilizer-P supply, as traditional rock phosphate reserves are projected to be diminished in 100 to 250 years at the current extraction rate [3]. Consequently struvite has significant potential as a means of recycling P from the wastewater stream for use as a fertilizer-P source. Furthermore, struvite recovery from wastewater could lead to reduced nutrient concentrations in discharged wastewater effluent, potentially lowering nutrient loads released into local waterways and protecting receiving waters from environmental impairment [15]. How-

ever, characterizing the interactions among struvite, soil, and water source must be further evaluated across multiple potential agricultural systems to be fully understood [5]. As such, results from the current study indicated that changes in runoff micronutrient concentrations (*i.e.*, K, Na, Mn, Zn, and Cu) were primarily due to interactions between water source and soil, but not fertilizer-P source, such as ECST_{syn} or ECST_{real} (Table 4). Significant fertilizer-P source interactions with micronutrient runoff concentrations were only identified for S (Table 4). Monoammonium phosphate was the only fertilizer treatment to produce a significant ($P \leq 0.05$) elevated S concentration in runoff ($0.20 \text{ mg}\cdot\text{L}^{-1}$) from a change of zero. Results could thus indicate that struvite fertilizer-P sources, which, by comparison, did not differ in an S concentration change from zero, could contribute less S to runoff water than MAP under similar conditions.

Though not directly measured nor observed in the current study, increased runoff-S concentrations in the form of sulfate could introduce S in greater concentrations to surface waters, potentially impacting aquatic zooplankton, macroinvertebrate, and fish species [31]. However, Cao [32] explored the impacts of increased sulfate concentration on zebrafish (*Danio rerio*), reporting that, as the aquatic sulfate concentration in their habitat increased, the external gills of the zebrafish were swollen or developed visibly damaged tissue. Additionally, the groups of zebrafish exposed to both low and high sulfate concentrations experienced a range of spinal curvature from slight in the low sulfate group to severe in the high sulfate group [32]. Zebrafish are just one example species showing severe reactions to elevated sulfate concentrations. Sulfate can additionally cause osmotic stress or specific ion toxicity in aquatic organisms, particularly when concentrations of Ca^{2+} and Mg^{2+} are low, such as in softer waters [33]. Thus, comparatively lower runoff-S concentrations from struvite treatments could be a notable environmental advantage over MAP; however, further characterization is needed at the field scale.

Dissolved nutrients, such as Mn, Zn, and Cu, are essential to aquatic organisms at low concentrations, but can have toxic effect on organism growth, metabolism, and reproduction at increased concentrations [34]. Changes in runoff Mn, Zn, and Cu concentrations were generally small or did not differ from a change of zero, indicating that neither struvite fertilizer treatments nor MAP significantly increased the runoff concentrations of potentially toxic dissolved nutrients (Table 5). A notable exception to this trend was the significant decrease in runoff Zn concentration among all soils under the groundwater source (Table 5). Though not directly measured nor observed in the current study, decreases in runoff Zn concentrations from groundwater may have been due to influences of the groundwater on the soils' exchange sites and competitive adsorption of Zn. However, this possible explanation for the Zn micronutrient response further supported that runoff micronutrient concentrations were largely controlled by interactions between soil and irrigation water source rather than fertilizer-P source. Nonetheless, though few ecologically significant water quality concentration thresholds exist for dissolved nutrients or plant micronutrients, other than for P and N, any in-

creased dissolved nutrient concentrations should be identified and monitored carefully so as to avoid any potential negative ramifications on surface water quality from various soils amended with commercially available, inorganic fertilizers or alternative fertilizer nutrient sources like struvite.

Since the current study was conducted at the laboratory scale using a constructed rainfall-runoff simulator with small soil masses, continued research is needed before results can confidently characterize struvite-soil-water interactions at the field scale. Field conditions, such as larger soil masses, variable irrigation intensities, and preferential runoff flow pathways over and through the soil could impact micronutrient responses. Further studies evaluating struvite's characteristics and interactions with soil and potential irrigation sources at the field scale are needed to continue assessing the implications and potential impacts of struvite's use as an alternative fertilizer-P source.

A key limitation to this study was scale. Soil-fertilizer treatment teabags used in the rainfall-runoff simulation contained only 175 g of soil each, constraining natural soil aggregation and macro-pore dynamics that may influence nutrient responses at the field scale. Furthermore, this study was unable to capture field-scale soil variability and irrigation event intensities, which have the potential to differentially mobilize dissolved nutrients. Future research considerations should evaluate the runoff micronutrient responses at the field scale across similar soils to validate response dynamics under more realistic agroecosystem conditions. Additionally, future research endeavors should consider micronutrient responses under varying application rates, application methods, and across multiple seasonal conditions.

4. Conclusions

The current study evaluated the effects of soil, irrigation water source, and fertilizer-P source on the response of micronutrient concentrations (*i.e.*, S, K, Na, Mn, Zn, and Cu) in runoff water from a laboratory scale, rainfall-runoff simulation experiment. Only runoff-S concentrations were affected by fertilizer-P source, while runoff-K, -Na, -Mn, -Zn, and -Cu concentrations were primarily controlled by soil-water-source interactions. Extending initial results from Morrison [9], results from the current study showed that struvite treatments had comparable impacts on the micronutrient responses compared to MAP, much like the macronutrient responses evaluated by Morrison [9]. Where struvite treatments did not respond similarly to MAP treatments, differences were attributed to the interactions among soil and water sources rather than to the fertilizer-P source.

Struvite is a relatively new, alternative fertilizer-P source that offers promising results in the ability to recover and reuse P from the wastewater stream. However, struvite's characteristics and impacts on agroecosystems have been understudied. While results from both the current study and recent prior studies suggest that struvite has similar runoff water quality implications as MAP, a widely used and thoroughly characterized fertilizer-P source, more research is needed to evaluate

the agricultural and environmental implications of struvite's use at the field scale to better simulate real-world conditions. Efforts to ensure that future agricultural production systems are not undermined by finite rock phosphate sources will be essential in addressing the continued and intensifying nutrient input demands of modern agricultural systems.

Acknowledgements

Machaela Morrison is gratefully acknowledged for her original work on this project.

Conflicts of Interest

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

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